

APPENDIX C: DATA VALIDATION REPORTS

ATTACHMENT C-1: WHOLE-BODY CHEMISTRY

Sayer Data Solutions, Inc.

DATA VALIDATION REPORT



Lower Duwamish Waterway: Juvenile Chinook Salmon, Whole Fish Samples

Prepared for:
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1.0 Introduction

Salmon were collected between May 12 and June 23, 2003. Tissues were homogenized and composited by Analytical Resources Inc., in Tukwila Washington, and were assigned the following sample IDs.

Batch	Original Sample ID	Lab Sample ID	Date Collected
K2307475	LDW-LWa-H-WF-Comp1	K2307475-004	06/23/2003
K2307475	LDW-LWa-H-WF-Comp2	K2307475-005	06/23/2003
K2307475	LDW-LWa-H-WF-Comp3	K2307475-006	06/23/2003
K2307475	LDW-LWa-W-WF-Comp1	K2307475-007	06/23/2003
K2307475	LDW-LWa-W-WF-Comp2	K2307475-008	06/23/2003
K2307475	LDW-LWa-W-WF-Comp3	K2307475-009	06/23/2003
K2307475	LDW-MWb-W-WF-Comp1	K2307475-001	06/23/2003
K2307475	LDW-MWb-W-WF-Comp2	K2307475-002	06/23/2003
K2307475	LDW-MWb-W-WF-Comp3	K2307475-003	06/23/2003
K2307475	LDW-MW-H-WF-Comp1	K2307475-010	06/23/2003
K2307475	LDW-MW-H-WF-Comp2	K2307475-011	06/23/2003
K2307475	LDW-MW-H-WF-Comp3	K2307475-012	06/23/2003
K2307475	LDW-RM18-H-WF-Comp1	K2307475-013	06/18/2003
K2307475	LDW-RM18-H-WF-Comp2	K2307475-014	06/19/2003
K2307475	LDW-RM18-H-WF-Comp3	K2307475-015	06/20/2003
K2307475	LDW-RM18-W-WF-Comp1	K2307475-016	06/18/2003
K2307475	LDW-RM18-W-WF-Comp2	K2307475-017	06/20/2003
K2307475	LDW-RM18-W-WF-Comp3	K2307475-018	06/25/2003
K2307486	LDW-SC-H-WF-Comp1	K2307486-001	05/21/2003
K2307487	LDW-GR-W-WF-Comp1	K2307487-001	05/14/2003
K2307487	LDW-GR-W-WF-Comp2	K2307487-002	05/14/2003
K2307487	LDW-GR-W-WF-Comp3	K2307487-003	05/14/2003
K2307487	LDW-LWa-W-WF-Comp1	K2307487-004	05/12/2003
K2307487	LDW-LWa-W-WF-Comp2	K2307487-005	05/12/2003

Batch	Original Sample ID	Lab Sample ID	Date Collected
K2307487	LDW-LWa-W-WF-Comp3	K2307487-006	05/12/2003
K2307487	LDW-MWa-W-WF-Comp4	K2307487-007	05/13/2003
K2307487	LDW-MWb-W-WF-Comp5	K2307487-008	05/13/2003
K2307487	LDW-MWc-W-WF-Comp6	K2307487-009	05/12/2003

Each sample was analyzed for pesticides, PCBs, tributyl tin, total solids and total lipids. Analyses were performed by Columbia Analytical Services in Kelso, Washington. Data are presented in laboratory reports dated November 20, 21, and December 2, 2003.

The chain of custody (COC) forms included two sets of samples collected on different dates with duplicate sample IDs. Because of the difficulty associated with tracking two different samples with identical sample IDs, the Windward QA/QC Manager (Tad Deshler) decided to alter the sample IDs used in the project database and data report, as follows:

Batch	Original Sample ID	Revised Sample ID	Date Collected
K2307487	LDW-LWa-W-WF-Comp1	LDW-LWa-W-WF-Comp1a	05/12/2003
	LDW-LWa-W-WF-Comp2	LDW-LWa-W-WF-Comp2a	05/12/2003
	LDW-LWa-W-WF-Comp3	LDW-LWa-W-WF-Comp3a	05/12/2003
K2307475	LDW-LWa-W-WF-Comp1	LDW-LWa-W-WF-Comp1b	06/23/2003
	LDW-LWa-W-WF-Comp2	LDW-LWa-W-WF-Comp2b	06/23/2003
	LDW-LWa-W-WF-Comp3	LDW-LWa-W-WF-Comp3b	06/23/2003

A full validation was performed on the analytical results. Validation was performed by Cari Sayler. Data qualifiers are summarized in section 6.0 of this report.

2.0 Pesticide Analyses

Samples were extracted by EPA Method 3540 (Soxhlet). Samples were cleaned using EPA Methods 3640 (GPC) and 3620 (Florisil). Samples were analyzed by EPA Method 8081A. The following data requirements were evaluated:

- Quality control analysis frequencies
- Holding times
- Instrument calibration
- Degradation standard results
- Laboratory blank contamination
- Surrogate recoveries
- Laboratory control sample (LCS) recoveries
- Standard reference material (SRM) results
- Matrix spike (MS) recoveries
- MS/MS Duplicate (MSD) relative percent differences (RPDs)
- Matrix duplicate RPDs
- Compound identifications

- Compound quantitations
- Reporting limits

Quality control analysis frequencies: The QAPP specifies that the following quality control samples be analyzed one per sample group or one per twenty samples, whichever is more frequent: method blank, matrix duplicate, MS, MSD, and SRM. In addition, surrogate compounds must be measured in each field and quality control sample. These frequency requirements were met.

Holding times: Tissue samples must be stored frozen and extracted within one year of collection. Extracts must be analyzed within 40 days of extraction. These holding times were met.

Instrument calibration: Data usability criteria for calibrations include minimum correlation coefficients of 0.990 or maximum RSDs of $\pm 20\%$ for each initial calibration, and maximum % differences of $\pm 25\%$ for each continuing calibration.

The initial calibration data met the 20% RSD criteria. Several of the % differences in the closing calibration standard on November 5 exceeded 25%. For all exceedances except mirex, the criteria were met on one of the two columns and no qualifiers are assigned. Mirex was not detected in the associated samples and the associated reporting limits were qualified as estimated.

Degradation standard results: Functional guidelines criteria for the degradation standard are maximum DDT breakdown of 20%, maximum endrin breakdown of 20%, and maximum combined breakdown of 30%. These criteria were met.

Laboratory blank contamination: Criteria for method blanks are that analyte concentrations must be below the PQL, or below 5% of the lowest associated sample concentration. Methoxychlor was detected in the method blank analyzed with batch K2307475 at a concentration of 1.3 $\mu\text{g}/\text{kg}$ and in the method blank analyzed with batches K2307486/K2307487 at a concentration of 1.5 $\mu\text{g}/\text{kg}$. Methoxychlor was detected in sample LDW-SC-H-WF-Comp1 at a concentration of 6.3 $\mu\text{g}/\text{kg}$. This result should be considered not detected at the reported concentration and was qualified "U". No other samples contained methoxychlor, and no other qualifiers were assigned.

Surrogate recoveries: The project DQI for accuracy was 30 to 150%. All surrogate recoveries were within these limits.

LCS recoveries: The project DQI for accuracy was 30 to 150%. The LCS recoveries were within these limits.

SRM results: The following SRM results exceeded the DQI goal for accuracy.

Compound	SRM True Value ($\mu\text{g}/\text{kg}$)	SRM Result ($\mu\text{g}/\text{kg}$)	Recovery (%)
SRM analyzed with batch K2307475			
Mirex	28.9	46	159
Cis-Nonachlor	48.7	84	172
2,4-DDT	106	200	189

Compound	SRM True Value (µg/kg)	SRM Result (µg/kg)	Recovery (%)
SRM analyzed with batch K2307486/K2307487			
delta-BHC	3.3	7.5	227
2,4'-DDT	106	205	193
beta-BHC	8	13.1	163

The matrix spike recoveries were all within limits and are considered to be more indicative of matrix effects on accuracy. No qualifiers were assigned.

The batch K2307475 SRM result for 4,4-DDD was misreported on the summary QA/QC Report form (page 432). The laboratory has been notified and the form has been hand-corrected.

MS recoveries: The project DQI for accuracy was 30 to 150%. The MS and MSD recoveries were within these limits.

MS/MSD RPDs: The project DQI for precision was <50%. The RPDs were below this level.

Matrix duplicate RPDs: The project DQI for precision was <50%. RPD criterion apply to results that are five times the reporting limit or higher. Results less than five times the reporting limit are evaluated based on the absolute difference between the results, and should be within two reporting limits of each other.

The results for 2,4'-DDE in LDW-LWa-W-WF-Comp3a analyzed in batch K2307486/K2307487 (19 µg/kg) and its matrix duplicate (3.9 µg/kg) exceeded the two times the reporting limit criterion (RL = 2.4 µg/kg). The sample result was qualified as estimated. All other results in the batch K2307486/K2307487 duplicate and all results in the batch K2307475 duplicate met duplicate criteria.

Compound identifications: Chromatograms and quantitation reports were reviewed for accuracy of compound identifications. No discrepancies were noted.

Compound quantitations: Concentrations of randomly selected compounds (gamma-chlordane, 4,4'-DDE, and 4,4'-DDT) from each sample were recalculated to verify sample quantitations. No discrepancies were noted.

Due to matrix interferences, results for several target analytes were reported by the laboratory as nondetected in several samples at an elevated reporting limit. The elevated reporting limits were sufficient to account for the observed levels of interference. All affected results were flagged by the laboratory "Ui", and no further qualification was necessary.

Various compounds throughout all batches had dual column RPDs exceeding the 40% method criterion and were appropriately flagged P by the laboratory. The corresponding validation qualifier of J (estimated) was assigned to each of these results.

Reporting Limits: The QAPP specifies a target detection limit of 3 µg/kg. The method detection limits (MDLs) for toxaphene (a mixture) were 20 to 50 times those of the individual pesticides and did not meet the target detection limit in any sample.

Approximately 10% of the method detection limits (MDLs) were above this level in various samples due to decreased sample volumes or interferences. No qualifiers were assigned based on elevated reporting limits.

Overall assessment: Documentation was found to be clear and complete. No calculation errors were noted. One minor transcription error was noted and corrected. Calibration and instrument check samples indicate acceptable instrument performance. Blank contamination resulted in one elevated reporting limit. Quality control results demonstrated acceptable levels of precision and accuracy.

Pesticide data, as qualified, are acceptable for use.

3.0 PCB Analyses

Samples were extracted by EPA Method 3540 (soxhlet). Samples were cleaned using EPA Methods 3640 (GPC), 3620 (Florisil), and 3665 (Acid). Samples were analyzed by EPA Method 8082. The following data requirements were evaluated:

- Quality control analysis frequencies
- Holding times
- Instrument calibration
- Laboratory blank contamination
- Surrogate recoveries
- LCS recoveries
- MS recoveries
- MS/MSD relative percent differences (RPDs)
- Matrix duplicate RPDs
- Compound identifications
- Compound quantitations
- Reporting limits

Quality control analysis frequencies: The QAPP specifies that the following quality control samples be analyzed one per sample group or one per twenty samples, whichever is more frequent: method blank, matrix duplicate, MS, and MSD. In addition, surrogate compounds must be measured in each field and quality control sample. These frequency requirements were met.

Holding times: Frozen samples must be extracted within one year of collection. Extracts must be analyzed within 40 days of extraction. All holding times were met.

Instrument calibration: Data usability criteria for calibrations include maximum RSDs of $\pm 20\%$ for each initial calibration and maximum % differences of $\pm 25\%$ for each continuing calibration. All initial calibration RSDs were within the $\pm 20\%$ criteria. All continuing calibration compound percent differences were within the 25% criteria.

Laboratory blank contamination: Criteria for method blanks are that analyte concentrations must be below the PQL, or below 5% of the lowest associated sample concentration. No contamination was detected in the method blanks.

Surrogate recoveries: Laboratory reported control limit for surrogate recovery was 22-172%. The project DQI for accuracy was 40-160%. All surrogate recoveries were within these limits.

LCS recoveries: The project DQI for LCS recoveries was 40 to 160%. The LCS recoveries were within these limits.

MS recoveries: The project DQI for MS recoveries was 40 to 160%. All MS and MSD recoveries were within limits.

MS/MSD RPDs: The project DQI for precision was <50%. All MS and MSD RPDs were within limits.

Matrix duplicate RPDs: The project DQI for precision was <50%. RPD criterion apply to results that are five times the reporting limit or higher. Results less than five times the reporting limit are evaluated based on the absolute difference between the results, and should be within two reporting limits of each other. These criteria were met.

Compound identifications: Chromatograms and quantitation reports were reviewed for accuracy of compound identifications. In several samples in batch K23070487, the chromatographic fingerprint pattern did not confirm the presence Aroclors even though peaks were present in the quantitation PCB retention time windows. In each case, the laboratory reported these results with elevated detection limits, flagged Ui, and no further action was required.

Compound quantitations: Concentrations of Aroclor 1254 from each sample were recalculated to verify sample quantitations. No calculation errors were noted.

Due to matrix interferences, results for several target analytes were reported as nondetected by the laboratory in several samples at an elevated reporting limit. The elevated reporting limits were sufficient to account for the observed levels of interference. All affected results were flagged by the laboratory Ui, and no further qualification was necessary.

Various compounds throughout all batches had dual column RPDs exceeding the 40% method criterion and were appropriately flagged P by the laboratory. The corresponding validation qualifier of J (estimated) was assigned to each of these results.

Reporting Limits: The QAPP specifies a target detection limit of 5 µg/kg. MDLs were above this level in various samples due to decreased sample volumes or interferences. Aroclors were detected in all samples with elevated MDLs except samples LDW-GR-W-WF-Comp2 and LDW-GR-W-WF-Comp3. No qualifiers were assigned based on elevated reporting limits.

Overall assessment: Documentation was found to be clear and complete. No calculation or transcription errors were noted. Initial and continuing calibration data indicated acceptable instrument performance. Interferences resulted in some elevated reporting limits and estimated concentrations. Quality control results demonstrated acceptable levels of precision and accuracy.

PCB sample data, as qualified, are acceptable for use.

4.0 Tributyl Tin Analyses

Analyses were performed by Stallard/GC-FPD. The following data requirements were evaluated:

- Quality control analysis frequencies
- Holding times
- Laboratory blank contamination
- Instrument calibration
- Surrogate recoveries
- LCS and MS recoveries
- SRM results
- MS/MSD RPDs
- Matrix duplicate RPDs
- Compound identifications
- Compound quantitations
- Reporting limits

Quality control analysis frequencies: The QAPP specifies that the following quality control samples be analyzed one per sample group or one per twenty samples, whichever is more frequent: method blank, matrix duplicate, MS, MSD, and SRM. In addition, surrogate compounds must be measured in each field and quality control sample. These frequency requirements were met.

Holding times: Frozen samples must be extracted within 1 year sample collection. Extracts must be analyzed within 40 days of extraction. These holding times were met.

Laboratory blank results: Criteria for method blanks are that analyte concentrations must be below the PQL, or below 5% of the lowest associated sample concentration. No contamination was detected in the method blanks.

Instrument calibration: Data usability criteria for calibrations include minimum correlation coefficients of 0.990 or maximum RSDs of $\pm 20\%$ for each initial calibration, and maximum % differences of $\pm 25\%$ for each continuing calibration. These criteria were met.

Surrogate recoveries: Project goals for accuracy were 50-150%. Laboratory control limits were 10-132%. Surrogate recoveries ranged from 33 to 50 percent with only one sample within project goals. All samples except LDW-LWa-H-WF-Comp1 were qualified as estimated for low surrogate recoveries.

LCS and MS recoveries: Project goals for accuracy were 50-150%.

All LCS and spike recoveries were well below the project DQI, although within the laboratory control limit of 10-152%.

Batch	Spike ID	TBT Recovery (%)
K2307475	LDW-MWb-W-WF-Comp3 MS	18
	LDW-MWb-W-WF-Comp3 MSD	17
	LCS 1	18
	LCS 2	27
K2307486 and K2307487	LDW-MWb-W-WF-Comp5 MS	16
	LDW-MWb-W-WF-Comp5 MSD	17
	LCS	17

All sample results were qualified as estimated due to low LCS and spike recoveries.

SRM results: Project goals for accuracy were 50-150%. An SRM was analyzed with the K2307475 batch and the K2307486/K2307487, each with a result of 1100 µg/kg. The published result for this SRM is 2200 ±190 µg/kg. The published acceptance limit is too small to allow for expected analytical variability, and the laboratory utilizes a wider limit of 1000-3600 µg/kg. An acceptability range base on the 50-150% project DQI would be 1100-3300 µg/kg, and this range was met. No qualifiers were assigned.

MS/MSD RPDs: Project goals for precision were less than 50%. The MS/MSD RPDs were within limits.

Matrix duplicate RPDs: Project goals for precision were less than 50%. TBT was not detected in either matrix duplicate or their associated samples and precision could not be evaluated.

Compound identifications: Chromatograms and quantitation reports were reviewed for compliance with identification criteria. No deviations were noted.

Compound quantitations: Concentrations from each sample were recalculated to verify sample quantitations. No discrepancies were noted.

Reporting limits: The QAPP specifies a target detection limit of 2 µg/kg. The 1.5 µg/kg method detection limit met this level.

Overall assessment: Documentation was found to be clear and complete. No calculation, identification, or transcription errors were noted. Calibration data indicated acceptable instrument performance. Quality control results demonstrated acceptable levels of precision. Recoveries below project goals for surrogates, spikes and laboratory control samples resulted in estimated data.

TBT data, as qualified, are acceptable for use.

5.0 General Chemistry Analyses

Total solids analysis was performed by a freeze dry method. The project QAPP specifies EPA Method 160.3. However, method 160.3 is a soil/sediment method and freeze drying is the appropriate method for tissue samples.

Total lipids analysis was performed by the PSEP method. The project QAPP specifies Bligh and Dyer. The laboratory verified that the methods are comparable

for tissue matrices. No qualifiers were assigned based on the different analysis method.

The following data requirements were evaluated:

- Quality control analysis frequencies
- Holding times
- Laboratory blank contamination
- Laboratory duplicate RPDs and triplicate RSDs
- Compound quantitations
- Reporting limits

Quality control analysis frequencies: The QAPP requires a laboratory triplicate for both total solids and total lipids. The following quality control samples were analyzed:

Analysis	QC samples
Total Solids	Laboratory duplicates
Total Lipids	Method blanks and laboratory triplicates

No qualifiers were assigned based on the lack of a laboratory triplicate for total solids.

Holding times: Frozen samples must be analyzed for total solids within 6 months of sample collection. Frozen samples must be analyzed for total lipids within 1 year sample collection. These holding times were met.

Laboratory blank results: Criteria for method blanks are that analyte concentrations must be below the PQL. The lipid method blank met this criteria.

Laboratory duplicate RPDs and triplicate RSDs: Project DQIs for precision were $\pm 20\%$ for total solids and $\pm 30\%$ for total lipids. Duplicate RPDs and triplicate RSDs were below this level.

Compound quantitations: Concentrations from each sample were recalculated to verify sample quantitations. No discrepancies were noted.

Reporting limits: Lipids and solids were present in each sample and reporting limit evaluation does not apply.

Overall assessment: Documentation was found to be clear and complete. No calculation or transcription errors were noted. Quality control results demonstrated acceptable levels of precision.

General chemistry data, as reported, are acceptable for use.

6.0 Qualifier Summary Table

Sample	Analyte	DV Qualifier	Reason
Pesticide Analyses			
LDW-GR-W-WF-Comp1	2,4'-DDT	J	High dual column RPD

Sample	Analyte	DV Qualifier	Reason
LDW-GR-W-WF-Comp2	4,4'-DDE	J	High dual column RPD
LDW-GR-W-WF-Comp2	Endrin	J	High dual column RPD
LDW-GR-W-WF-Comp2	gamma-BHC (Lindane)	J	High dual column RPD
LDW-GR-W-WF-Comp2	trans-Nonachlor	J	High dual column RPD
LDW-GR-W-WF-Comp3	2,4'-DDE	J	High dual column RPD
LDW-GR-W-WF-Comp3	2,4'-DDT	J	High dual column RPD
LDW-GR-W-WF-Comp3	Dieldrin	J	High dual column RPD
LDW-GR-W-WF-Comp3	Heptachlor Epoxide	J	High dual column RPD
LDW-GR-W-WF-Comp3	trans-Nonachlor	J	High dual column RPD
LDW-LWa-H-WF-Comp1	2,4'-DDT	J	High dual column RPD
LDW-LWa-H-WF-Comp2	2,4'-DDE	J	High dual column RPD
LDW-LWa-H-WF-Comp2	2,4'-DDT	J	High dual column RPD
LDW-LWa-H-WF-Comp2	4,4'-DDE	J	High dual column RPD
LDW-LWa-H-WF-Comp2	Heptachlor Epoxide	J	High dual column RPD
LDW-LWa-H-WF-Comp3	2,4'-DDT	J	High dual column RPD
LDW-LWa-H-WF-Comp3	4,4'-DDD	J	High dual column RPD
LDW-LWa-H-WF-Comp3	Endosulfan I	J	High dual column RPD
LDW-LWa-H-WF-Comp3	Endrin Aldehyde	J	High dual column RPD
LDW-LWa-W-WF-Comp1b	2,4'-DDD	J	High dual column RPD
LDW-LWa-W-WF-Comp1b	2,4'-DDT	J	High dual column RPD
LDW-LWa-W-WF-Comp1b	Heptachlor	J	High dual column RPD
LDW-LWa-W-WF-Comp1b	Heptachlor Epoxide	J	High dual column RPD
LDW-LWa-W-WF-Comp1a	2,4'-DDD	J	High dual column RPD
LDW-LWa-W-WF-Comp1a	2,4'-DDT	J	High dual column RPD
LDW-LWa-W-WF-Comp1a	4,4'-DDD	J	High dual column RPD
LDW-LWa-W-WF-Comp2b	2,4'-DDD	J	High dual column RPD
LDW-LWa-W-WF-Comp2a	2,4'-DDE	J	High dual column RPD
LDW-LWa-W-WF-Comp2a	2,4'-DDT	J	High dual column RPD
LDW-LWa-W-WF-Comp2a	Heptachlor	J	High dual column RPD
LDW-LWa-W-WF-Comp2a	Oxychlordane	J	High dual column RPD
LDW-LWa-W-WF-Comp3b	2,4'-DDT	J	High dual column RPD
LDW-LWa-W-WF-Comp3b	Endosulfan I	J	High dual column RPD
LDW-LWa-W-WF-Comp3b	gamma-Chlordane	J	High dual column RPD
LDW-LWa-W-WF-Comp3b	Heptachlor Epoxide	J	High dual column RPD
LDW-LWa-W-WF-Comp3a	2,4'-DDE	J	High dual column RPD, Matrix duplicate variability
LDW-LWa-W-WF-Comp3a	2,4'-DDT	J	High dual column RPD
LDW-LWa-W-WF-Comp3a	Oxychlordane	J	High dual column RPD
LDW-MWa-W-WF-Comp4	Mirex	UJ	High CCal %difference
LDW-MWa-W-WF-Comp4	Endrin	J	High dual column RPD
LDW-MWa-W-WF-Comp4	Heptachlor	J	High dual column RPD
LDW-MWa-W-WF-Comp4	trans-Nonachlor	J	High dual column RPD
LDW-MWb-W-WF-Comp1	2,4'-DDD	J	High dual column RPD
LDW-MWb-W-WF-Comp1	Aldrin	J	High dual column RPD
LDW-MWb-W-WF-Comp1	Endosulfan I	J	High dual column RPD
LDW-MWb-W-WF-Comp2	4,4'-DDE	J	High dual column RPD
LDW-MWb-W-WF-Comp2	beta-BHC	J	High dual column RPD
LDW-MWb-W-WF-Comp2	gamma-Chlordane	J	High dual column RPD
LDW-MWb-W-WF-Comp3	4,4'-DDE	J	High dual column RPD
LDW-MWb-W-WF-Comp3	4,4'-DDT	J	High dual column RPD
LDW-MWb-W-WF-Comp3	beta-BHC	J	High dual column RPD
LDW-MWb-W-WF-Comp3	Endosulfan Sulfate	J	High dual column RPD
LDW-MWb-W-WF-Comp3	gamma-BHC (Lindane)	J	High dual column RPD
LDW-MWb-W-WF-Comp5	alpha-Chlordane	J	High dual column RPD
LDW-MWb-W-WF-Comp5	Endrin	J	High dual column RPD
LDW-MWb-W-WF-Comp5	Endrin Aldehyde	J	High dual column RPD
LDW-MWb-W-WF-Comp5	Mirex	UJ	High CCal %difference
LDW-MWc-W-WF-Comp6	2,4'-DDE	J	High dual column RPD
LDW-MWc-W-WF-Comp6	2,4'-DDT	J	High dual column RPD
LDW-MWc-W-WF-Comp6	4,4'-DDE	J	High dual column RPD
LDW-MWc-W-WF-Comp6	gamma-Chlordane	J	High dual column RPD
LDW-MWc-W-WF-Comp6	Mirex	UJ	High CCal %difference
LDW-MWc-W-WF-Comp6	Oxychlordane	J	High dual column RPD
LDW-MW-H-WF-Comp1	Endosulfan I	J	High dual column RPD

Sample	Analyte	DV Qualifier	Reason
LDW-MW-H-WF-Comp1	Heptachlor	J	High dual column RPD
LDW-MW-H-WF-Comp1	Heptachlor Epoxide	J	High dual column RPD
LDW-MW-H-WF-Comp1	Oxychlordane	J	High dual column RPD
LDW-MW-H-WF-Comp2	2,4'-DDT	J	High dual column RPD
LDW-MW-H-WF-Comp2	Endrin	J	High dual column RPD
LDW-MW-H-WF-Comp2	Oxychlordane	J	High dual column RPD
LDW-MW-H-WF-Comp3	2,4'-DDT	J	High dual column RPD
LDW-MW-H-WF-Comp3	4,4'-DDE	J	High dual column RPD
LDW-MW-H-WF-Comp3	alpha-Chlordane	J	High dual column RPD
LDW-MW-H-WF-Comp3	Endrin	J	High dual column RPD
LDW-MW-H-WF-Comp3	Heptachlor Epoxide	J	High dual column RPD
LDW-RM18-H-WF-Comp1	4,4'-DDD	J	High dual column RPD
LDW-RM18-H-WF-Comp1	Endosulfan Sulfate	J	High dual column RPD
LDW-RM18-H-WF-Comp2	2,4'-DDT	J	High dual column RPD
LDW-RM18-H-WF-Comp2	Endosulfan I	J	High dual column RPD
LDW-RM18-H-WF-Comp2	Endosulfan Sulfate	J	High dual column RPD
LDW-RM18-H-WF-Comp2	gamma-Chlordane	J	High dual column RPD
LDW-RM18-H-WF-Comp3	2,4'-DDE	J	High dual column RPD
LDW-RM18-H-WF-Comp3	2,4'-DDT	J	High dual column RPD
LDW-RM18-H-WF-Comp3	4,4'-DDD	J	High dual column RPD
LDW-RM18-H-WF-Comp3	Heptachlor Epoxide	J	High dual column RPD
LDW-RM18-W-WF-Comp1	beta-BHC	J	High dual column RPD
LDW-RM18-W-WF-Comp1	Endosulfan I	J	High dual column RPD
LDW-RM18-W-WF-Comp1	trans-Nonachlor	J	High dual column RPD
LDW-RM18-W-WF-Comp2	2,4'-DDE	J	High dual column RPD
LDW-RM18-W-WF-Comp2	Endosulfan I	J	High dual column RPD
LDW-RM18-W-WF-Comp2	Endrin Aldehyde	J	High dual column RPD
LDW-RM18-W-WF-Comp3	2,4'-DDE	J	High dual column RPD
LDW-RM18-W-WF-Comp3	Dieldrin	J	High dual column RPD
LDW-RM18-W-WF-Comp3	Endrin	J	High dual column RPD
LDW-RM18-W-WF-Comp3	Endrin Aldehyde	J	High dual column RPD
LDW-RM18-W-WF-Comp3	gamma-BHC (Lindane)	J	High dual column RPD
LDW-RM18-W-WF-Comp3	Heptachlor	J	High dual column RPD
LDW-RM18-W-WF-Comp3	Heptachlor Epoxide	J	High dual column RPD
LDW-SC-H-WF-Comp1	2,4'-DDD	J	High dual column RPD
LDW-SC-H-WF-Comp1	Dieldrin	J	High dual column RPD
LDW-SC-H-WF-Comp1	Methoxychlor	U	Method blank contamination
LDW-SC-H-WF-Comp1	trans-Nonachlor	J	High dual column RPD
PCB Analyses			
LDW-GR-W-WF-Comp1	Aroclor 1254	J	High dual column RPD
LDW-GR-W-WF-Comp1	Aroclor 1260	J	High dual column RPD
LDW-LWa-H-WF-Comp1	Aroclor 1254	J	High dual column RPD
LDW-LWa-W-WF-Comp1a	Aroclor 1254	J	High dual column RPD
LDW-LWa-W-WF-Comp1a	Aroclor 1260	J	High dual column RPD
LDW-LWa-W-WF-Comp2a	Aroclor 1254	J	High dual column RPD
LDW-LWa-W-WF-Comp2a	Aroclor 1260	J	High dual column RPD
LDW-LWa-W-WF-Comp3b	Aroclor 1254	J	High dual column RPD
LDW-LWa-W-WF-Comp3a	Aroclor 1260	J	High dual column RPD
LDW-LWa-W-WF-Comp3a	Aroclor 1254	J	High dual column RPD
LDW-MWb-W-WF-Comp2	Aroclor 1254	J	High dual column RPD
LDW-MWb-W-WF-Comp3	Aroclor 1254	J	High dual column RPD
LDW-MWb-W-WF-Comp5	Aroclor 1254	J	High dual column RPD
LDW-MWc-W-WF-Comp6	Aroclor 1254	J	High dual column RPD
LDW-MW-H-WF-Comp1	Aroclor 1254	J	High dual column RPD
LDW-MW-H-WF-Comp1	Aroclor 1260	J	High dual column RPD
LDW-MW-H-WF-Comp2	Aroclor 1254	J	High dual column RPD
LDW-MW-H-WF-Comp2	Aroclor 1260	J	High dual column RPD
LDW-MW-H-WF-Comp3	Aroclor 1254	J	High dual column RPD
LDW-RM18-H-WF-Comp2	Aroclor 1254	J	High dual column RPD
LDW-RM18-H-WF-Comp3	Aroclor 1254	J	High dual column RPD
LDW-RM18-W-WF-Comp1	Aroclor 1254	J	High dual column RPD
LDW-RM18-W-WF-Comp2	Aroclor 1254	J	High dual column RPD
LDW-RM18-W-WF-Comp3	Aroclor 1254	J	High dual column RPD
LDW-SC-H-WF-Comp1	Aroclor 1254	J	High dual column RPD

Sample	Analyte	DV Qualifier	Reason
TBT Analyses			
All samples except LDW-LWa-H-WF-Comp1	TBT	J/UJ	Low surrogate, LCS, MS, and MSD recoveries
LDW-LWa-H-WF-Comp1	TBT	J/UJ	Low LCS, MS, and MSD recoveries

7.0 Abbreviations and Definitions

<u>DV Qualifier</u>	<u>Definition</u>
U	The material was analyzed for, but was not detected above the level of the associated value.
J	The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
UJ	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
R	The sample result is rejected. The presence or absence of the analyte cannot be verified and data are not usable.

<u>Abbreviation</u>	<u>Definition</u>
DV	Data validation
LCS	Laboratory control sample
MS	Matrix spike
MSD	Matrix spike duplicate
SRM	Standard reference material
RPD	Relative percent difference
Surr	Surrogate
CCal	Continuing calibration

8.0 References

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Method 8081A: Organochlorine Pesticides by Gas Chromatography, *SW-846, Third Edition*, US Environmental Protection Agency, Office of Solid Waste, December 1996.

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ATTACHMENT C-2: STOMACH CONTENT CHEMISTRY

Sayler Data Solutions, Inc.

DATA VALIDATION REPORT



Lower Duwamish Waterway: Juvenile Chinook Salmon, Stomach Contents Samples

Prepared for:
Windward Environmental LLC
200 W. Mercer St. Suite 401
Seattle, WA 98119

February 20, 2004

1.0 Introduction

Salmon were collected on June 24, 2003. The stomach contents were composited by Windward Environmental LLC in Seattle, Washington and homogenized by Columbia Analytical Services (CAS) in Kelso, Washington. Analyses were also performed by CAS. The sample was assigned laboratory batch number K2308006. Data are presented in a laboratory report dated November 13, 2003.

A full validation was performed on the analytical results. Validation was performed by Cari Sayler. Data qualifiers are summarized in section 5.0 of this report.

2.0 Polynuclear Aromatic Hydrocarbon and Alkylated PAH Homolog Analysis

The laboratory referenced EPA Method 8270C SIM as the analysis method for these compounds. However, due to the lack of availability of PAH homolog standards, the quality control frequencies, quantitation, and integration procedures for PAH homologs are significantly different from those of Method 8270C and CAS's method is better described as a laboratory in-house method. The following data requirements were evaluated:

- Quality control analysis frequencies
- Holding times
- Instrument performance check (tune) ion abundances
- Instrument calibration
- Laboratory blank contamination
- Surrogate recoveries
- Laboratory control sample (LCS) recoveries
- Matrix spike (MS) recoveries

- Standard reference material (SRM) results
- Internal standard areas and retention time shifts
- Compound identifications
- Compound quantitations
- Reporting limits

Quality control analysis frequencies: Method 8270 specifies that the following quality control samples be analyzed one per analytical batch or one per twenty samples, whichever is more frequent: method blank, LCS, MS, and either MSD, or laboratory duplicate. In addition, surrogate compounds must be measured in each field and quality control sample. Section 3.5.3 of the QAPP states that analytical replicates are not required for the stomach contents sample due to the limited sample volume.

This laboratory batch included a method blank, LCS, LCSD, MS, SRM, and appropriate surrogates. However, due to the lack of availability of PAH homolog standards, no PAH homolog compounds were included in the quality control samples. This meets laboratory method requirements and no qualifiers were assigned.

Holding times: Tissue samples must be stored frozen and extracted within 1 year of sample collection. Extracts must be analyzed within 40 days of extraction. All holding times were met.

Instrument performance check (tune) ion abundances: Ion abundance criteria exist for 11 ions in decafluorotriphenylphosphine. These criteria were met in each 12 hour standard.

Instrument calibration: Functional guidelines criteria for calibrations include minimum response factors of 0.05, initial calibration maximum relative standard deviations (RSDs) of 30%, and continuing calibration maximum % differences of $\pm 25\%$.

The method criteria allow for lower responses and exceedances of individual compound RSDs if the average of all RSDs is below 15%. All method and functional guideline criteria for instrument calibration were met for PAH compounds. As per the laboratory method, PAH homologs were not included in the standards used for calibration and linearity and were not evaluated for these analytes.

Laboratory blank contamination: Criteria for method blanks are that analyte concentrations must be below the PQL, or below 5% of the lowest associated sample concentration. The method blank contained biphenyl (0.31 $\mu\text{g}/\text{kg}$), indeno (1,2,3-cd)pyrene (0.76 $\mu\text{g}/\text{kg}$), dibenzo(a,h)anthracene (0.46 $\mu\text{g}/\text{kg}$) and benzo(g,h,i)perylene (1.3 $\mu\text{g}/\text{kg}$) at concentrations below the PQL. The dibenzo(a,h)anthracene result in sample LDW-LW-H-SC-Comp1 was within 10 times this concentration. This result should be considered not detected at the reported concentration and was qualified "U". Sample concentrations of the three remaining blank contaminants were above 10 times the blank concentration and no qualifiers were required.

Surrogate recoveries: Laboratory reported control limits for surrogate recoveries ranged from 54-102 to 53-116%. All surrogate recoveries were within the laboratory limits and within project DQI goals for accuracy.

LCS recoveries: Project DQI goal for accuracy was 40-130%. The LCS recoveries were within these limits.

MS recoveries: Project DQI goal for accuracy was from 40-130%.

Recoveries in the MS were as follows:

Compound	Sample Result (µg/kg)	MS Result (µg/kg)	Recovery (%)	Lab Control Limit (%)	Exceeds DQI Goal (40-130%)
Naphthalene	21	404	77	46-109	
2-Methylnaphthalene	26	454	86	44-118	
1-Methylnaphthalene	19	438	84	57-96	
Biphenyl	12	426	83	58-103	
Acenaphthylene	2.7	409	81	63-114	
Dibenzofuran	40	646	121	64-102	
Acenaphthene	38	590	111	59-117	
Fluorene	55	784	146	67-115	*
Dibenzothiophene	26	613	118	70-130	
Phenanthrene	380	4000 E	724	67-109	*
Anthracene	21	540	104	71-114	
Fluoranthene	350	4090 E	749	66-120	*
Pyrene	240	2610 E	477	59-110	*
Benz(a)anthracene	32	669	128	55-108	
Chrysene	62	1200	227	61-110	*
Benzo(b)fluoranthene	22	755	147	64-119	*
Benzo(k)fluoranthene	28	651	125	68-117	
Benzo(e)pyrene	19	546	105	60-118	
Benzo(a)pyrene	10	556	109	67-117	
Perylene	2.7	424	84	70-130	
Indeno(1,2,3-cd)pyrene	8.4	600	119	47-137	
Dibenz(a,h)anthracene	0.9	415	83	51-133	
Benzo(g,h,i)perylene	6.5	472	93	58-114	

The sample was spiked at a level of 499 µg/kg. Recoveries in the MS were most affected for the compounds with high native concentrations, suggesting sample in-homogeneity. Concentration differences of up to an order of magnitude are suggested for the higher concentration compounds. Positive results for compounds with recoveries outside the DQI goal and their associated alkylated homologs were qualified as estimated.

SRM results: Results for 6 of the 8 PAH compounds in the SRM were below the CAS advisory limit.

Compound	SRM True Value (µg/kg)	SRM Result (µg/kg)	Recovery (%)	Advisory Limit (µg/kg)	Below DQI Goal (40-130%)
Phenanthrene	2.5	1.0	40.0%	1.1-5.6	
Fluoranthene	19	6.6	34.7%	8.8-39	*

Compound	SRM True Value (µg/kg)	SRM Result (µg/kg)	Recovery (%)	Advisory Limit (µg/kg)	Below DQI Goal (40-130%)
Pyrene	18	5.7	31.7%	8.3-36	*
Benzo(a)anthracene	3.7	0.99	26.8%	1.6-8.5	*
Chrysene	11	3.4	30.9%	4.9-23	*
Benzo(b)fluoranthene	5.3	1.4	26.4%	2.4-11	*
Benzo(k)fluoranthene	4.6	1.1	23.9%	2.2-9.9	*
Benzo(a)pyrene	1.8	0.74	41.1%	0.85-3.7	

The sample results for the six compounds that are also below the DQI goal and their alkylated homologs were qualified as estimated.

Internal standard areas and retention time shifts: Internal standard area counts in each sample must not vary by more than a factor of 2 from the associated 12 hour standard. The internal standard retention times in each sample must not vary by more than ± 30 seconds from the associated 12-hour standard. All internal standard area counts and retention times were within range.

Compound identifications: Compound identification criteria in a SIM analysis include: 1) the relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the standard component, 2) the intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other, and 3) the relative intensities of the characteristic ions agree within 30% of those in the reference spectra.

These criteria were evaluated and no deviations were noted for the PAH compounds.

The retention time window for the PAH homologs is based on the evaluation of a petroleum crude oil which has been cleaned up using GPC and silica gel and which contains all of the target homologs as well as other components. With several slight exceptions, the manual integrations were within the retention time window. All exceptions were based on the presence of the primary and secondary ion in the correct ratios and were considered acceptable.

For C2-chrysenes, discrepancies in the fingerprint pattern (i.e. peaks were not maximizing in the same scan for all peaks) were observed, possibly due to the low concentration present. The sample result was qualified as both estimated "J" and presumptively present "N".

For three of the homologs the secondary ion ratio criteria were not met: C4-naphthalenes, C1-dibenzothiophenes, and C4-phenanthrenes/anthracene. Review of the ion abundance chromatographs for C4-naphthalenes and C4-phenanthrenes/anthracene show that the secondary ion was present in the correct ratio for most of the peaks in the integration range, confirming the identification of each homolog group. However, for one or two large primary ion peaks there was no corresponding large peak for the secondary ion. This indicates that one or two non-homolog compounds were included in the quantitation of these sample results. The laboratory was contacted and the sample was re-integrated excluding the non-homolog peaks and the data were resubmitted. For C1-dibenzothiophenes, the error was determined to be an incorrectly tabulated criterion. Secondary ion ratios in the

resubmitted data were either within criteria or slightly outside criteria and the resubmitted data were considered acceptable. Corrected concentrations are 146 µg/kg for C4-naphthalenes, 110 µg/kg for C1-dibenzothiophenes, and 58 µg/kg for C4-phenanthrenes/anthracene.

Compound quantitations: Concentrations of five randomly selected PAH compounds, phenanthrene, fluoranthene, benzo(k)fluoranthene, indeno(1,2,3-cd)pyrene, and benzo(g,h,i)perylene were recalculated to verify sample quantitations. No discrepancies were noted.

For PAH homologs, sample concentrations are calculated using the response factor of the most similar PAH as defined on the initial calibration summary form. Concentrations of two randomly selected PAH homologs, C2-phenanthrenes/anthracenes and C1-chrysenes were recalculated to verify sample quantitations. No discrepancies were noted.

No MDL study has been performed for the PAH homologs. Reporting limits for the two non-detected homologs have been qualified as estimated.

Reporting limits: The QAPP specifies a target detection limit of 1 to 5 µg/kg. The 4 µg/kg reporting limit met this target. However, the reporting limits have been estimated due to the absence of an MDL study. The impact on the target detection limit is unknown.

Overall assessment: Documentation was found to be clear and complete. The method reference was corrected and three results were re-quantitated by the laboratory. No other calculation, identification, or transcription errors were noted. Calibration data and internal standard results indicate acceptable instrument performance. High MS recoveries, possibly due to sample variability, and low SRM recoveries resulted in some estimated concentrations. The presence of blank contamination resulted in one elevated reporting limit. The absence of an MDL study resulted in estimated reporting limits.

PAH and alkylated PAH homolog data, as qualified, are acceptable for use.

3.0 Metals Analysis

Analyses were performed by EPA Method 6010B and 6020. The following data requirements were evaluated:

- Quality control analysis frequencies
- Holding times
- Instrument tune mass calibration and resolution (6020 only)
- Instrument calibration
- Reporting limit standard recoveries
- Laboratory blank contamination
- Interference Check Sample (ICS) results
- MS recoveries
- SRM results

- Laboratory duplicate RPDs
- Serial dilution RPDs
- Internal standard relative intensities (6020 only)
- Compound quantitations
- Reporting limits

Quality control analysis frequencies: The method specifies that the following quality control samples be analyzed one per analytical batch or one per twenty samples, whichever is more frequent: method blank, LCS or SRM, MS and laboratory duplicate. This batch included a method blank, SRM, MS, and laboratory duplicate.

Holding times: Sediment samples must be analyzed within 1 year of collection. The sample was analyzed within the holding time.

Instrument tune mass calibration and resolution: Method criteria for ICP-MS mass calibration are peak widths of 0.9 amu or less at 10% peak height and mass differences of 0.1 amu or less. These criteria were met.

Instrument calibration: The method criterion for calibration verification is a maximum % difference of $\pm 10\%$ for ICP and ICP-MS metals. This criterion was met.

Reporting limit standard: The functional guidelines criterion for the reporting limit standard is 70-130% recovery for both ICP and ICP-MS analyses. The recoveries of copper (135%), lead (136%), and silver (135%) were above this level. The sample results for copper and lead were greater than 2 times the reporting limit, and no qualifiers are necessary. The sample result for silver was qualified as estimated.

Laboratory blank contamination: Criteria for method blanks are that analyte concentrations must be below the PQL, or below 10% of the lowest associated sample concentration. Criteria for calibration blanks are that analyte concentrations must be below the PQL, or below 10% of the lowest associated sample concentration. No contamination was detected in the method or calibration blanks.

ICS results: The method requires analysis of an interference check sample (ICS) at the beginning and end of the analytical sequence or twice each eight-hour shift, whichever is more frequent. The functional guidelines criteria for the ICS are 80-120% recovery of the spiked analytes and the absolute values of the non-spiked analytes must be less than the reporting limit. These criteria were met.

MS recoveries: All MS recoveries were within the project DQI of 75 to 125%.

SRM results: The laboratory analyzed NRCC Dorm-2 and NRCC Dolt-3 Tissue SRMs. All results were within the published SRM limits.

Serial dilutions RPDs: Serial dilution RPDs were within the functional guidelines criterion.

Internal standard relative intensities: The functional guidelines criterion for internal standards is relative intensities within 60-125% of the initial calibration blank. This criterion was met.

Compound quantitations: Concentrations were recalculated to verify sample quantitations. No discrepancies were noted:

Reporting limits: Each metal was detected in the sample and reporting limit evaluation does not apply.

Overall assessment: Documentation was found to be clear and complete. No calculation, identification, or transcription errors were noted. One result was estimated due to a high reporting limit standard recovery. Instrument performance was otherwise acceptable. Quality control results demonstrated acceptable levels of precision and accuracy.

Metals data, as qualified, are acceptable for use.

4.0 Total Solids Analysis

Analysis was performed by a freeze-dry method. The project QAPP specifies EPA Method 160.3. However, method 160.3 is a soil/sediment method and freeze drying is the appropriate method for tissue samples. The following data requirements were evaluated:

- Quality control analysis frequencies
- Holding times
- Compound quantitations
- Reporting limits

Quality control analysis frequencies: Triplicate analysis was not required for the stomach contents samples due to the limited sample volume available.

Holding times: The QAPP specified a maximum holding time of 6 months for frozen samples. This holding time was met.

Compound quantitations: The total solid result was recalculated to verify sample quantitations. No discrepancy was noted.

Reporting limits: Solids were present in the sample and reporting limit evaluation does not apply.

Total solid data, as reported, are acceptable for use.

5.0 Qualifier Summary Table

Sample	Analyte	DV Qualifier	Reason
PAH and Alkylated PAH Homolog Analysis			
LDW-LW-H-SC-Comp1	Fluorene	J	MS results indicative of variability
	C1-Fluorenes	J	MS results indicative of variability
LDW-LW-H-SC-Comp1	C2-Fluorenes	J	MS results indicative of variability
	C3-Fluorenes	J	MS results indicative of variability
	Phenanthrene	J	MS results indicative of variability
	C1-Phenanthrenes/Anthracenes	J	MS results indicative of variability
	C2-Phenanthrenes/Anthracenes	J	MS results indicative of variability

Sample	Analyte	DV Qualifier	Reason
	C3-Phenanthrenes/Anthracenes	J	MS results indicative of variability
	C4-Phenanthrenes/Anthracenes	J	MS results indicative of variability
	Fluoranthene	J	MS results indicative of variability, low SRM recovery
	Benzo(a)anthracene	J	Low SRM recovery
	Pyrene	J	MS results indicative of variability, low SRM recovery
	Chrysene	J	MS results indicative of variability, low SRM recovery
	C1-Chrysenes	J	MS results indicative of variability, low SRM recovery
	C2-Chrysenes	JN	Weak fingerprint pattern match. MS results indicative of variability, low SRM recovery
	C3-Chrysenes	UJ	Low SRM recovery, no MDL study
	C4-Chrysenes	UJ	Low SRM recovery, no MDL study
	Benzo(b)fluoranthene	J	MS results indicative of variability, low SRM recovery
	Benzo(k)fluoranthene	J	Low SRM recovery
	Dibenzo(a,h)anthracene	U	Blank Contamination
Metals Analysis			
LDW-LW-H-SC-Comp1	Silver	J	High reporting limit standard recovery

6.0 Abbreviations and Definitions

<u>DV Qualifier</u>	<u>Definition</u>
U	The material was analyzed for, but was not detected above the level of the associated value.
J	The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
UJ	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
R	The sample result is rejected. The presence or absence of the analyte cannot be verified and data are not usable.

<u>Abbreviation</u>	<u>Definition</u>
DV	Data validation
LCS	Laboratory control sample
MS	Matrix spike
MSD	Matrix spike duplicate
SRM	Standard reference material
RPD	Relative percent difference
Surr	Surrogate

7.0 References

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