

APPENDIX F

Data Validation Reports



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

March 16, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05364201 - 05424009)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Forty Seven (47) tissue samples were analyzed for total elements by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05364201	05364202	05364203	05364204	05364205	05364206	05364207
05364208	05364209	05364210	05374200	05374201	05374202	05374203
05374204	05374205	05374206	05374207	05374208	05374209	05374210
05374213	05374214	05374215	05374216	05374217	05374218	05374219
05374220	05374221	05374222	05374223	05374224	05374225	05374226
05414001	05414002	05414003	05414004	05414005	05414006	05414007
05424001	05424002	05424005	05424006	05424009		

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding time from the date of collection to the date of digestion and analyses were met for all elements (180 days). Samples were collected on 9/6/05 thru 10/20/05. ICP-AES analysis was conducted on 2/1/06 thru 2/20/06, ICP-MS analysis on 2/8/06 thru 2/25/06 and mercury analyses on 1/31/06 and 2/1/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For ICP-AES analysis, instrument calibration was performed with a blank and six multi-component standards. Recoveries for instrument verification standards (98-110%) met recovery (90-110%) criterion.

For ICP-MS analysis, instrument calibration was performed with a blank and five multi-component standards. Recoveries for instrument verification standards (96-107%) met the recovery (90-110%) criterion.

For mercury, instrument calibration was performed with a blank and eight standards. Correlation coefficients (0.999) met the criterion (≥ 0.995). Recoveries for verification standards (88-105%) met the recovery (80-120%) criterion.

3.0 ICP-AES INTERFERENCE CHECK SAMPLE (ICS) - Acceptable

ICS recoveries met the acceptance criterion (\pm MRL) for all elements.

4.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (96-108%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within the control limits.

5.0 BLANKS - Acceptable

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

6.0 MATRIX SPIKE ANALYSIS

Matrix spikes were analyzed for samples 05374210, 05424002 and 05424006. Percent recoveries (82-122%) met the recovery criterion (75-125%) for all elements with the exception of aluminum (56-59%). Affected sample results were qualified (J or UJ).

7.0 DUPLICATE SAMPLE ANALYSIS - Acceptable

Duplicate samples were analyzed for samples 05374210, 05424002 and 05424006. Relative percent differences ($\leq 17\%$) were within the control limits ($\pm 20\%$).

8.0 ICP-AES SERIAL DILUTION - Acceptable

A five-fold serial dilution was analyzed for samples 05374210, 05424002 and 05424006. Percent differences ($\leq 9\%$) met the control limits ($\leq 10\%$) for all applicable elements.

9.0 ASSESSMENT SUMMARY

All results for total elements were reported on a "wet weight" basis. The following is a summary of qualified data:

A number of reported values for aluminum were qualified (J or UJ) due to a low matrix spike recovery. Qualified aluminum values may be biased low.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 15, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05424057 - 05424080)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Twenty Four (24) tissue samples were analyzed for total elements by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05424057	05424058	05424059	05424060	05424061	05424062	05424063
05424064	05424065	05424066	05424067	05424068	05424069	05424070
05424071	05424072	05424073	05424074	05424075	05424076	05424077
05424078	05424079	05424080				

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding times from the date of collection to the date of digestion and analyses were within the water criteria (180 days). Samples were collected on 10/18/05 thru 10/22/05. ICP-AES analysis was conducted on 4/10/06 thru 4/17/06 and ICP-MS analysis on 3/31/06 thru 4/17/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For ICP-AES analysis, instrument calibration was performed with a blank and six multi-component standards. Recoveries for instrument verification standards (98-108%) met recovery (90-110%) criterion.

For ICP-MS analysis, instrument calibration was performed with a blank and five multi-component standards. Recoveries for instrument verification standards (93-107%) met the recovery (90-110%) criterion.

3.0 ICP-AES INTERFERENCE CHECK SAMPLE (ICS) - Acceptable

ICS recoveries met the acceptance criterion (\pm MRL) for all elements.

4.0 LABORATORY CONTROL SAMPLES (LCS)

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (96-112%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within the control limits with the exception of magnesium and zinc. Magnesium data for 05424057 thru 05424073 and zinc data for 05424074 thru 05424080 were qualified (J) and may be biased high.

5.0 BLANKS - Acceptable

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

6.0 MATRIX SPIKE ANALYSIS - Acceptable

Matrix spikes were analyzed for samples 05424064 and 05424074. Percent recoveries (78-119%) met the recovery criterion (75-125%) for all elements.

7.0 DUPLICATE SAMPLE ANALYSIS

Duplicate samples were analyzed for samples 05424064 and 05424074. Relative percent differences ($\leq 18\%$) were within the control limits ($\pm 20\%$) with the exception of calcium (25%). Calcium data for 05424057 thru 05424073 were qualified (J).

8.0 ICP-AES SERIAL DILUTION - Acceptable

A five-fold serial dilution was analyzed for samples 05424064 and 05424074. Percent differences ($\leq 9\%$) met the control limits ($\leq 10\%$) for all applicable elements.

9.0 ASSESSMENT SUMMARY

For those tests for which Manchester Environmental Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The analyses for this sample set consisted of two digestion batches consisting of samples 05424057 - 05424073 and 05424074 - 05424080. The following is a summary of qualified data:

A number of magnesium and zinc results were qualified (J) due to high recoveries for the laboratory control sample. Qualified magnesium and zinc values may be biased high.

A number of calcium results were qualified (J) due to a high relative percent difference for the sample duplicate.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 5, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05424016 - 05424043)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Twenty Eight (28) tissue samples were analyzed for total elements by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05424016	05424017	05424018	05424019	05424020	05424021	05424022
05424023	05424024	05424025	05424026	05424027	05424028	05424029
05424030	05424031	05424032	05424033	05424034	05424035	05424036
05424037	05424038	05424039	05424040	05424041	05424042	05424043

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding time from the date of collection to the date of digestion and analyses were met for all elements (180 days). Samples were collected on 10/18/05 thru 10/21/05. ICP-AES analysis was conducted on 3/29/06 thru 3/20/06 and ICP-MS analysis on 3/30/06 thru 4/4/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For ICP-AES analysis, instrument calibration was performed with a blank and six multi-component standards. Recoveries for instrument verification standards (94-110%) met recovery (90-110%) criterion.

For ICP-MS analysis, instrument calibration was performed with a blank and five multi-component standards. Recoveries for instrument verification standards (93-107%) met the recovery (90-110%) criterion.

3.0 ICP-AES INTERFERENCE CHECK SAMPLE (ICS) - Acceptable

ICS recoveries met the acceptance criterion (\pm MRL) for all elements.

4.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (95-108%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within the control limits.

5.0 BLANKS - Acceptable

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

6.0 MATRIX SPIKE ANALYSIS - Acceptable

Matrix spikes were analyzed for samples 05424024 and 05424026. Percent recoveries (76-118%) met the recovery criterion (75-125%) for all elements.

7.0 DUPLICATE SAMPLE ANALYSIS

Duplicate samples were analyzed for samples 05424024 and 05424026. Relative percent differences (\leq 14%) were within the control limits (\pm 20%) with the exception of calcium (27%) in 05424024. Affected samples were qualified (J).

8.0 ICP-AES SERIAL DILUTION - Acceptable

A five-fold serial dilution was analyzed for samples 05424024 and 05424026. Percent differences (\leq 6%) met the control limits (\leq 10%) for all applicable elements.

9.0 ASSESSMENT SUMMARY

All results for total elements were reported on a "dry weight" basis. The following is a summary of qualified data:

A number of reported values for calcium were qualified (J) due to a high relative percent difference for the sample duplicate.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 16, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05374245 - 05374256, 05414008, 05424044 - 05424056)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Twenty Six (26) tissue samples were analyzed for total elements by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05374245	05374246	05374247	05374248	05374249	05374250	05374251
05374252	05374253	05374254	05374255	05374256	05414008	05424044
05424045	05424046	05424047	05424048	05424049	05424050	05424051
05424052	05424053	05424054	05424055	05424056		

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding times from the date of collection to the date of analyses were outside the water criteria (180 days). However, as the samples were frozen and the holding time did not exceed a one year criteria used for tissues, no action was taken. Samples were collected on 9/13/05 thru 10/21/05. ICP-AES analysis was conducted on 3/29/06 thru 4/18/06 and ICP-MS analysis on 3/30/06 thru 4/17/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For ICP-AES analysis, instrument calibration was performed with a blank and six multi-component standards. Recoveries for instrument verification standards (93-108%) met recovery (90-110%) criterion.

For ICP-MS analysis, instrument calibration was performed with a blank and five multi-component standards. Recoveries for instrument verification standards (94-104%) met the recovery (90-110%) criterion.

3.0 ICP-AES INTERFERENCE CHECK SAMPLE (ICS) - Acceptable

ICS recoveries met the acceptance criterion (\pm MRL) for all elements.

4.0 LABORATORY CONTROL SAMPLES (LCS)

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (96-110%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within the control limits with the exception of magnesium. Magnesium data for 05424050 thru 05424056 were qualified (J) and may be biased high.

5.0 BLANKS

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results with the exception of cobalt. Affected samples were qualified (U) for cobalt.

6.0 MATRIX SPIKE ANALYSIS - Acceptable

Matrix spikes were analyzed for samples 05424045 and 05424050. Percent recoveries (76-117%) met the recovery criterion (75-125%) for all elements.

7.0 DUPLICATE SAMPLE ANALYSIS

Duplicate samples were analyzed for samples 05424045 and 05424050. Relative percent differences ($\leq 15\%$) were within the control limits ($\pm 20\%$) with the exception of calcium (27%). Calcium data for 05374245 - 05374256, 05414008, 0542044 - 05424049 were qualified (J).

8.0 ICP-AES SERIAL DILUTION - Acceptable

A five-fold serial dilution was analyzed for samples 05424045 and 05424050. Percent differences ($\leq 8\%$) met the control limits ($\leq 10\%$) for all applicable elements.

9.0 ASSESSMENT SUMMARY

For those tests for which Manchester Environmental Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The analyses for this sample set consisted of two digestion batches consisting of samples 05374245 - 05374256, 05414008, 0542044 - 05424049 and 05424050 - 05424056. The following is a summary of qualified data:

A number of cobalt results were qualified (U) due to the detection of cobalt in the sample preparation blank.

A number of magnesium results were qualified (J) due to a high recovery for the laboratory control sample. Qualified magnesium values may be biased high.

A number of calcium results were qualified (J) due to a high relative percent difference for the sample duplicate.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 15, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05374257 - 05374265)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Seven (7) tissue samples were analyzed for total elements by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05374257 05374258 05374261 05374262 05374263 05374264 05374265

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding times from the date of collection to the date of digestion and analyses were outside the water criteria (180 days). However, as the samples were frozen and the holding time did not exceed a one year criteria used for tissues, no action was taken. Samples were collected on 9/13/05 and 9/14/05. ICP-AES analysis was conducted on 3/27/06 thru 4/19/06 and ICP-MS analysis on 3/30/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For ICP-AES analysis, instrument calibration was performed with a blank and six multi-component standards. Recoveries for instrument verification standards (96-108%) met recovery (90-110%) criterion.

For ICP-MS analysis, instrument calibration was performed with a blank and five multi-component standards. Recoveries for instrument verification standards (93-104%) met the recovery (90-110%) criterion.

3.0 ICP-AES INTERFERENCE CHECK SAMPLE (ICS) - Acceptable

ICS recoveries met the acceptance criterion (\pm MRL) for all elements.

4.0 LABORATORY CONTROL SAMPLES (LCS)

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (96-107%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within the control limits with the exception of sodium. Sodium values were qualified (J) and may be biased low.

5.0 BLANKS - Acceptable

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

6.0 MATRIX SPIKE ANALYSIS

Matrix spikes were analyzed for sample 05374257. Percent recoveries (79-116%) met the recovery criterion (75-125%) for all elements with the exception of copper (74%), iron (-14%) and zinc (186%). All copper, iron and zinc data were qualified (J). Iron data may be biased low whereas zinc data may be biased high.

7.0 DUPLICATE SAMPLE ANALYSIS

Duplicate samples were analyzed for sample 05374257. Relative percent differences (\leq 9%) were within the control limits (\pm 20%) with the exception of copper (24%). All copper data were qualified (J).

8.0 ICP-AES SERIAL DILUTION - Acceptable

A five-fold serial dilution was analyzed for sample 05374257. Percent differences (\leq 5%) met the control limits (\leq 10%) for all applicable elements.

9.0 ASSESSMENT SUMMARY

For those tests for which Manchester Environmental Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The following is a summary of qualified data:

Sodium data were qualified (J) due to a low recovery for the laboratory control sample. Sodium values may be biased low.

Copper data were qualified (J) due to a low matrix spike recovery and a high relative percent difference for the sample duplicate.

Iron data were qualified (J) due to a low matrix spike recovery. Iron data may be biased low.

Zinc data were qualified (J) due to a high matrix spike recovery. Zinc data may be biased high.

DATA QUALIFIERS

U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.

J - The associated value is an estimated quantity.

R - The data are unusable. The analyte may or may not be present in the sample.

UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 16, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05364216 - 05364222, 05414009 - 05414018, 05414025, 05414026)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Nineteen (19) tissue samples were analyzed for total elements by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05364216	05364217	05364218	05364219	05364220	05364221	05414222
05414009	05414010	05414011	05414012	05414013	05414014	05414015
05414016	05414017	05414018	05414025	05414026		

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding times from the date of collection to the date of analyses were outside the water criteria (180 days). However, as the samples were frozen and the holding time did not exceed a one year criteria used for tissues, no action was taken. Samples were collected on 9/6/05 thru 10/14/05. ICP-AES analysis was conducted on 4/24/06 and ICP-MS analysis on 4/25/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For ICP-AES analysis, instrument calibration was performed with a blank and six multi-component standards. Recoveries for instrument verification standards (95-110%) met recovery (90-110%) criterion.

For ICP-MS analysis, instrument calibration was performed with a blank and five multi-component standards. Recoveries for instrument verification standards (91-110%) met the recovery (90-110%) criterion.

3.0 ICP-AES INTERFERENCE CHECK SAMPLE (ICS) - Acceptable

ICS recoveries met the acceptance criterion (\pm MRL) for all elements.

4.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (101-109%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within the control limits.

5.0 BLANKS - Acceptable

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

6.0 MATRIX SPIKE ANALYSIS

Matrix spikes were analyzed for sample 05364221. Percent recoveries (82-120%) met the recovery criterion (75-125%) for all elements with the exception of aluminum (184%), copper (-131%), chromium (72%) and lead (-9%, 47%). Data for these elements were qualified (J). Aluminum values may be biased high whereas chromium values may be biased low.

7.0 DUPLICATE SAMPLE ANALYSIS

Duplicate samples were analyzed for sample 05364221. Relative percent differences (\leq 17%) were within the control limits (\pm 20%) with the exception of copper (23%), lead (100%) and zinc (39%). Data for these elements were qualified (J).

8.0 ICP-AES SERIAL DILUTION - Acceptable

A five-fold serial dilution was analyzed for sample 05364221. Percent differences (\leq 10%) met the control limits (\leq 10%) for all applicable elements.

9.0 ASSESSMENT SUMMARY

For those tests for which Manchester Environmental Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The following is a summary of qualified data:

Aluminum data were qualified (J) due to a high spike recovery. Aluminum values may be biased high.

Chromium data were qualified (J) due to a low spike recovery. Chromium values may be biased low.

Copper and lead data were qualified (J) due to low spike recoveries and high relative percent differences for the duplicate sample comparison.

Zinc data were qualified (J) due to a high relative percent difference for the duplicate sample comparison.

DATA QUALIFIERS

U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.

J - The associated value is an estimated quantity.

R - The data are unusable. The analyte may or may not be present in the sample.

UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101**

May 17, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05414019, 05414027 - 05414029, 05424081 - 05424094)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Eighteen (18) tissue samples were analyzed for total elements by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05414019	05414027	05414028	05414029	05424081	05424082	05424083
05424084	05424085	05424086	05424087	05424088	05414089	05424090
05424091	05424092	05424093	05424094			

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding times from the date of collection to the date of preparation and analyses were within the water criteria (180 days). Samples were collected on 10/14/05 thru 10/19/05 and digested on 3/29/06. ICP-AES analysis was conducted on 4/25/06 and ICP-MS analysis on 4/27/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For ICP-AES analysis, instrument calibration was performed with a blank and six multi-component standards. Recoveries for instrument verification standards (94-108%) met recovery (90-110%) criterion.

For ICP-MS analysis, instrument calibration was performed with a blank and five multi-component standards. Recoveries for instrument verification standards (93-103%) met the recovery (90-110%) criterion.

3.0 ICP-AES INTERFERENCE CHECK SAMPLE (ICS) - Acceptable

ICS recoveries met the acceptance criterion (\pm MRL) for all elements.

4.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (101-112%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within the control limits.

5.0 BLANKS - Acceptable

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

6.0 MATRIX SPIKE ANALYSIS

Matrix spikes were analyzed for samples 05414019. Percent recoveries (76-114%) met the recovery criterion (75-125%) for all elements with the exception of aluminum (71%) and manganese (-0.3%; 55%). Aluminum and manganese data were qualified (J). Aluminum values may be biased low.

7.0 DUPLICATE SAMPLE ANALYSIS

Duplicate samples were analyzed for samples 05414019. Relative percent differences ($\leq 20\%$) were within the control limits ($\pm 20\%$) with the exception of barium (31%), lead (31%), manganese (31%) and calcium (50%). Results for these elements were qualified (J).

8.0 ICP-AES SERIAL DILUTION - Acceptable

A five-fold serial dilution was analyzed for samples 05414019. Percent differences ($\leq 6\%$) met the control limits ($\leq 10\%$) for all applicable elements.

9.0 ASSESSMENT SUMMARY

For those tests for which Manchester Environmental Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The following is a summary of qualified data:

Aluminum results were qualified (J) due to a low matrix spike recovery. Aluminum values may be biased low.

Manganese results were qualified (J) due to low matrix spike recoveries and a high relative percent difference for the duplicate sample comparison.

Barium, calcium and lead results were qualified (J) due to high relative percent differences for the duplicate sample comparison.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 17, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05424095, 05424096, 05424253 - 05424258, 05424265 - 05424270)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Fourteen (14) tissue samples were analyzed for total elements by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05424095	05424096	05424253	05424254	05424255	05424256	05424257
05424258	05424265	05424266	05424267	05424268	05414269	05424270

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding times from the date of collection to the date of preparation and analyses were within the water criteria (180 days). Samples were collected on 10/18/05 and 10/19/05 and digested on 4/03/06. ICP-AES analysis was conducted on 5/1/06 and ICP-MS analysis on 4/27/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For ICP-AES analysis, instrument calibration was performed with a blank and six multi-component standards. Recoveries for instrument verification standards (90-109%) met recovery (90-110%) criterion.

For ICP-MS analysis, instrument calibration was performed with a blank and five multi-component standards. Recoveries for instrument verification standards (93-104%) met the recovery (90-110%) criterion.

3.0 ICP-AES INTERFERENCE CHECK SAMPLE (ICS) - Acceptable

ICS recoveries met the acceptance criterion (\pm MRL) for all elements.

4.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (101-112%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within the control limits.

5.0 BLANKS - Acceptable

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

6.0 MATRIX SPIKE ANALYSIS

Matrix spikes were analyzed for sample 05424096. Percent recoveries (77-120%) met the recovery criterion (75-125%) for all elements with the exception of iron (16%). Iron data were qualified (J). Iron values may be biased low.

7.0 DUPLICATE SAMPLE ANALYSIS

Duplicate samples were analyzed for sample 05424096. Relative percent differences ($\leq 20\%$) were within the control limits ($\pm 20\%$) with the exception of barium (27%), calcium (40%) and manganese (23%). Results for these elements were qualified (J).

8.0 ICP-AES SERIAL DILUTION - Acceptable

A five-fold serial dilution was analyzed for sample 05424096. Percent differences ($\leq 5\%$) met the control limits ($\leq 10\%$) for all applicable elements.

9.0 ASSESSMENT SUMMARY

For those tests for which Manchester Environmental Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The following is a summary of qualified data:

Iron results were qualified (J) due to a low matrix spike recovery. Iron values may be biased low.

Barium, calcium and manganese results were qualified (J) due to high relative percent differences for the duplicate sample comparison.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 16, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05364211 - 05364215, 05414020 - 05414022, 05414024, 05424097 - 05424099, 05424250 - 05424252, 05424259, 05424260, 05424262, 05424263)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Nineteen (19) tissue samples were analyzed for total elements by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05364211	05364212	05364213	05364214	05364215	05414020	05414021
05414022	05414024	05424097	05424098	05424099	05424250	05424251
05424252	05424259	05424260	05424262	05424263		

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding times from the date of collection to the date of analyses were outside the water criteria (180 days). However, as the samples were frozen and the holding time did not exceed a one year criteria used for tissues, no action was taken. Samples were collected on 9/7/05 thru 10/19/05. ICP-AES analysis was conducted on 4/27/06 and ICP-MS analysis on 4/27/06 and 4/28/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For ICP-AES analysis, instrument calibration was performed with a blank and six multi-component standards. Recoveries for instrument verification standards (96-108%) met recovery (90-110%) criterion.

For ICP-MS analysis, instrument calibration was performed with a blank and five multi-component standards. Recoveries for instrument verification standards (91-103%) met the recovery (90-110%) criterion.

3.0 ICP-AES INTERFERENCE CHECK SAMPLE (ICS) - Acceptable

ICS recoveries met the acceptance criterion (\pm MRL) for all elements.

4.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (97-107%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within the control limits.

5.0 BLANKS - Acceptable

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

6.0 MATRIX SPIKE ANALYSIS

Matrix spikes were analyzed for sample 05414020. Percent recoveries (76-125%) met the recovery criterion (75-125%) for all elements with the exception of manganese (-27%, -32%). Manganese data were qualified (J).

7.0 DUPLICATE SAMPLE ANALYSIS

Duplicate samples were analyzed for sample 05414020. Relative percent differences (\leq 18%) were within the control limits (\pm 20%) with the exception of manganese (32%) and nickel (21%). Manganese and nickel data were qualified (J).

8.0 ICP-AES SERIAL DILUTION - Acceptable

A five-fold serial dilution was analyzed for sample 05414020. Percent differences (\leq 4%) met the control limits (\leq 10%) for all applicable elements.

9.0 ASSESSMENT SUMMARY

For those tests for which Manchester Environmental Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. Barium was reported for both the ICP-AES and ICP-MS analyses. No significant differences were observed between the two data sets. The following is a summary of qualified data:

Manganese results were qualified (J) due to a low recoveries for the matrix spikes and a high relative percent difference for the duplicate sample comparison.

Nickel results were qualified (J) due to a high relative percent difference for the duplicate sample comparison.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 17, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05414023, 05424261, 05424264)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Three (3) tissue samples were analyzed for total elements by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05414023 05424261 05424264

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding times from the date of collection to the date of preparation and analyses were within the water criteria (180 days). Samples were collected on 10/14/05 and 10/18/05 and digested on 4/5/06. ICP-AES analysis was conducted on 4/25/06 and ICP-MS analysis on 4/18/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For ICP-AES analysis, instrument calibration was performed with a blank and six multi-component standards. Recoveries for instrument verification standards (94-108%) met recovery (90-110%) criterion.

For ICP-MS analysis, instrument calibration was performed with a blank and five multi-component standards. Recoveries for instrument verification standards (95-103%) met the recovery (90-110%) criterion.

3.0 ICP-AES INTERFERENCE CHECK SAMPLE (ICS) - Acceptable

ICS recoveries met the acceptance criterion (\pm MRL) for all elements.

4.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (100-110%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within the control limits.

5.0 BLANKS - Acceptable

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

6.0 MATRIX SPIKE ANALYSIS

Matrix spikes were analyzed for sample 05424261. Percent recoveries (93-122%) met the recovery criterion (75-125%) for all elements with the exception of antimony (56%; 56%) and nickel (127%). Antimony and nickel data were qualified (J). Antimony values may be biased low whereas nickel values may be biased high.

7.0 DUPLICATE SAMPLE ANALYSIS - Acceptable

Duplicate samples were analyzed for sample 05424261. Relative percent differences (\leq 6%) were within the control limits (\pm 20%).

8.0 ICP-AES SERIAL DILUTION - Acceptable

A five-fold serial dilution was analyzed for sample 05424261. Percent differences (\leq 7%) met the control limits (\leq 10%) for all applicable elements.

9.0 ASSESSMENT SUMMARY

For those tests for which Manchester Environmental Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The following is a summary of qualified data:

Antimony results were qualified (J) due to a low matrix spike recoveries. Antimony values may be biased low.

Nickel results were qualified (J) due to a high matrix spike recovery. Nickel values may be biased high.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

March 22, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Mercury in Fish Tissue (05424016 - 05424043)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Twenty eight (28) tissue samples were analyzed for total mercury by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05424016	05424017	05424018	05424019	05424020	05424021	05424022
05424023	05424024	05424025	05424026	05424027	05424028	05424029
05424030	05424031	05424032	05424033	05424034	05424035	05424036
05424037	05424038	05424039	05424040	05424041	05424042	05424043

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding time from the date of collection to the date of digestion and analyses were met for all elements (180 days). Samples were collected on 10/18/05 thru 10/21/05. Mercury analyses was conducted on 2/9/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For mercury, instrument calibration was performed with a blank and eight standards. Correlation coefficients (0.999) met the criterion (≥ 0.995). Recoveries for verification standards (102-105%) met the recovery (80-120%) criterion. Low level standard recoveries (105-109%) met the recovery (70-130%) criterion.

3.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

An aqueous Laboratory Control Sample and a fish tissue reference standard were digested and analyzed. Recoveries for the aqueous LCS (104-105%) met percent recovery requirements (85-115%) and recoveries for the fish tissue standard were within established control limits.

4.0 BLANKS - Acceptable

Preparation and instrument control blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

5.0 MATRIX SPIKE ANALYSIS - Acceptable

Matrix spikes were analyzed for samples 05424024, 05424026 and 05424034. Percent recoveries (93-99%) met the recovery criterion (75-125%) for all samples.

6.0 DUPLICATE SAMPLE ANALYSIS - Acceptable

Duplicate samples were analyzed for samples 05424024, 05424026 and 05424034. Relative percent differences ($\leq 6\%$) were within the control limits ($\pm 20\%$).

7.0 ASSESSMENT SUMMARY

All results for total elements were reported on a "wet weight" basis. No data were qualified for this review.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

March 22, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Mercury in Fish Tissue (05374227-05374244; 05424010-05424015)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Twenty four (24) tissue samples were analyzed for total mercury by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05374227	05374228	05374229	05374230	05374231	05374232	05374233
05374234	05374235	05374236	05374237	05374238	05374239	05374240
05374241	05374242	05374243	05374244	05424010	05424011	05424012
05424013	05424014	05424015				

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding time from the date of collection to the date of digestion and analyses were met for all elements (180 days). Samples were collected on 9/13/05 thru 10/22/05. Mercury analyses was

conducted on 1/17/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For mercury, instrument calibration was performed with a blank and eight standards. Correlation coefficients (0.999) met the criterion (≥ 0.995). Recoveries for verification standards (87-93%) met the recovery (80-120%) criterion. Low level standard recoveries (89-104%) met the recovery (70-130%) criterion.

3.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

Both aqueous and fish tissue reference standards were digested and analyzed. Recoveries for the aqueous LCS (86-91%) met percent recovery requirements (85-115%) with the exception of one standard (83%). As this was within 20% of the true value (Functional Guidelines), no action was taken. Recoveries for the fish tissue standard were within established control limits.

4.0 BLANKS - Acceptable

Preparation and instrument blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

5.0 MATRIX SPIKE ANALYSIS - Acceptable

Matrix spikes were analyzed for samples 05374228, 05374237 and 05424010. Percent recoveries (78-83%) met the recovery criterion (75-125%) for all samples.

6.0 DUPLICATE SAMPLE ANALYSIS - Acceptable

Duplicate samples were analyzed for samples 05374228, 05374237 and 05424010. Relative percent differences ($\leq 4\%$) were within the control limits ($\pm 20\%$).

7.0 ASSESSMENT SUMMARY

All results for total elements were reported on a "wet weight" basis. No data were qualified for this review.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

March 22, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Mercury in Fish Tissue (05374245-05374256; 05414008; 05424044-05424056)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Twenty six (26) tissue samples were analyzed for total mercury by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05374245	05374246	05374247	05374248	05374249	05374250	05374251
05374252	05374253	05374254	05374255	05374256	05414008	05424044
05424045	05424046	05424047	05424048	05424049	05424050	05424051
05424052	05424053	05424054	05424055	05424056		

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding time from the date of collection to the date of digestion and analyses were met for all elements (180 days). Samples were collected on 9/13/05 thru 10/21/05. Mercury analyses was

conducted on 2/15/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For mercury, instrument calibration was performed with a blank and eight standards. Correlation coefficients (0.999) met the criterion (≥ 0.995). Recoveries for verification standards (100-104%) met the recovery (80-120%) criterion. Low level standard recoveries (106-114%) met the recovery (70-130%) criterion.

3.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

Both aqueous and fish tissue reference standards were digested and analyzed. Recoveries for the aqueous LCS (98-102%) met percent recovery requirements (85-115%). Recoveries for the fish tissue standard were within established control limits.

4.0 BLANKS - Acceptable

Preparation and instrument blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

5.0 MATRIX SPIKE ANALYSIS - Acceptable

Matrix spikes were analyzed for samples 05374245, 05374255 and 05424045. Percent recoveries (89-98%) met the recovery criterion (75-125%) for all samples.

6.0 DUPLICATE SAMPLE ANALYSIS - Acceptable

Duplicate samples were analyzed for samples 05374245, 05374255 and 05424045. Relative percent differences ($\leq 9\%$) were within the control limits ($\pm 20\%$).

7.0 ASSESSMENT SUMMARY

All results for total elements were reported on a "wet weight" basis. No data were qualified for this review.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

March 22, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Mercury in Fish Tissue (05374257, 05374258; 05374261-05374265)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Seven (7) tissue samples were analyzed for total mercury by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05374257 05374258 05374261 05374262 05374263 05374264 05374265

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding time from the date of collection to the date of digestion and analyses were met for all elements (180 days). Samples were collected on 9/13/05 thru 9/14/05. Mercury analyses was conducted on 2/15/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For mercury, instrument calibration was performed with a blank and eight standards. Correlation coefficients (0.999) met the criterion (≥ 0.995). Recoveries for verification standards (100-104%) met the recovery (80-120%) criterion. Low level standard recoveries (105-106%) met the recovery (70-130%) criterion.

3.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

Both aqueous and fish tissue reference standards were digested and analyzed. Recoveries for the aqueous LCS (101-102%) met percent recovery requirements (85-115%). Recoveries for the fish tissue standard were within established control limits.

4.0 BLANKS - Acceptable

Preparation and instrument blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

5.0 MATRIX SPIKE ANALYSIS - Acceptable

A matrix spike analysis was conducted for sample 05374257. Percent recoveries (84-89%) met the recovery criterion (75-125%).

6.0 DUPLICATE SAMPLE ANALYSIS - Acceptable

A duplicate analysis was conducted for sample 05374257. The relative percent difference ($\leq 1\%$) was within the criterion ($\pm 20\%$).

7.0 ASSESSMENT SUMMARY

All results for total elements were reported on a "wet weight" basis. No data were qualified for this review.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

March 22, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Mercury in Fish Tissue (05424057-05424080)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Twenty four (24) tissue samples were analyzed for total mercury by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05424057	05424058	05424059	05424060	05424061	05424062	05424063
05424064	05424065	05424066	05424067	05424068	05424069	05424070
05424071	05424072	05424073	05424074	05424075	05424076	05424077
05424078	05424079	05424080				

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding time from the date of collection to the date of digestion and analyses were met for all elements (180 days). Samples were collected on 10/18/05 thru 10/22/05. Mercury analyses was conducted on 3/14/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For mercury, instrument calibration was performed with a blank and seven standards. Correlation coefficients (0.999) met the criterion (≥ 0.995). Recoveries for verification standards (96-98%) met the recovery (80-120%) criterion. Low level standard recoveries (97-101%) met the recovery (70-130%) criterion.

3.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

Both aqueous and fish tissue reference standards were digested and analyzed. Recoveries for the aqueous LCS (98-101%) met percent recovery requirements (85-115%). Recoveries for the fish tissue standard were within established control limits.

4.0 BLANKS - Acceptable

Preparation and instrument blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

5.0 MATRIX SPIKE ANALYSIS - Acceptable

Matrix spikes were analyzed for samples 05424064 and 05424077. Percent recoveries (92-102%) met the recovery criterion (75-125%).

6.0 DUPLICATE SAMPLE ANALYSIS - Acceptable

Duplicate samples were analyzed for samples 05424064 and 05424077. Relative percent differences ($\leq 8\%$) were within the criterion ($\pm 20\%$).

7.0 ASSESSMENT SUMMARY

All results for total elements were reported on a "wet weight" basis. No data were qualified for this review.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

March 28, 2006

Reply To
Attn. Of: **OEA-095**

MEMORANDUM

SUBJECT: Data Validation for Upper Columbia RI-FS, Analysis of Total Mercury in Fish Tissue (05364221, 05364222, 05414009-05414019, 05424081-05424096)

FROM: Donald Matheny, Chemist
Technical Support Unit, OEA

TO: Sally Thomas, Regional Project Manager
Office of Environmental Cleanup (ECL-111)

CC: Jim Stefanoff, CH2MHill

The data validation of inorganic analyses for the above sample set is complete. Twenty nine (29) tissue samples were analyzed for total mercury by the USEPA Region 10 Manchester Environmental Laboratory, Port Orchard, WA. Sample numbers are as follows:

05364221	05364222	05414009	05414010	05414011	05414012	05414013
05414014	05414015	05414016	05414017	05414018	05414019	05424081
05424082	05424083	05424084	05424085	05424086	05424087	05424088
05424089	05424090	05424091	05424092	05424093	05424094	05424095
05424096						

DATA QUALIFICATIONS

Data qualifications are based on the quality control requirements outlined in the Laboratory's Quality Assurance Manual, the project QAPP, the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94-013" and the reviewer's judgment. The comments presented herein are based on the documentation provided for the review.

1.0 TIMELINESS - Acceptable

The holding time from the date of collection to the date of digestion and analyses were met for all elements (180 days). Samples were collected on 9/6/05 thru 10/19/05. Mercury analyses was conducted on 2/23/06 thru 3/9/06.

2.0 INSTRUMENT CALIBRATION/VERIFICATION - Acceptable

For mercury, instrument calibrations were performed with a blank and seven standards. Correlation coefficients (0.999) met the criterion (≥ 0.995). Recoveries for verification standards (97-100%) met the recovery (80-120%) criterion. Low level standard recoveries (91-105%) met the recovery (70-130%) criterion.

3.0 LABORATORY CONTROL SAMPLES (LCS) - Acceptable

Both aqueous and fish tissue reference standards were digested and analyzed. Recoveries for the aqueous LCS (98-100%) met percent recovery requirements (85-115%). Recoveries for the fish tissue standard were within established control limits.

4.0 BLANKS - Acceptable

Preparation and instrument blanks were prepared and analyzed in accordance with method requirements. Blank values were either non-detected or below a concentration that could impact sample results.

5.0 MATRIX SPIKE ANALYSIS - Acceptable

Matrix spikes were analyzed for samples 05364221, 05414019 and 05424088. Percent recoveries (94-100%) met the recovery criterion (75-125%).

6.0 DUPLICATE SAMPLE ANALYSIS - Acceptable

Duplicate samples were analyzed for samples 05364221, 05414019 and 05424088. Relative percent differences ($\leq 5\%$) were within the criterion ($\pm 20\%$).

7.0 ASSESSMENT SUMMARY

All results for total elements were reported on a "wet weight" basis. For those tests for which Manchester Environmental Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. No data were qualified for this review.

DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. The analyte may or may not be present in the sample.
- UJ - The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10 LABORATORY
7411 Beach Dr. East
Port Orchard, Washington 98366

February 3, 2006

MEMORANDUM

SUBJECT: Data Review for PCB Aroclors and Percent Lipids in fish tissue for Upper Columbia River

Project Code: TEC-774G Account Code: 05T10P302DD2C106XLA00

FROM: Steven Reimer, Chemist, USEPA Region 10 Laboratory
Office of Environmental Assessment

TO: Sally Thomas, Project Manager, USEPA Region 10
Office of Environmental Cleanup

CC: Kevin Rochlin, USEPA Region 10
Office of Environmental Cleanup

Monica Tonel, USEPA Region 10
Office of Environmental Cleanup

Jim Stefanoff, CH2M Hill

The data review of the PCB Aroclor analysis results for the fish tissue samples has been completed. The samples were prepared and analyzed by the USEPA Region 10 Laboratory staff located in Manchester, WA using EPA methods SW-846 3541, 3620, 3665A and 8082.

Reviewed in this report are data for sample numbers:

05364201 05364202 05364203 05364204 05364205 05364206 05364207 05364208
05364209 05364210

DATA QUALIFICATIONS

The following comments refer to laboratory performance in meeting the quality control specifications outlined in the analytical method, the Manchester Laboratory Quality Assurance Manual, standard operating procedures, and professional judgment. For those tests for which the USEPA Region 10 Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met.

The conclusions presented herein are based on the information provided for the review.

Holding Time - Acceptable

The samples were received frozen on November 16, extracted December 29 and January 5 and analyzed on January 31, 2006. The holding time criteria of one year for frozen tissue and 40 days for extracts were met.

Initial Calibration - Acceptable

An initial calibration was acquired on January 31, 2006. A seven point quadratic calibration was used for 1016 and 1260. Single points were analyzed for Aroclors 1221, 1232, 1242, 1248, 1254, 1262 and 1268. All points used Coefficient of Determination > 0.995. The residual difference from calculation of the value of each calibration point against the appropriate calibration curve was <20% of the calibration point.

Continuing Calibration – Acceptable

The continuing calibration check was within the criterion of $\pm 15\%$ of the expected value for each analyte.

Blanks - Acceptable

A method blank was prepared and analyzed to evaluate the potential for laboratory contamination and the effect on sample results. The target analytes were not detected at the quantitation limit in the blank.

Surrogates - Acceptable

A solution containing decachlorobiphenyl was added as a surrogate for this analysis. All surrogate recoveries were within the expected range of 50 to 150%.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Separate aliquots of two samples, 05364201 and 05364206, were spiked in duplicate with a solution of Aroclors 1260 and 1016 and analyzed with the other samples. The MS/MSD recoveries were within the 30 to 150%, <50% RSD criteria for sample 05364201.

The recoveries for 1016 in 05364206 were also within the criteria. Once the native contribution of 1260 was subtracted from the total the calculated recovery was low to near zero. Results for Aroclor 1260 in sample 05364206 were qualified as estimated, “J”, due to the low recovery.

There were no other indications of Aroclor 1260 recovery problems from the other MS/MSD pair or the LCSs. All three aliquots were from the same jar (N4). The low recovery is likely to stem from sample homogeneity, with the native amount of Aroclor 1260 higher in the aliquot taken for analysis than in those taken for the MS/MSD.

Laboratory Control Sample - Acceptable

Data for laboratory control samples (LCS) are generated to provide information on the accuracy of the analytical method and the laboratory performance. Two blank aliquots were spiked with a solution of Aroclors 1016 and 1260. Both LCSs met the applied recovery criterion of 70-130%.

Target Compound Identification - Acceptable

PCBs were identified in most samples. Four of the samples, 05364202 through 05364205, contain PCBs at levels that are near the reporting limit and the patterns do not match any of the Aroclor standards. They have been quantitated as Aroclor 1260 and qualified as "NJ". Five of the samples, 05364206 through 05364210, contain Aroclor 1260 with a contribution from 1254 resulting in a poor pattern match. They have been reported as Aroclor 1260 qualified as estimated "J."

Compound Quantitation

The initial calibration functions were used for calculations. Reported quantitation limits were based on the initial calibration standards, the sample size used for the analysis and the final extract volume.

Five of the samples, 05364201 through 05364205, contain PCBs at levels that are near the reporting limit with patterns that do not match any of the Aroclor standards. They have been quantitated as Aroclor 1260 and qualified as tentatively identified and the quantity estimated "NJ".

Five of the samples, 05364206 through 05364210, contain PCBs that originated in Aroclor 1260 and 1254. The 1260 is higher in concentration and the samples are quantitated as Aroclor 1260 and qualified as estimated "J".

Manual Integrations - Acceptable

Manual integrations were reviewed and found to be acceptable.

Percent Lipid Determination – Acceptable

Percent lipids were determined from a portion of the extract generated for the PCB analysis. This procedure determines non-polar lipids.

Overall Assessment

All requirements for data qualifiers from the preceding sections were accumulated. Each sample data summary sheet and each compound was checked for positive or negative results. From this, the overall need for data qualifiers for each analysis was determined. In cases where more than one of the preceding sections required data qualifiers, the most restrictive qualifier has been added to the data.

In general, all unqualified data can be used without restriction. The usefulness of qualified data should be treated according to the severity of the qualifier. Should questions arise regarding the qualification of data and its relation to the usefulness, the reader is encouraged to contact Steven Reimer at the Region 10 Laboratory, phone number (360)871-8718.

LABORATORY QUALIFIER/REMARK CODE DEFINITIONS

Qualifier/ Remark Code	Definition (Codes Assigned to Values)
<	<p>Microbiology – Level of target organism present in the sample is less than detection limit. The reported value is the detection limit.</p> <p>Flash Point – The expected flash point temperature is less than the reported value.</p>
>	<p>Microbiology – Level of target organism exceeds upper limit for acceptable range of countable colonies (MF only) or exceeds MPN indices based on number of positive tubes (MPN only). The reported value is the upper limit.</p> <p>Flash Point – If the sample has a flashpoint, it is greater than the reported value.</p>
J	The identification of the analyte is acceptable; the reported value is an estimate.
JK	The identification of the analyte is acceptable; the reported value is an estimate and may be <u>biased high</u> . The actual value is expected to be less than the reported value.
JL	The identification of the analyte is acceptable; the reported value is an estimate and may be <u>biased low</u> . The actual value is expected to be greater than the reported value.
K	The identification of the analyte is acceptable; the reported value may be <u>biased high</u> . The actual value is expected to be less than the reported value.
L	The identification of the analyte is acceptable; the reported value may be <u>biased low</u> . The actual value is expected to be greater than the reported value.
N	There is presumptive evidence that the analyte is present; the analyte is reported as a tentative identification.
NJ	There is presumptive evidence that the analyte is present; the analyte is reported as a tentative identification. The reported value is an estimate.
U	The analyte was not detected at or above the reported value.
UJ	The analyte was not detected at or above the reported value. The reported value is an estimate.

Qualifier/ Remark Code	Definition (Codes With No Reported Values)
A	Absent – The target parameter was analyzed for but was not present or was undetected. <u>No value is reported with this qualification.</u>
NA	Not Applicable, the parameter was not analyzed for, or there is no analytical result for this parameter. <u>No value is reported with this qualification.</u>
P	Present at an undetermined level – The target parameter is present but not quantifiable or no quantifiable result was determined. <u>No value is reported with this qualification.</u>

Qualifier/ Remark Code	Definition (Codes With No Reported Values)
R	The presence or absence of the analyte can not be determined from the data due to severe quality control problems. The data are rejected and considered unusable. <u>No value is reported with this qualification.</u>
T	A trace of the subject parameter was present. For asbestos analysis the subject parameter was identified but at a low level that a quantifiable percentage of content is unreliable. <u>No value is reported with this qualification.</u>
TNTC	Too Numerous To Count – Any membrane where the total number of bacterial colonies exceeds 200 per membrane, or if the colonies are not distinct enough for accurate counting (i.e. confluent growth).

Qualifier/ Remark Code	Definition (Codes Assigned To Values Generated via Field or Screening Methods)
F	The associated datum was generated using field methods and/or screening methods. The identification of the analyte is acceptable and the reported value has been found to be acceptable for use.
JF	The associated datum was generated using field methods and/or screening methods. The identification of the analyte is acceptable and the reported value is an estimate.
JKF	The associated datum was generated using field methods and/or screening methods. The identification of the analyte is acceptable; the reported value is an estimate and may be <u>biased high</u> . The actual value is expected to be less than the reported value.
JLF	The associated datum was generated using field methods and/or screening methods. The identification of the analyte is acceptable; the reported value is an estimate and may be <u>biased low</u> . The actual value is expected to be greater than the reported value.
UF	The associated datum was generated using field methods and/or screening methods. The analyte was not detected at or above the reported value.
UJF	The associated datum was generated using field methods and/or screening methods. The analyte was not detected at or above the reported value. The reported value is an estimate.

Qualifier/ Remark Code	Cross Reference to Older Codes
A	UND, ND – Undetected, Not detected
NA	NAR, NAF – No analytical result, Not analyzed for
P	PNQ – Present but not quantified
R	REJ - Rejected
T	TRACE

NOTE: For any qualifier code see the QA memo or case narrative for a more detailed description of its use.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

April 15, 2006

Reply to
Attn of: **MGREPOGR**
OEA-095

MEMORANDUM

Subject: Data Validation Report for the Polychlorinated Biphenyl (PCB) Aroclor and Percent Lipid (% lipid) Analysis of Fish Tissue Samples Collected for the Phase I Upper Columbia River (UCR) Remedial Investigation/Feasibility Study (RI/FS) September 2005

From: Ginna Grepo-Grove, Senior Chemist
Technical Support Unit, OEA

To: Sally Thomas, RPM, UCR, Fish Tissue Study
USEPA, ECL

Jim Stefanoff, Project Manager, CH2MHill
Artemis Antipas, QA Manager, CH2MHill

The quality assurance (QA) review of 57 fish tissue samples collected from the above referenced site has been completed. These samples were analyzed for PCB Aroclors in accordance with the SW846 Method 8082, "Polychlorinated Biphenyls by Gas Chromatography". The analyses were performed by the USEPA Manchester Environmental Laboratory located in Port Orchard WA. The following samples were evaluated in this validation report:

Field Sample Numbers	Sample Description	Region 10 Sample Tracking Number
WE3F15	Walleye FSCA#3 Comp# 1 - 5 Fish Fillet Skin-on (R & L)	05374200
WE3F25	Walleye FSCA#3 Comp# 2 - 5 Fish Fillet Skin-on (R & L)	05374201
WE3F35	Walleye FSCA#3 Comp# 3 - 5 Fish Fillet Skin-on (R & L)	05374202
WE3F45	Walleye FSCA#3 Comp# 4 - 5 Fish Fillet Skin-on (R & L)	05374203
WE3F55	Walleye FSCA#3 Comp# 5 - 5 Fish Fillet Skin-on (R & L)	05374204
WE3O15	Walleye FSCA#3 Comp# 1 - 5 Offals	05374205
WE3O25	Walleye FSCA#3 Comp# 2 - 5 Offals	05374206
WE3O35	Walleye FSCA#3 Comp# 3 - 5 Offals	05374207
WE3O45	Walleye FSCA#3 Comp# 3 - 5 Offals	05374208
WE3O55	Walleye FSCA#3 Comp# 5 - 5 Offals	05374209

Field Sample Numbers	Sample Description	Region 10 Sample Tracking Number
WE4W15	Walleye FSCA#4 Whole body Comp #1 - 5 Fish	05374210
WE4W25	Walleye FSCA#4 Whole body Comp # 2 - 5 Fish	05374213
WE4W35	Walleye FSCA#4 Whole body Comp # 3 - 5 Fish	05374214
WE4W45	Walleye FSCA#4 Whole body Comp # 4 - 5 Fish	05374215
WE4W55	Walleye FSCA#4 Whole body Comp # 5- 5 Fish	05374216
WE6F15	Walleye FSCA#6 Comp# 1 - 5 Fish Fillet Skin-on (R & L)	05374217
WE6F25	Walleye FSCA#6 Comp# 2 - 5 Fish Fillet Skin-on (R & L)	05374218
WE6F35	Walleye FSCA#6 Comp# 3 - 5 Fish Fillet Skin-on (R & L)	05374219
WE6F45	Walleye FSCA#6 Comp# 4 - 5 Fish Fillet Skin-on (R & L)	05374220
WE6F55	Walleye FSCA#6 Comp# 5 - 5 Fish Fillet Skin-on (R & L)	05374221
WE6O15	Walleye FSCA#6 Comp# 1 -5 Offals	05374222
WE6O25	Walleye FSCA#6 Comp# 2 - 5 Offals	05374223
WE6O35	Walleye FSCA#6 Comp# 3 - 5 Offals	05374224
WE6O45	Walleye FSCA#6 Comp# 4 - Offals	05374225
WE6O55	Walleye FSCA#6 Comp# 5 - Offals	05374226
LW3W15	Lake White Fish FSCA#3 Whole body Comp# 1 - 5 Fish	05374227
LW3W55	Lake White Fish FSCA#3 Whole body Comp# 5 - 5 Fish	05374228
LW3W25	Lake White Fish FSCA#3 Whole body Comp# 2 - 5 Fish	05374229
LW3W35	Lake White Fish FSCA#3 Whole body Comp# 3 - 5 Fish	05374230
LW3W45	Lake White Fish FSCA#3 Whole body Comp# 4 - 5 Fish	05374231
LW5W25	Lake White Fish FSCA#5 Whole body Comp# 2 - 5 Fish	05374234
LW5W15	Lake White Fish FSCA#5 Whole body Comp# 1 - 5 Fish	05374235
LW5W35	Lake White Fish FSCA#5 Whole body Comp# 3 - 5 Fish	05374236
LW5W45	Lake White Fish FSCA#5 Whole body Comp# 4 - 5 Fish	05374237
LW5W55	Lake White Fish FSCA#5 Whole body Comp# 5 - 5 Fish	05374238
LW4W25	Lake White Fish FSCA#4 Whole body Comp# 2 - 5 Fish	05374239
LW4W35	Lake White Fish FSCA#4 Whole body Comp# 3 - 5 Fish	05374240
LW4W45	Lake White Fish FSCA#4 Whole body Comp# 4 - 5 Fish	05374241
LW4W55	Lake White Fish FSCA#4 Whole body Comp# 4 - 5 Fish	05374242
LW6W13	Lake White Fish FSCA#6 Whole body Comp# 1 - 3 Fish	05374243
LW4W15	Lake White Fish FSCA#4 Whole body Comp# 1 - 5 Fish	05374244
RW1F15	Rainbow Trout Wild FSCA#1 Comp#1 -5 Fish Fillets Skin-on (L &R)	05374245
RW1F25	Rainbow Trout Wild FSCA#1 Comp#2 -5 Fish Fillets Skin-on (L &R)	05374246
RW1F35	Rainbow Trout Wild FSCA#1 Comp#3 -5 Fish Fillets Skin-on (L &R)	05374247
RW1F45	Rainbow Trout Wild FSCA#1 Comp#4 -5 Fish Fillets Skin-on (L &R)	05374248
RW1F55	Rainbow Trout Wild FSCA#1 Comp#5 -5 Fish Fillets Skin-on (L &R)	05374249
RW1F65	Rainbow Trout Wild FSCA#1 Comp#6 -5 Fish Fillets Skin-on (L &R) *	05374250
RW1O15	Rainbow Trout Wild FSCA#1 Comp# 1 -5 Offals	05374251
RW1O25	Rainbow Trout Wild FSCA#1 Comp# 2 -5 Offals	05374252
RW1O35	Rainbow Trout Wild FSCA#1 Comp# 3 -5 Offals	05374253
RW1O45	Rainbow Trout Wild FSCA#1 Comp# 4 -5 Offals	05374254
RW1O55	Rainbow Trout Wild FSCA #1 Comp# 1 - 5 Offals	05374255
RW1O65	Rainbow Trout Wild FSCA #1 Comp# 6 - 5 Offals *	05374256
MW1W45	Mountain Whitefish FSCA#1 Whole body Comp# 4 - 5 Fish	05374257
MW1W55	Mountain Whitefish FSCA#1 Whole body Comp# 5 - 5 Fish	05374258
MW1W65	Mountain Whitefish FSCA#1 Whole body Comp# 6 - 5 Fish *	05374261
MW1W75	Mountain Whitefish FSCA#1 Whole body Comp# 7 - 5 Fish *	05374262
MW1W15	Mountain Whitefish FSCA#1 Whole body Comp# 1 - 5 Fish	05374263
MW1W25	Mountain Whitefish FSCA#1 Whole body Comp# 2 - 5 Fish	05374264
MW1W35	Mountain Whitefish FSCA#1 Whole body Comp# 3 - 5 Fish	05374265

* Field Duplicate/Triplicate

DATA QUALIFICATIONS

The following comments refer to the laboratory's performance in meeting the Quality Control specifications outlined in the Phase 1 Fish Tissue Sampling Quality Assurance Project Plan (QAPP) for the Upper Columbia River Site CERCLA RI/FS, the analytical method SW846 Method 8082, the MEL's Standard Operating Procedure (SOP) #Or_Fish3541 and the MEL SOP for lipid determination.

The conclusions presented herein are based on the information provided for the review.

Field Sample Collection

The fish tissue sample collection was accomplished through a multi-agency/tribal effort with the CH2MHill team as the overall lead. Sample vessels and vessel operators were provided by the following tribal and federal agencies under an interagency or sub-contracting agreement with EPA and/or CH2MHill: Spokane Tribe of Indians, Confederated Tribes of the Colville Reservation, US Fish and Wildlife Services and the USEPA Investigation and Engineering Unit, of the Office of Environmental Assessment.

The sample collection dates were based on the fish availability and fish species' spawning season. There were two sample collection events conducted, first one was conducted in September 2005 and the second one was in October 2005. The fish species that were collected from the designated fish sample collection areas (FSCA 1 -6) were Walleye (*Sander vitreus*) Rainbow trout (*Oncorhynchus mykiss*), Lake white fish (*Coregonus clupeaformis*), Large-scale sucker (*Catostomas macrocheilus*), and Burbot (*Lota lota*). Long-nose suckers and Mountain whitefish were not originally listed in the QAPP as target fish species but were also collected and added to the target fish species due to their availability in the FSCAs. The mountain white fish were analyzed while the long-nose suckers were archived. The rainbow trout samples were grouped into three categories – wild, hatchery and mixed wild and hatchery. Only the wild and hatchery rainbow trouts were analyzed for the compounds of concern. The mixed wild and hatchery rainbow trouts were archived for future analysis, if needed.

The fish samples were generally collected using gill nets, electro-fishing, burbot traps and angling, if necessary. The field sample collection process was audited by the project's EPA and CH2MHill QA Managers. There were no significant problems encountered during sample collection, on-site processing, sampling documentation and sample shipment.

Sample Processing and Chain-of-Custody Documentation

CH2MHill set-up a trailer dedicated for the on-site fish sample processing which included visual inspection of the fish, sex determination, conducting field measurements (fish length and weight) and otolith removal. Otoliths are then later sent to the State Fish and Wildlife for fish age determination. All of the field forms generated for these measurements and determination were evaluated and cross – checked with the homogenization forms and chain-of-custody (COC) documentation. All of the field measurements, field sampling documentation (COCs) and sample preservation (freezing to -20C) were conducted by CH2MHill within 24 hours of sample collection.

Frozen whole fish samples were shipped to CH2MHill laboratory - Applied Science Laboratory (ASL) located in Corvallis, OR for filleting (if needed), homogenization, compositing, aliquot distribution and storage. There were four types of tissues prepared and analyzed for the compounds of potential concern (COPCs) for the sites, namely: fillets with skin-on, offals (remaining tissue, internal organs and fish

bones after filleting, guts (for large scale sucker only) and whole body (includes fish head, skin and entrails).

As specified in the EPA approved site QAPP, the following tissue types and homogenates were prepared by ASL and shipped to USEPA Manchester Environmental Laboratory (MEL) for subsequent PCB Aroclor, metals, percent lipids and speciated arsenic analyses and/or archival:

- Walleye – fillets and offals at three FSCAs and whole body composites from three FSCAs
- Rainbow trout – fillets and offals at three FSCAs and whole body composites from three FSCAs
- Lake whitefish – whole body composites
- Mountain whitefish – whole body composites
- Large scale sucker – whole body; guts/internal organs composites
- Burbot – whole body composites
- Long-nose suckers – whole body composites

Sample Homogenization and Compositing

Fish samples from each sample location were individually homogenized at ASL. The fish samples were grinded using a commercial grade stainless steel blender/grinder (Robo-Coupe Blixer 6) with liquid nitrogen. Equal amounts of homogenized whole body, fillet or offal tissue samples were mixed and composited to form a single sample. The homogenization forms and the resulting fish sample composites were evaluated by this reviewer. There were no discrepancies noted between the sample collection forms, homogenization forms and the sample composite COCs. Fillet and whole body samples included the fish skin. Care was taken to prevent cross-contamination between sample homogenates. Prior to the start of the project samples, homogenization process was audited by the project's EPA and CH2MHill QA Managers. To monitor processing cross-contamination, proof blanks were collected at the QAPP specified frequency and sent to the Contract Laboratory program (CLP) for the analysis of the project target compound.

Deviation from the QAPP: In a mock sample processing and homogenization conducted during the EPA's and CH2MHill's QA lab audit, it was found out that otoliths were very hard to remove when the fish samples were already frozen. In addition, subjecting the fish to freezing and defrosting ruptures the internal organs, make the fish muscles mushy and thus made the separation of fillets from the offals quite a challenge.

To avoid cross-contamination of the fish tissue samples with the offals and to better preserve the otoliths, it was agreed by the project team that the removal of otolith will be conducted on-site after field measurements and before sample preservation (freezing to -20C) and if bench space and resources will allow, filleting of fish samples will also be performed on-site prior to freezing the samples.

Sample Receipt and Storage

All of the sample homogenates were received frozen and intact at MEL from ASL. The remaining whole fish samples (un-homogenized) were also sent to MEL for archiving and maybe future chemical analysis, if needed. After inspection, inventory and logging-in, the sample homogenates and un-homogenized fish samples were stored in a freezer at -20C. The fish samples remained frozen at -20C while waiting for extraction and analysis. The temperature of the freezer used for sample storage is monitored 24 hours by MEL. The integrity of the fish samples and homogenates were maintained by MEL while on storage, during and after extraction and analysis.

COC Corrective Action

There were two COC corrective actions initiated by MEL to reconcile discrepancies between the regional tracking sample numbers and the field sample numbers for a few of the samples in this sample delivery group. The corrective actions and resolutions were sufficiently documented and new regional tracking sample numbers were issued by ALS to correct the regional sample number duplication.

Holding Times - Acceptable

All of the fish sample analyses met the project-specified extraction and analytical holding times of 6 months from the date of sample collection. None of the data were qualified on this basis.

The list of samples, cross-referenced to the fish species, station locations, and the dates of sample collection, VTSR at the lab, extraction, extract clean-up and analysis dates are listed in Table 1 at the end of this report.

Sample Preparation and Clean-up

All of the samples were extracted following the technical specifications of the analytical methods used. Prior to acid clean-up, 10% of the primary extracts were taken for % lipid determination. The rest of the primary extracts went through concentrated sulfuric acid clean-up (SW846 Method 3665) to isolate the PCBs and remove most of the organic material that would interfere with the analysis. A 35% or 70% fraction of the original extract (depending on the amount extracted) was concentrated to 1.0 ml and passed through florisil cartridge clean-up (SW846 Method 3620) prior to GC analysis.

All of the analysts involved in sample extraction, extract clean-up and analysis of the samples in this data package performed an acceptable initial demonstration of capability (IDOC) studies prior to handling the samples.

In addition, the efficiency of the sample extraction procedure, clean-up and analytical processes were also monitored through the routine analysis of in-house Quality Control sample analyses and incorporation of routine-in-house QC checks (recoveries of the surrogate standards and the spike compounds in the laboratory control samples and matrix spike and duplicate analyses).

Instrument Performance Checks - Acceptable

A dual-column GC analyses was used during the PCB Aroclor analysis. The designated primary column used in the quantitation of target compounds was Restek's CLP2 in all analytical sequences. The secondary, confirmatory column was Restek's CLP1. Baseline and retention time shifts were monitored

and the instrument remained stable throughout the course of the analyses. None of the data were qualified on this basis.

Initial Calibrations - Acceptable

Five ICALs using 5-concentration levels of Aroclors 1016 and 1260 and one ICAL using 5-concentration levels of Aroclor 1254 were performed and used during the analysis of the samples listed in this validation report. A single-point concentration was analyzed for Aroclors 1221, 1232, 1242, 1248, 1254, 1262 and 1268 with each 1016/1260 ICAL. The frequency of analysis and the regression coefficients of the 5 major peaks used in the Aroclor identification and quantitation were all >0.995 for the primary column. Some of the peaks of from the secondary column did not meet the criteria of $r>0.995$, however, since this column was only used for confirmatory analyses, none of the data were qualified on this basis.

Continuing Calibrations - Acceptable

A mid-point concentration Aroclor 1016/1260 and/or Aroclor 1254 were analyzed for continuing calibration verification (CCVs) checks. The CCVs met the criteria for the frequency of analysis, the percent differences (%D) of the daily calibration factors (CFs) as compared to the mean CFs from the ICALs and the retention time shifts. None of the data were qualified on this basis.

Quantitation and Reporting Limits (QLs & RLs)

The QLs which are based on the lowest concentration level of the Aroclors in the ICALs, the amount of sample extracted and the final extract volume were about twice the project analytical concentration goals (ACGs) listed in Table 2-3 of the QAPP. Aroclor detections at concentrations $<QLs$, however, were reported by MEL with an estimated, "J", qualifier. All of the target compounds detected in the samples were calculated off the primary column using the CFs from the applicable ICALs.

Due to the low level concentrations of Aroclors 1260 and 1254 native to most of the fish samples and interferences of other organic materials causing baseline noise and drifts, the RLs of most of the non-detected Aroclors in the samples were elevated to about 10 times the QLs.

The concentrations of the Aroclors 1260 mixed with 1254 detected in most of the samples were qualified estimated, "J", due to the co-eluting peaks used in the calculations.

Laboratory Method Blanks - Acceptable

The frequency of analysis of laboratory blank was met. All of the method blanks associated with the fish sample extraction, clean-up and analyses were clean. None of the data were qualified on this basis.

Homogenization Proof Blanks – Acceptable

A composite of final rinses during the decontamination of the Robo-Coupe Blixer 6 used for fish tissue and offal homogenization were collected. An aliquot of the composite rinses called "proof blanks" were collected every three days and shipped to the Superfund Contract Laboratory Program (CLP) labs. A total of 17 proof blanks were shipped to A4 Laboratory, Inc. of Woodlands TX and were analyzed for semi-volatile organic compounds (SVOCs), pesticides and PCB Aroclors. No PCB Aroclors were detected in any of the proof blanks. None of the fish tissue sample results were qualified on this basis.

Analytical Sequence - Acceptable

All of the standards, blanks, samples and QC samples were analyzed in accordance with the method specified analytical sequence. All of the analytical sequences were also bracketed by the continuing calibration check standards. None of the data were qualified on this basis.

Surrogate Recoveries – Acceptable

Decachlorobiphenyl (DCB) was used as the surrogate standard during PCB Aroclor analyses. Known concentrations of DCB were added to all samples and QC samples to monitor efficiency during sample extraction, clean-up and analysis. The DCB surrogate recoveries for all samples, QC samples and dilution runs were acceptable (50-150%). The DCB retention time shifts were also within the established retention time windows. None of the data were qualified on this basis.

Matrix Spike and Matrix Spike Duplicate (MS/MSD) Analysis

Samples WE4W15 (05374210), LW3W55 (05374228), LW5W15 (05374235), RW1F15 (05374245) and MW1W45 (05374257) were the designated QC samples and analyzed for MS and MSD. The frequency of analysis of MS/MSD (about 10%) was met. Known concentrations of Aroclors 1016 and 1260 were spiked into the QC samples and went through the same extraction, clean-up and analytical procedures as the project samples.

The MS/MSD recoveries and relative percent differences (RPDs) criteria (30-150% and 50%, respectively) of the Aroclor 1016 and 1260 were met for all QC samples with the following exceptions:

- The Aroclor 1260 MS/MSD recoveries in the QC samples WE4W15 (05374210) and MW1W45 (05374257), however, did not meet the acceptance criteria due to the Aroclor 1254/1260 mixture native to the samples interfering with the calculations. The amount of 1254/1260 in sample 05374257 was greater than 4 times the amount of spike. The data associated with this QC sample were not qualified. Since the detected Aroclor (1254/1260) in sample 05374210 was already qualified due to peak co-elutions, no further qualification is warranted due to MS/MSD recoveries.
- The recovery and relative percent difference (RPD) criteria for the Aroclors 1016 and 1260 in the QC samples LW3W55 MS and MSD met the acceptance criteria. None of the data associated with these QC samples were qualified on this basis.

The lipid analytical method does not include MS/MSD analyses.

Laboratory Control Sample and Duplicate Analyses (LCS/LCSD) – Acceptable

Four sets of LCS and LCSD were prepared and analyzed with the samples. For LCS and LCSD, the hydromatrix extraction media was spiked with known concentrations of Aroclors 1016 and 1260. The frequency of analysis, recovery (70-130%) and RPD (50%) criteria were met for all LCS and LCSD analyses. None of the PCB Aroclor sample data were qualified on this basis. There were no LCS/LCSD runs for lipid determination.

Analytical Duplicate Analyses - Acceptable

Samples LW4W45 (05374241), RW1O15 (05374251) and MW1W65 (05374261) were analyzed in duplicates. A mixture of Aroclors 1254 and 1260 were detected during the initial and duplicate analysis of all three QC samples. The RPDs between the concentrations of 1254/1260 detected in the original and duplicate sample runs ranged from 3-21%. The RPDs of the % lipids calculated the original and duplicate %lipid runs ranged form 9 – 24%. All of the RPDs were acceptable and within the QC limits of 50%. None of the data were qualified on this basis.

Field Duplicate Sample Analyses – Acceptable

RW1F55 (05374249) and RW1F65 (05374250) are field duplicate samples submitted blind to MEL. A mixture of Aroclors 1260 and 1254 were identified in both samples at 21.6 and 19.1 ug/Kg estimated concentrations, respectively. The % lipids was 4.9% and 4.3% (RPD=6.7%). There was not much variability between the lipids and Aroclor duplicate values. None of the wild trout fillet Aroclor data was qualified on this basis.

MW1W45 (05374257), MW1W65 (05374261) and MW1W75 (05374262) are field triplicate samples submitted blind to MEL. Aroclors 1260/1254 was detected in all three samples at estimated concentrations of 65.5, 37.6 and 53.1 ug/Kg, respectively. The %RSD was 27% for the Aroclors and 16.6% for the lipids. There was not much variability between the lipids and Aroclor duplicate values. None of the mountain whitefish PCB data were qualified on this basis.

Compound Identification

Aroclors 1254 and 1260 were evidently present in all of the samples. The chromatogram overlays for the Aroclors 1260, 1254 and sample runs were mapped and evaluated by this reviewer. In instances where concentrations of Aroclors 1254 and 1260 were very low that only the major co-eluting peaks are quantifiable, the Aroclor detections were reported by the analysts as a combined Aroclor 1254/1260 with an estimated qualification. Where Aroclor 1254 and 1260 peaks could be isolated, separate concentrations for each Aroclor was reported. All of the extracts went through additional clean-ups when needed. Other than the usual low-level baseline noise, there were no other interferences observed with the chromatograms. All of the Aroclors reported somewhat matched the standard fingerprint patterns. All of the Aroclors identified were verified and are acceptable.

Laboratory Contact

The laboratory was not contacted for this review.

Overall Assessment

All of the samples were analyzed in accordance with the method specifications. There were no significant problems found with the data. The data, as qualified, are acceptable and can be used for all purposes.

Data Qualifiers		
	U	The analyte was not detected at or above the reported numeric result.
	J	The analyte was positively identified. The associated numerical result is an estimate.
	UJ	The analyte was not detected at or above the reported estimated result. The associated numerical value is an estimate of the quantitation limit of the analyte in this sample.
	R	The data are unusable for all purposes.
	N	There is evidence the analyte is present in this sample.
	JN	There is evidence that the analyte is present. The associated numerical result is an estimate.

TABLE 1- SUMMARY OF HOLDING TIMES

Sample Number	Region 10 Sample Tracking Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	Extraction Date	% Lipid Analysis Date	PCB Analysis Date
WE3F15	05374200	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3F25	05374201	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3F35	05374202	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3F45	05374203	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3F55	05374204	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3O15	05374205	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3O25	05374206	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE3O35	05374207	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE3O45	05374208	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE3O55	05374209	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE4W15	05374210	09/10/05	09/14/05	11/09/05	11/16/05	01/09/06	01/09/06	01/23/06
WE4W25	05374213	09/12/05	09/14/05	11/09/05	11/16/05	01/09/06	01/09/06	01/23/06
WE4W35	05374214	09/12/05	09/14/05	11/09/05	11/16/05	01/09/06	01/09/06	01/31/06
WE4W45	05374215	09/12/05	09/14/05	11/09/05	11/16/05	01/09/06	01/09/06	01/31/06
WE4W55	05374216	09/12/05	09/14/05	11/09/05	11/16/05	01/09/06	01/09/06	01/23/06
WE6F15	05374217	09/14/05	09/15/05	11/11/05	11/16/05	01/09/06	01/09/06	01/23/06
WE6F25	05374218	09/14/05	09/15/05	11/11/05	11/16/05	01/10/06	01/10/06	02/16/06
WE6F35	05374219	09/14/05	09/15/05	11/11/05	11/16/05	01/10/06	01/10/06	02/16/06
WE6F45	05374220	09/14/05	09/15/05	11/13/05	11/16/05	01/10/06	01/10/06	02/16/06
WE6F55	05374221	09/14/05	09/15/05	11/13/05	11/16/05	01/10/06	01/10/06	02/16/06
WE6O15	05374222	09/14/05	09/15/05	11/11/05	11/16/05	01/10/06	01/10/06	02/17/06
WE6O25	05374223	09/14/05	09/15/05	11/11/05	11/16/05	01/10/06	01/10/06	02/17/06
WE6O35	05374224	09/14/05	09/15/05	11/11/05	11/16/05	01/10/06	01/10/06	02/17/06
WE6O45	05374225	09/14/05	09/15/05	11/13/05	11/16/05	01/10/06	01/10/06	02/17/06
WE6O55	05374226	09/14/05	09/15/05	11/13/05	11/16/05	01/10/06	01/10/06	02/17/06
LW3W15	05374227	09/13/05	09/14/05	11/22/05	12/08/05	01/10/06	01/10/06	02/16/06
LW3W55	05374228	09/13/05	09/14/05	11/22/05	12/08/05	01/17/06	01/17/06	02/16/06
LW3W25	05374229	09/14/05	09/15/05	11/21/05	12/08/05	01/17/06	01/17/06	02/16/06
LW3W35	05374230	09/14/05	09/15/05	10/14/05	12/08/05	01/17/06	01/17/06	02/17/06
LW3W45	05374231	09/14/05	09/15/05	11/21/05	12/08/05	01/17/06	01/17/06	02/17/06
LW3W75	05374233	09/14/05	09/15/05	11/21/05	12/08/05	01/17/06	01/17/06	02/17/06
LW5W15	05374235	09/15/05	09/16/05	11/16/05	12/08/05	01/31/06	01/31/06	03/07/06
LW5W35	05374236	09/15/05	09/16/05	11/16/05	12/08/05	01/31/06	01/31/06	03/07/06
LW5W45	05374237	09/15/05	09/16/05	11/22/05	12/08/05	01/31/06	01/31/06	03/07/06
LW5W55	05374238	09/15/05	09/16/05	11/28/05	12/08/05	01/31/06	01/31/06	03/07/06

Sample Number	Region 10 Sample Tracking Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	Extraction Date	% Lipid Analysis Date	PCB Analysis Date
LW4W25	05374239	09/17/05	09/18/05	11/22/05	12/08/05	01/31/06	01/31/06	03/07/06
LW4W35	05374240	09/17/05	09/18/05	11/22/05	12/08/05	01/31/06	01/31/06	03/08/06
LW4W45	05374241	09/17/05	09/18/05	11/28/05	12/08/05	01/31/06	01/31/06	03/08/06
LW4W55	05374242	09/17/05	09/18/05	11/28/05	12/08/05	02/01/06	02/01/06	03/08/06
LW6W13	05374243	09/17/05	09/18/05	11/18/05	12/08/05	02/01/06	02/01/06	03/08/06
LW4W15	05374244	09/17/05	09/18/05	11/21/05	12/08/05	02/01/06	02/01/06	03/08/06
RW1F15	05374245	09/13/05	09/14/05	12/20/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1F25	05374246	09/13/05	09/14/05	12/22/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1F35	05374247	09/13/05	09/14/05	12/22/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1F45	05374248	09/13/05	09/14/05	12/23/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1F55	05374249	09/13/05	09/14/05	12/21/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1F65	05374250	09/13/05	09/14/05	12/21/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1O15	05374251	09/13/05	09/14/05	12/20/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1O25	05374252	09/13/05	09/14/05	12/22/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1O35	05374253	09/13/05	09/14/05	12/22/05	01/06/06	02/07/06	02/07/06	03/08/06
RW1O45	05374254	09/13/05	09/14/05	12/23/05	01/06/06	02/07/06	02/07/06	03/08/06
RW1O55	05374255	09/13/05	10/22/05	12/21/05	01/06/06	02/07/06	02/07/06	03/08/06
RW1O65	05374256	09/13/05	10/22/05	12/21/05	01/06/06	02/07/06	02/07/06	03/08/06
MW1W45	05374257	09/13/05	09/14/05	11/29/05	01/12/06	02/07/06	02/07/06	03/08/06
MW1W55	05374258	09/13/05	09/14/05	11/30/05	01/12/06	02/07/06	02/07/06	03/08/06
MW1W65	05374261	09/14/05	09/14/05	11/29/05	01/12/06	02/15/06	02/15/06	03/08/06
MW1W75	05374262	09/43/05	09/14/05	11/29/05	01/12/06	02/15/06	02/15/06	03/08/06
MW1W15	05374263	09/13/05	09/14/05	11/30/05	01/12/06	02/15/06	02/15/06	03/08/06
MW1W25	05374264	09/13/05	09/14/05	11/29/05	01/12/06	02/15/06	02/15/06	03/08/06
MW1W35	05374265	09/13/05	09/14/05	11/30/05	01/12/06	02/15/06	02/15/06	03/08/06
LW4W45 Dup	05374241 D	09/17/05	09/18/05	11/28/05	12/08/05	02/15/06	02/15/06	03/08/06
RW1O15 Dup	05374251 D	09/13/05	09/14/05	12/20/05	01/06/06	02/15/06	02/15/06	03/08/06
MW1W65 Dup	05374261 D	09/14/05	09/14/05	11/29/05	01/12/06	02/15/06	02/15/06	03/08/06



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 15, 2006

Reply to
Attn of: **MGREPOGR**
OEA-095

MEMORANDUM

Subject: Data Validation Report for the Polychlorinated Biphenyl (PCB) Aroclor and Percent Lipid (% lipid) Analysis of Fish Tissue Samples Collected for the Phase I Upper Columbia River (UCR) Remedial Investigation/Feasibility Study (RI/FS) September 2005

From: Ginna Grepo-Grove, Senior Chemist
Technical Support Unit, OEA

To: Sally Thomas, RPM, UCR, Fish Tissue Study
USEPA, ECL

CC: Marc Stifelman, Human Health Risk Assessment, USEPA, OEA
Burt Shephard, Ecological Risk Assessment, USEPA, OEA
Jim Stefanoff, Project Manager, CH2MHill
Artemis Antipas, QA Manager, CH2MHill

The quality assurance (QA) review of 198 fish tissue samples collected from the above referenced site has been completed. These samples were analyzed for PCB Aroclors in accordance with the SW846 Method 8082, "Polychlorinated Biphenyls by Gas Chromatography". The analyses were performed by the USEPA Manchester Environmental Laboratory located in Port Orchard, WA. The following samples were evaluated in this validation report:

Field Sample Numbers	Sample Description	Region 10 Sample Tracking Number
WE1F15	Walleye FSCA#1 Comp# 1 - 5 Fish Fillet Skin-on (R & L)	05364201
WE1F25	Walleye FSCA#1 Comp# 2 - 5 Fish Fillet Skin-on (R & L)	05364202
WE1F35	Walleye FSCA#1 Comp# 3 - 5 Fish Fillet Skin-on (R & L)	05364203
WE1F45	Walleye FSCA#1 Comp# 4 - 5 Fish Fillet Skin-on (R & L)	05364204

Field Sample Numbers	Sample Description	Region 10 Sample Tracking Number
WE1F55	Walleye FSCA#1 Comp# 5 - 5 Fish Fillet Skin-on (R & L)	05364205
WE1O15	Walleye FSCA#1 Comp# 1 - 5 Offals	05364206
WE1O25	Walleye FSCA#1 Comp# 2 - 5 Offals	05364207
WE1O35	Walleye FSCA#1 Comp# 3 - 5 Offals	05364208
WE1O45	Walleye FSCA#1 Comp# 4 - 5 Offals	05364209
WE1O55	Walleye FSCA#1 Comp# 5 - 5 Offals	05364210
LS1W25	Lake White Fish FSCA#1 Whole body Comp# 2 - 5 Fish	05364221
LS2W35	Lake White Fish FSCA#1 Whole body Comp# 3 - 5 Fish	05364222
WE3F15	Walleye FSCA#3 Comp# 1 - 5 Fish Fillet Skin-on (R & L)	05374200
WE3F25	Walleye FSCA#3 Comp# 2 - 5 Fish Fillet Skin-on (R & L)	05374201
WE3F35	Walleye FSCA#3 Comp# 3 - 5 Fish Fillet Skin-on (R & L)	05374202
WE3F45	Walleye FSCA#3 Comp# 4 - 5 Fish Fillet Skin-on (R & L)	05374203
WE3F55	Walleye FSCA#3 Comp# 5 - 5 Fish Fillet Skin-on (R & L)	05374204
WE3O15	Walleye FSCA#3 Comp# 1 - 5 Offals	05374205
WE3O25	Walleye FSCA#3 Comp# 2 - 5 Offals	05374206
WE3O35	Walleye FSCA#3 Comp# 3 - 5 Offals	05374207
WE3O45	Walleye FSCA#3 Comp# 3 - 5 Offals	05374208
WE3O55	Walleye FSCA#3 Comp# 5 - 5 Offals	05374209
WE4W15	Walleye FSCA#4 Whole body Comp #1 - 5 Fish	05374210
WE4W25	Walleye FSCA#4 Whole body Comp # 2 - 5 Fish	05374213
WE4W35	Walleye FSCA#4 Whole body Comp # 3 - 5 Fish	05374214
WE4W45	Walleye FSCA#4 Whole body Comp # 4 - 5 Fish	05374215
WE4W55	Walleye FSCA#4 Whole body Comp # 5- 5 Fish	05374216
WE6F15	Walleye FSCA#6 Comp# 1 - 5 Fish Fillet Skin-on (R & L)	05374217
WE6F25	Walleye FSCA#6 Comp# 2 - 5 Fish Fillet Skin-on (R & L)	05374218
WE6F35	Walleye FSCA#6 Comp# 3 - 5 Fish Fillet Skin-on (R & L)	05374219
WE6F45	Walleye FSCA#6 Comp# 4 - 5 Fish Fillet Skin-on (R & L)	05374220
WE6F55	Walleye FSCA#6 Comp# 5 - 5 Fish Fillet Skin-on (R & L)	05374221
WE6O15	Walleye FSCA#6 Comp# 1 - 5 Offals	05374222
WE6O25	Walleye FSCA#6 Comp# 2 - 5 Offals	05374223
WE6O35	Walleye FSCA#6 Comp# 3 - 5 Offals	05374224
WE6O45	Walleye FSCA#6 Comp# 4 - Offals	05374225
WE6O55	Walleye FSCA#6 Comp# 5 - Offals	05374226
LW3W15	Lake White Fish FSCA#3 Whole body Comp# 1 - 5 Fish	05374227
LW3W55	Lake White Fish FSCA#3 Whole body Comp# 5 - 5 Fish	05374228
LW3W25	Lake White Fish FSCA#3 Whole body Comp# 2 - 5 Fish	05374229
LW3W35	Lake White Fish FSCA#3 Whole body Comp# 3 - 5 Fish	05374230
LW3W45	Lake White Fish FSCA#3 Whole body Comp# 4 - 5 Fish	05374231
RH5W65	Rainbow Trout Hatchery FSCA #5 Whole body Comp# 6 - 5 Fish*	05374232
RH5W75	Rainbow Trout Hatchery FSCA #5 Whole body Comp# 7 - 5 Fish*	05374233
LW5W25	Lake White Fish FSCA#5 Whole body Comp# 2 - 5 Fish	05374234
LW5W15	Lake White Fish FSCA#5 Whole body Comp# 1 - 5 Fish	05374235
LW5W35	Lake White Fish FSCA#5 Whole body Comp# 3 - 5 Fish	05374236
LW5W45	Lake White Fish FSCA#5 Whole body Comp# 4 - 5 Fish	05374237
LW5W55	Lake White Fish FSCA#5 Whole body Comp# 5 - 5 Fish	05374238

Field Sample Numbers	Sample Description	Region 10 Sample Tracking Number
LW4W25	Lake White Fish FSCA#4 Whole body Comp# 2 - 5 Fish	05374239
LW4W35	Lake White Fish FSCA#4 Whole body Comp# 3 - 5 Fish	05374240
LW4W45	Lake White Fish FSCA#4 Whole body Comp# 4 - 5 Fish	05374241
LW4W55	Lake White Fish FSCA#4 Whole body Comp# 4 - 5 Fish	05374242
LW6W13	Lake White Fish FSCA#6 Whole body Comp# 1 - 3 Fish	05374243
LW4W15	Lake White Fish FSCA#4 Whole body Comp# 1 - 5 Fish	05374244
RW1F15	Rainbow Trout Wild FSCA#1 Comp#1 -5 Fish Fillets Skin-on (L &R)	05374245
RW1F25	Rainbow Trout Wild FSCA#1 Comp#2 -5 Fish Fillets Skin-on (L &R)	05374246
RW1F35	Rainbow Trout Wild FSCA#1 Comp#3 -5 Fish Fillets Skin-on (L &R)	05374247
RW1F45	Rainbow Trout Wild FSCA#1 Comp#4 -5 Fish Fillets Skin-on (L &R)	05374248
RW1F55	Rainbow Trout Wild FSCA#1 Comp#5 -5 Fish Fillets Skin-on (L &R) *	05374249
RW1F65	Rainbow Trout Wild FSCA#1 Comp#6 -5 Fish Fillets Skin-on (L &R) *	05374250
RW1O15	Rainbow Trout Wild FSCA#1 Comp# 1 -5 Offals	05374251
RW1O25	Rainbow Trout Wild FSCA#1 Comp# 2 -5 Offals	05374252
RW1O35	Rainbow Trout Wild FSCA#1 Comp# 3 -5 Offals	05374253
RW1O45	Rainbow Trout Wild FSCA#1 Comp# 4 -5 Offals	05374254
RW1O55	Rainbow Trout Wild FSCA #1 Comp# 1 - 5 Offals *	05374255
RW1O65	Rainbow Trout Wild FSCA #1 Comp# 6 - 5 Offals *	05374256
MW1W45	Mountain Whitefish FSCA#1 Whole body Comp# 4 - 5 Fish	05374257
MW1W55	Mountain Whitefish FSCA#1 Whole body Comp# 5 - 5 Fish	05374258
MW1W65	Mountain Whitefish FSCA#1 Whole body Comp# 6 - 5 Fish *	05374261
MW1W75	Mountain Whitefish FSCA#1 Whole body Comp# 7 - 5 Fish *	05374262
MW1W15	Mountain Whitefish FSCA#1 Whole body Comp# 1 - 5 Fish	05374263
MW1W25	Mountain Whitefish FSCA#1 Whole body Comp# 2 - 5 Fish	05374264
MW1W35	Mountain Whitefish FSCA#1 Whole body Comp# 3 - 5 Fish	05374265
WE2W15	Walleye FSCA#2 Whole body Comp# 1 - 5 fish	05414001
WE2W25	Walleye FSCA#2 Whole body Comp# 2 - 5 fish	05414002
WE2W35	Walleye FSCA#2 Whole body Comp# 3 - 5 fish	05414003
WE2W45	Walleye FSCA#2 Whole body Comp# 4 - 5 fish	05414004
WE2W55	Walleye FSCA#2 Whole body Comp# 5 - 5 fish*	05414005
WE2W65	Walleye FSCA#2 Whole body Comp# 6 - 5 fish*	05414006
WE2W75	Walleye FSCA#2 Whole body Comp# 7 - 5 fish*	05414007
RW2W53	Rainbow Trout Wild FSCA #2 Whole body Comp# 5 - 3 Fish	05414008
LS2W15	Large Scale Sucker FSCA# 2 Whole body Comp# 1 - 5 Fish	05414009
LS2W25	Large Scale Sucker FSCA# 2 Whole body Comp# 2 - 5 Fish *	05414010
LS2W35	Large Scale Sucker FSCA# 2 Whole body Comp# 3 - 5 Fish	05414011
LS2W45	Large Scale Sucker FSCA# 2 Whole body Comp# 4 - 5 Fish	05414012
LS2W65	Large Scale Sucker FSCA# 2 Whole body Comp# 6 - 5 Fish *	05414013
LS2W75	Large Scale Sucker FSCA# 3 Whole body Comp# 1 - 5 Fish *	05414014
LS3W15	Large Scale Sucker FSCA# 3 Whole body Comp# 2 - 5 Fish	05414015
LS3W25	Large Scale Sucker FSCA# 3 Whole body Comp# 3 - 5 Fish	05414016
LS3W35	Large Scale Sucker FSCA# 3 Whole body Comp# 3 - 5 Fish	05414017
LS3W55	Large Scale Sucker FSCA# 3 Whole body Comp# 5 - 5 Fish	05414018
LS4W25	Large Scale Sucker FSCA# 4 Whole body Comp# 2 - 5 Fish	05414019
WE5W15	Walleye FSCA#5 Whole body Comp# 1 - 5 fish	05424001

Field Sample Numbers	Sample Description	Region 10 Sample Tracking Number
WE5W25	Walleye FSCA#5 Whole body Comp# 2 - 5 fish	05424002
WE5W35	Walleye FSCA#5 Whole body Comp# 3 -5 fish	05424005
WE6W65	Walleye FSCA#6 Whole body Comp# 6 - 5 fish	05424006
WE6W75	Walleye FSCA#6 Whole body Comp# 7 - 5 fish	05424009
LW2W15	Lake Whitefish FSCA #2 Whole Body Comp #1 – 5 fish	05424010
LW2W25	Lake Whitefish FSCA #2 Whole Body Comp #2 – 5 fish	05424011
LW2W45	Lake Whitefish FSCA #2 Whole Body Comp #4 – 5 fish	05424012
LW2W55	Lake Whitefish FSCA #2 Whole Body Comp #5 – 5 fish	05424013
LW2W35	Lake Whitefish FSCA #2 Whole Body Comp #31 – 5 fish	05424014
LW6W23	Lake Whitefish FSCA #6 Whole Body Comp #2 – 3 fish	05424015
RH3F15	Rainbow Trout Hatchery FSCA #3 Fillet (R& L – skin on)# Comp #1 -5 Fish	05424016
RH3F25	Rainbow Trout Hatchery FSCA #3 Fillet (R& L – skin on)# Comp #2 -5 Fish	05424017
RH3F35	Rainbow Trout Hatchery FSCA #3 Fillet (R& L – skin on)# Comp #3 -5 Fish	05424018
RH3O15	Rainbow Trout Hatchery FSCA #3 Offal Comp #1 – 5 Fish	05424019
RH3O25	Rainbow Trout Hatchery FSCA #3 Offal Comp #2 – 5 Fish	05424020
RH3O35	Rainbow Trout Hatchery FSCA #3 Offal Comp #3 – 5 Fish	05424021
RH4W15	Rainbow Trout Hatchery FSCA #4 Whole body Comp #1 – 5 Fish	05424022
RH4W25	Rainbow Trout Hatchery FSCA #4 Whole body Comp #2 – 5 Fish	05424023
RH4W35	Rainbow Trout Hatchery FSCA #4 Whole body Comp #3 – 5 Fish	05424024
RH4W45	Rainbow Trout Hatchery FSCA #4 Whole body Comp #4 – 5 Fish	05424025
RH4W55	Rainbow Trout Hatchery FSCA #4 Whole body Comp #5 – 5 Fish	05424026
RH5W15	Rainbow Trout Hatchery FSCA #5 Whole body Comp #1 – 5 Fish	05424027
RH5W25	Rainbow Trout Hatchery FSCA #5 Whole body Comp #2 – 5 Fish	05424028
RH5W35	Rainbow Trout Hatchery FSCA #5 Whole body Comp #3 – 5 Fish	05424029
RH5W45	Rainbow Trout Hatchery FSCA #5 Whole body Comp #4 – 5 Fish	05424030
RH5W55	Rainbow Trout Hatchery FSCA #5 Whole body Comp #5 – 5 Fish *	05424031
RH5W65	Rainbow Trout Hatchery FSCA #5 Whole body Comp #6 – 5 Fish *	05424032
RH5W75	Rainbow Trout Hatchery FSCA #5 Whole body Comp #7 – 5 Fish *	05424033
RH6F15	Rainbow Trout Hatchery FSCA #6Fillet (R& L – skin on)# Comp #1 -5 Fish	05424034
RH6F25	Rainbow Trout Hatchery FSCA #6Fillet (R& L – skin on)# Comp #2 -5 Fish	05424035
RH6F35	Rainbow Trout Hatchery FSCA #6Fillet (R& L – skin on)# Comp #3 -5 Fish	05424036
RH6F45	Rainbow Trout Hatchery FSCA #6Fillet (R& L – skin on)# Comp #4 -5 Fish	05424037
RH6F55	Rainbow Trout Hatchery FSCA #6Fillet (R& L – skin on)# Comp #5 -5 Fish	05424038
RH6O15	Rainbow Trout Hatchery FSCA #6- Offal Comp #1 – 5 Fish	05424039
RH6O25	Rainbow Trout Hatchery FSCA #6- Offal Comp #2 – 5 Fish	05424040
RH6O35	Rainbow Trout Hatchery FSCA #6- Offal Comp #3 – 5 Fish	05424041
RH6O45	Rainbow Trout Hatchery FSCA #6- Offal Comp #4 – 5 Fish	05424042
RH6O55	Rainbow Trout Hatchery FSCA #6- Offal Comp #5 – 5 Fish	05424043
RW2W15	Rainbow Trout Wild FSCA #2 Whole body Comp #1 – 5 Fish	05424044
RW2W25	Rainbow Trout Wild FSCA #2 Whole body Comp #2 – 5 Fish	05424045
RW2W35	Rainbow Trout Wild FSCA #2 Whole body Comp #3 – 5 Fish *	05424046
RW2W45	Rainbow Trout Wild FSCA #2 Whole body Comp #4 – 5 Fish	05424047
RW2W65	Rainbow Trout Wild FSCA #2 Whole body Comp #6– 5 Fish *	05424048
RW2W75	Rainbow Trout Wild FSCA #2 Whole body Comp #7– 5 Fish *	05424049
RW3F15	Rainbow Trout Wild FSCA #3 Fillet (R& L – skin on)# Comp #1 -5 Fish	05424050

Field Sample Numbers	Sample Description	Region 10 Sample Tracking Number
RW3F25	Rainbow Trout Wild FSCA #3 Fillet (R& L – skin on)# Comp #1 -5 Fish	05424051
RW3O15	Rainbow Trout Wild FSCA #3 Offal Comp #1 – 5 Fish	05424052
RW3O25	Rainbow Trout Wild FSCA #3 Offal Comp #1 – 5 Fish	05424053
RW5W15	Rainbow Trout Wild FSCA #5 Whole body Comp #1 – 5 Fish	05424054
RW6F14	Rainbow Trout Wild FSCA #6 Fillet (R& L – skin on)# Comp #1 -5 Fish	05424055
RW6O14	Rainbow Trout Wild FSCA #6 Offal Comp #1 – 5 Fish	05424056
BB2W13	Burbot FSCA #2 Whole body Comp #1 – 3 Fish	05424057
BB2W23	Burbot FSCA #2 Whole body Comp #2 – 3 Fish	05424058
BB2W33	Burbot FSCA #2 Whole body Comp #3 – 3 Fish	05424059
BB3W15	Burbot FSCA #3 Whole body Comp #1 – 5 Fish	05424060
BB3W25	Burbot FSCA #3 Whole body Comp #2 – 5 Fish	05424061
BB3W35	Burbot FSCA #3 Whole body Comp #3 – 5 Fish *	05424062
BB3W45	Burbot FSCA #3 Whole body Comp #4 – 5 Fish	05424063
BB3W55	Burbot FSCA #3 Whole body Comp #4 – 5 Fish	05424064
BB3W65	Burbot FSCA #3 Whole body Comp #6 – 5 Fish *	05424065
BB3W75	Burbot FSCA #3 Whole body Comp #7 – 5 Fish *	05424066
BB4W15	Burbot FSCA #4 Whole body Comp #1 – 5 Fish	05424067
BB4W25	Burbot FSCA #4 Whole body Comp #2 – 5 Fish	05424068
BB4W35	Burbot FSCA #4 Whole body Comp #3 – 5 Fish	05424069
BB4W45	Burbot FSCA #4 Whole body Comp #4 – 5 Fish	05424070
BB5W15	Burbot FSCA #5 Whole body Comp #1 – 5 Fish	05424071
BB5W25	Burbot FSCA #5 Whole body Comp #2 – 5 Fish	05424072
BB5W35	Burbot FSCA #5 Whole body Comp #3 – 5 Fish	05424073
BB5W45	Burbot FSCA #5 Whole body Comp #4 – 5 Fish	05424074
BB5W55	Burbot FSCA #5 Whole body Comp #5 – 5 Fish	05424075
BB6W15	Burbot FSCA #6 Whole body Comp #1 – 5 Fish	05244076
BB6W25	Burbot FSCA #6 Whole body Comp #2 – 5 Fish	05424077
BB6W35	Burbot FSCA #6 Whole body Comp #3 – 5 Fish	05424078
BB6W45	Burbot FSCA #6 Whole body Comp #4 – 5 Fish	05424079
BB6W55	Burbot FSCA #6 Whole body Comp #5 – 5 Fish	05424080
LS1W45	Large Scale Sucker FSCA #1 Whole body Comp #4 – 5 Fish	05424081
LS4W15	Large Scale Sucker FSCA #4 Whole body Comp #1– 5 Fish	05424082
LS4W35	Large Scale Sucker FSCA #4 Whole body Comp #3– 5 Fish	05424083
LS4W45	Large Scale Sucker FSCA #4 Whole body Comp #4 – 5 Fish	05424084
LS4W55	Large Scale Sucker FSCA #4 Whole body Comp #5 – 5 Fish	05424085
LS5W15	Large Scale Sucker FSCA #5 Whole body Comp #1 – 5 Fish	05424086
LS5W25	Large Scale Sucker FSCA #5 Whole body Comp #2 – 5 Fish	05424087
LS5W35	Large Scale Sucker FSCA #5 Whole body Comp #3 – 5 Fish	05424088
LS5W45	Large Scale Sucker FSCA #5 Whole body Comp #4 – 5 Fish	05424089
LS5W55	Large Scale Sucker FSCA #5 Whole body Comp #5 – 5 Fish	05424090
LS6W15	Large Scale Sucker FSCA #6 Whole body Comp #1 – 5 Fish	05424091
LS6W25	Large Scale Sucker FSCA #6 Whole body Comp #2 – 5 Fish	05424092
LS6W35	Large Scale Sucker FSCA #6 Whole body Comp # 3 – 5 Fish *	05424093
LS6W45	Large Scale Sucker FSCA #6 Whole body Comp #4 – 5 Fish	05424094
LS6W65	Large Scale Sucker FSCA #6 Whole body Comp #6 – 5 Fish *	05424095

Field Sample Numbers	Sample Description	Region 10 Sample Tracking Number
LS6W75	Large Scale Sucker FSCA #6 Whole body Comp #7 – 5 Fish *	05424096
LS1G50769	Large Scale Sucker Guts FSCA 1 Comp #50769	05424097
LS1G50771	Large Scale Sucker Guts FSCA 1 Comp #50771	05424099
LS1W50769	Large Scale Sucker FSCA 1 Whole body (no guts) Comp #50769	05424253
LS1W50770	Large Scale Sucker FSCA 1 Whole body (no guts) Comp # 50770	05424254
LS1W50771	Large Scale Sucker FSCA 1 Whole body (no guts) Comp # 50771	05424255
LS1W50775	Large Scale Sucker FSCA 1 Whole body (no guts) Comp # 50775	05424256
LS1W50778	Large Scale Sucker FSCA 1 Whole body (no guts) Comp # 50778	05424257
LS1W60778	Large Scale Sucker FSCA 1 Whole body (no guts) Comp #60778	05424258
LS6W50727	Large Scale Sucker FSCA 6 Whole body (no guts) Comp # 50727	05424265
LS6W50732	Large Scale Sucker FSCA 6 Whole body (no guts) Comp # 50732	05424266
LS6W50734	Large Scale Sucker FSCA 6 Whole body (no guts) Comp # 50734	05424267
LS6W50744	Large Scale Sucker FSCA 6 Whole body (no guts) Comp # 50744	05424268
LS6W50747	Large Scale Sucker FSCA 6 Whole body (no guts) Comp # 50747	05424269
LS6W60734	Large Scale Sucker FSCA 6 Whole body (no guts) Comp # 60734	05424270

* Field Duplicate/Triplicate

DATA QUALIFICATIONS

The following comments refer to the laboratory's performance in meeting the Quality Control specifications outlined in the Phase 1 Fish Tissue Sampling Quality Assurance Project Plan (QAPP) for the Upper Columbia River Site CERCLA RI/FS, the analytical method SW846 Method 8082, the MEL's Standard Operating Procedure (SOP) #Or_Fish3541 and the MEL SOP for lipid determination.

The conclusions presented herein are based on the information provided for the review.

Field Sample Collection

The fish tissue sample collection was accomplished through a multi-agency/tribal effort with the CH2MHill team as the overall lead. Sample vessels and vessel operators were provided by the following tribal and federal agencies under an interagency or sub-contracting agreement with EPA and/or CH2MHill: Spokane Tribe of Indians, Confederated Tribes of the Colville Reservation, US Fish and Wildlife Services and the USEPA Region 10 Investigation and Engineering Unit of the Office of Environmental Assessment.

The sample collection dates were based on the fish availability and fish species' spawning season. There were two sample collection events conducted, first one was conducted in September 2005 and the second one was in October 2005. The fish species that were collected from the designated fish sample collection areas (FSCA 1 -6) were Walleye (*Sander vitreus*), Rainbow trout (*Oncorhynchus mykiss*), Lake white fish (*Coregonus clupeaformis*), Large-scale sucker (*Catostomas macrocheilus*), and Burbot (*Lota lota*). Long-nose suckers and Mountain whitefish were not originally listed in the QAPP as target fish species but were also collected and added to the target fish species due to their availability in the FSCAs. The mountain white fish were analyzed while the long-nose suckers were archived. The rainbow trout samples were grouped into three categories – wild, hatchery and mixed wild and hatchery. Only the wild and

hatchery rainbow trouts were analyzed for the chemical compounds of concern. The mixed wild and hatchery rainbow trouts were archived for future analysis, if needed.

The fish samples were generally collected using gill nets, electro-fishing, burbot traps and angling, if necessary. The field sample collection process was audited by the project's EPA and CH2MHill QA Managers. There were no significant problems encountered during sample collection, on-site processing, sampling documentation and sample shipment.

Sample Processing and Chain-of-Custody Documentation

CH2MHill set-up a trailer dedicated for the on-site fish sample processing which included visual inspection of the fish, sex determination, conducting field measurements (fish length and weight) and otolith removal. Otoliths are then later sent to the Washington Department of Fish and Wildlife (WDFW) for fish age determination. All of the field forms generated for these measurements and determination were evaluated and cross-checked with the homogenization forms and chain-of-custody (COC) documentation. All of the field measurements, field sampling documentation and sample preservation (freezing to -20C) were conducted by CH2MHill within 24 hours of sample collection.

Frozen whole fish samples were shipped to CH2MHill laboratory, Applied Science Laboratory (ASL), located in Corvallis, OR for filleting (if needed), homogenization, compositing, aliquot distribution and storage. There were four types of tissue sample composites prepared and analyzed for the chemical compounds of potential concern (COPCs) for the site, namely: fish fillets (both right and left side) with skin-on, offals (remaining tissue, internal organs and fish bones after filleting), guts (for large scale sucker only) and whole body (includes fish head, skin and entrails).

As specified in the EPA approved site QAPP, the following tissue types and homogenates were prepared by ASL and shipped to USEPA Manchester Environmental Laboratory (MEL) for subsequent PCB Aroclor, metals, percent lipids and speciated arsenic analyses and/or archival:

- Walleye – fillets and offals at three FSCAs and whole body composites from three FSCAs
- Rainbow trout – fillets and offals at three FSCAs and whole body composites from three FSCAs
- Lake whitefish – whole body composites
- Mountain whitefish – whole body composites
- Large scale sucker – whole body and guts/internal organs composites for metals analyses only
- Burbot – whole body composites
- Long-nose suckers – whole body composites

Sample Homogenization and Compositing

Fish samples from each sample location were individually homogenized at ASL. Appendix C lists the homogenized individual fish samples comprising a composite sample per fish specie. The fish samples were grinded using a commercial grade stainless steel blender/grinder (Robo-Coupe Blixer 6) with liquid

nitrogen. Equal amounts of homogenized whole body, fillet or offal tissue samples were mixed and composited to form a single sample. The homogenization forms and the resulting fish sample composites were evaluated by this reviewer. There were no major discrepancies noted between the sample collection forms, homogenization forms and the sample composite chain-of-custody documentation. Fillet samples are comprised of both right and left side with skin on. Whole body sample homogenates included the fish skin. Care was taken to prevent cross-contamination between sample homogenates. Prior to the start of the project samples, the filleting, removal of otoliths and homogenization processes were audited by the project's EPA and CH2MHill QA Managers. To monitor processing cross-contamination, proof blanks were collected at the QAPP specified frequency and sent to the Contract Laboratory Program (CLP) laboratory for the analysis of the project target compound.

Deviation from the QAPP: In a mock sample processing and homogenization conducted during the EPA's and CH2MHill's QA lab audit, it was found out that otoliths were very hard to remove when the fish samples were already frozen. In addition, subjecting the fish to freezing and defrosting ruptures the internal organs, make the fish muscles mushy and thus, made the separation of fillets from the offals quite a challenge.

To avoid cross-contamination of the fish tissue samples with the offals and to better preserve the otoliths, it was agreed by the project team that the removal of otolith will be conducted on-site after field measurements and before sample preservation (freezing to -20C) and if bench space and resources will allow, filleting of fish samples will also be performed on-site prior to freezing the samples.

Fish Age Determination

The following methods were used to determine the age of the fish: otoliths (inner ear of a fish) were used to determine the age of Lake whitefish, burbot and mountain whitefish. Both otoliths and scales were used to determine the age of the walleye, wild and hatchery rainbow trouts. The opercular covers (also called opercula) were used for large-scale suckers.

Otolith, scales and operculas were read with the knowledge of the place of capture, sex and size of the fish. The readings were performed by only one individual, Mr. John Sneva of WDFW. Precision and consistency of readings were checked through the comparison of annuli (otoliths) and the occuli (scales) readings when both specimens are available.

Fish age logs indicated the approximate ages of fish species comprising a composite as follows: lake whitefish ranged from 1-3 years; hatchery rainbow trout ranged from 1-2 years; wild rainbow trout from 1-4 years; mountain whitefish ranged from 0-15 years old; large-scale suckers, nine were <10 years old while the age of the rest of this specie ranged from >10-36 years old, walleyes and burbot ranged from 1-2 years.

Sample Receipt and Storage

All of the sample homogenates were received frozen and intact at MEL from ASL. The remaining whole fish samples (un-homogenized) were also sent to MEL for archiving and maybe future chemical analysis, if needed. After inspection, inventory and logging-in, the sample homogenates and un-homogenized fish samples were stored in a freezer at -20C. The fish samples remained frozen at -20C while waiting for extraction and analysis. The temperature of the freezer used for sample storage is monitored 24 hours by

MEL. The integrity of the fish samples and homogenates were maintained by MEL while on storage, during and after extraction and analysis.

COC Corrective Action

There were two COC corrective actions initiated by MEL to reconcile discrepancies between the regional tracking sample numbers and the field sample numbers for a few of the samples in this sample delivery group. The corrective actions and resolutions were sufficiently documented and new regional tracking sample numbers were issued by ALS to correct the regional sample number duplication.

Some minor discrepancies and missing information were also noted on the composite sample numbers listed on the COCs and the fish processing forms. ASL (represented by Mr. Robert Wong) and CH2MHill QA Manager, (Ms. Artemis Antipas) were contacted to clarify and correct these discrepancies on April 26, 2006. An explanation, reasons for the discrepancies and corrections were immediately sent to the reviewer.

Holding Times - Acceptable

A few of the fish sample analyses missed the project-specified extraction and analytical holding times of 6 months from the date of sample collection. However, none of the PCB data were qualified since the PSEP and National Fish Advisory holding times recommended for frozen fish tissue samples for PCB analyses is one year.

The list of samples, cross-referenced to the fish species, station locations, and the dates of sample collection, VTSR at the lab, extraction, extract clean-up and PCB and % lipid analysis dates are listed in Table 1 – Summary of Holding Times, at the end of this report.

Sample Preparation and Clean-up

All of the samples were extracted following the technical specifications of the analytical methods used. Prior to acid clean-up, 10% of the primary extracts were taken for % lipid determination. The rest of the primary extracts went through concentrated sulfuric acid clean-up (SW846 Method 3665) to isolate the PCBs and remove most of the organic material that would interfere with the analysis. Most of the samples also underwent through additional acid-base back extract clean-up to further remove oily interferences in the extracts. A 35% or 70% fraction of the original extract (depending on the amount extracted) was concentrated to 1.0 ml and passed through florisil cartridge clean-up (SW846 Method 3620) prior to GC analysis.

All of the analysts involved in sample extraction, extract clean-up and analysis of the samples in this data package performed an acceptable initial demonstration of capability (IDOC) studies prior to handling the samples.

In addition, the efficiency of the sample extraction procedure, clean-up and analytical processes were also monitored through the routine analysis of in-house Quality Control sample analyses and incorporation of routine-in-house QC checks (recoveries of the surrogate standards and the spike compounds in the laboratory control samples and matrix spike and duplicate analyses).

A single concentration polybrominated diphenyl ether (PBDE) standard runs were also conducted to monitor the presence of PBDE that would be potentially interfering with the Aroclor analyses.

Instrument Performance Checks - Acceptable

A dual-column GC analyses was used during the PCB Aroclor analysis. The designated primary column used in the quantitation of target compounds was Restek's CLP2 in all analytical sequences. The secondary, confirmatory column was Restek's CLP1. Baseline and retention time shifts were monitored and the instrument remained stable throughout the course of the analyses. None of the data were qualified on this basis.

Initial Calibrations - Acceptable

Five ICALs using 5-concentration levels of Aroclors 1016 and 1260 and one ICLA using 5-concentration levels of Aroclor 1254 were performed and used during the analysis of the samples listed in this validation report. A single-point concentration was analyzed for Aroclors 1221, 1232, 1242, 1248, 1254, 1262 and 1268 with each 1016/1260 ICAL. The frequency of analysis and the regression coefficients of the 5 major peaks used in the Aroclor identification and quantitation were all >0.995 for the primary column. Some of the peaks of from the secondary column did not meet the criteria of $r>0.995$, however, since this column was only used for confirmatory analyses, none of the data were qualified on this basis.

Continuing Calibrations - Acceptable

A mid-point concentration Aroclor 1016/1260 and/or Aroclor 1254 were analyzed for continuing calibration verification (CCVs) checks. The CCVs met the criteria for the frequency of analysis, the percent differences (%D) of the daily calibration factors (CFs) as compared to the mean CFs from the ICALs and the retention time shifts. None of the data were qualified on this basis.

Quantitation and Reporting Limits (QLs & RLs)

The QLs which are based on the lowest concentration level of the Aroclors in the ICALs, the amount of sample extracted and the final extract volume were about twice the project analytical concentration goals (ACGs) listed in Table 2-3 of the QAPP. Aroclor detections at concentrations $<QLs$, however, were reported by MEL with an estimated, "J", qualifier. All of the target compounds detected in the samples were calculated off the primary column using the CFs from the applicable ICALs.

Due to the low level concentrations of Aroclors 1260 and 1254 native to all of the fish samples and the interferences of other organic materials causing baseline noise and drifts, the reporting limits (RLs) for most of the non-detected Aroclors in the samples were elevated to about 10 times the QLs.

The concentrations of the Aroclors 1260 mixed with 1254 detected in most of the samples were qualified estimated, "J", due to the co-eluting peaks used in the calculations. PCB results from samples with poor chromatographic separations due to the interfering oily peaks were qualified estimated, "J", with a possible high bias.

Laboratory Method Blanks - Acceptable

The frequency of analysis of laboratory blank was met. All of the method blanks associated with the fish sample extraction, clean-up and analyses were clean. None of the data were qualified on this basis.

Homogenization Proof Blanks – Acceptable

A composite of final rinses during the decontamination of the Robo-Coupe Blixer 6 used for fish tissue and offal homogenization were collected. An aliquot of the composite rinses called “proof blanks” were collected every three days and shipped to the Superfund Contract Laboratory Program (CLP) labs. A total of 17 proof blanks were shipped to A4 Laboratory, Inc. of Woodlands TX and were analyzed for semi-volatile organic compounds (SVOCs), pesticides and PCB Aroclors. No PCB Aroclors were detected in any of the proof blanks. None of the fish tissue sample results were qualified on this basis.

Analytical Sequence - Acceptable

All of the standards, blanks, samples and QC samples were analyzed in accordance with the method specified analytical sequence. All of the analytical sequences were also bracketed by the continuing calibration check standards. None of the data were qualified on this basis.

Surrogate Recoveries – Acceptable

Tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) were used as the surrogate standards during analyses. Known concentrations of TCX and DCB were added to all samples and QC samples to monitor efficiency during sample extraction, clean-up and analysis. DCB is the surrogate associated with most of the PCB Aroclors. The DCB surrogate recoveries for all samples, QC samples and dilution runs were acceptable (50-150%). The DCB retention time shifts were also within the established retention time windows. None of the data were qualified on this basis.

Matrix Spike and Matrix Spike Duplicate (MS/MSD) Analysis

Sixteen QC samples were analyzed for MS/MD. Samples WE1F15 (05364201), LS1W25 (05364221), WE4W15 (05374210), LW3W55 (05374228), LW5W15 (05374235), RW1F15 (05374245), MW1W45 (05374257), RW2W53 (05414008), LS4W25 (05414019), WE5W25 (05424002), WE6W65 (05424006), LW2W15 (05424010), RH4W35 (05424024), RH4W55 (05424026), RW2W25 (05424045), RW3O15 (05424052), BB3W55 (05424064), BB5W45 (05414074) and LS4W15 (05424082) were the designated QC samples and analyzed for MS and MSD. The frequency of analysis of MS/MSD (10%) was met. Known concentrations of Aroclors 1016 and 1260 were spiked into the QC samples and went through the same extraction, clean-up and analytical procedures as the project samples.

The MS/MSD recoveries and relative percent differences (RPDs) criteria (30-150% and 50%, respectively) for the Aroclor 1016 were met for all QC samples. The recoveries of the low level Aroclor 1260 spiked into most of the QC samples could not be determined accurately due to the presence of Aroclors 1254 and 1260 in the samples and were reported as “NA” by the lab. To compensate for the Aroclors that are native to the QC samples, higher concentration levels of Aroclors 1016 and 1260 were spiked into the QC samples BB3W55 (05424064), BB5W45 (05414074) and LS4W15 (05424082). The Aroclor 1016 and 1260 recoveries for these three MS/MSD pairs were acceptable and ranged from 81 – 117%. None of the data were qualified on the basis of MS/MSD analyses.

Laboratory Control Sample and Duplicate Analyses (LCS/LCSD) – Acceptable

For LCS and LCSD, the hydromatrix extraction media was spiked with known concentrations of Aroclors 1016 and 1260. The frequency of analysis, recovery (70-130%) and RPD (50%) criteria were met for all LCS and LCSD analyses. None of the PCB Aroclor sample data were qualified on this basis. The analysis of LCS/LCSD is not applicable to lipid determination.

Analytical Duplicate Analyses - Acceptable

Samples LW4W45 (05374241), RW1O15 (05374251) and MW1W65 (05374261) were analyzed in duplicates. A mixture of Aroclors 1254 and 1260 were detected during the initial and duplicate analysis of all three QC samples. The RPDs between the concentrations of 1254/1260 detected in the original and duplicate sample runs ranged from 3-21%. The RPDs of the % lipids calculated the original and duplicate % lipid runs ranged from 9 – 24%. All of the RPDs were acceptable and within the QC limits of 50%. None of the data were qualified on this basis.

Field Duplicate Sample Analyses – Acceptable

RW1F55 (05374249) and RW1F65 (05374250) are the field duplicate samples of wild rainbow trout fillets collected from FSCA 1. A mixture of Aroclors 1260 and 1254 were identified in both samples at 21.6 and 19.1 ug/Kg estimated concentrations, respectively. The % lipids was 4.9% and 4.3% (RPD=6.7%). There was not much variability between the lipids and Aroclor duplicate values. None of the wild trout fillet Aroclor data was qualified on this basis.

RW1O55 (05374255) and RW1O65 (05374256) are the field duplicate samples of wild rainbow trout offals collected from FSCA 1. A mixture of Aroclors 1260 and 1254 were identified in both samples at 35 and 39.2 ug/Kg estimated concentrations, respectively. The % lipids was 11.2 % and 11.6 % (RPD=3.5%). There was not much variability between the lipids and Aroclor duplicate values. None of the wild trout offal Aroclor data was qualified on this basis.

MW1W45 (05374257), MW1W65 (05374261) and MW1W75 (05374262) are the mountain whitefish field triplicate samples collected from FSCA 1 submitted blind to MEL. Aroclors 1260/1254 was detected in all three samples at estimated concentrations of 65.5, 37.6 and 53.1 ug/Kg, respectively. The %RSD was 27% for the Aroclors and 16.6% for the lipids. There was not much variability between the lipids and Aroclor duplicate values. None of the mountain whitefish PCB data were qualified on this basis.

RW2W35 (05424046), RW2W65 (05424048) and RW2W75 (905424049) are the wild rainbow trout whole body field triplicates collected from FSCA 2. Aroclors 1254 and 1260 were detected in all three samples at the following concentrations: PCBs 1254 are 8.9 and 1260 at 20 ug/Kg in sample RW2W35; PCBs 1254 are 11 and 1260 at 20 ug/Kg in sample RW2W65 and PCBs 1254 are at 9.2 and 1260 at 22 ug/Kg in sample RW2W75. The PCB 1254 and 1260 %RSDs are 12% and 6%, respectively. The % lipids were 10.2, 9.7 and 9.9 yielding a %RSD of 4%. There was not much variability between the lipids and Aroclor triplicate values. None of the wild rainbow trout (whole body) PCB or % lipid data was qualified on this basis.

LS2W25 (05414010), LS2W65 (05414013) and LS2W75 (05414014) are the large scale sucker (whole body) field triplicate samples collected from FSCA 2 submitted blind to MEL. Aroclors 1254 and 1260 were detected in all three samples at the following concentrations: PCBs 1254 at 29 and 1260 at 67 ug/Kg in sample LS2W25; PCBs 1254 at 23 and 1260 at 59 ug/Kg in sample LS2W65 and PCBs 1254 at 20 and 1260 at 42 ug/Kg in sample LS2W75. The PCB 1254 and 1260 %RSDs are 19% and 23%, respectively. The % lipids were 3.3, 2.8 and 2.4, yielding a %RSD of 16%. There was not much variability between the lipids and Aroclor triplicate values. None of the large scale sucker PCB or % lipid data was qualified on this basis.

RH5W55 (05424031), RH5W65 (05424032) and RH5W75 (05424033) are the hatchery rainbow trout field triplicates collected from FSCA 5. PCBs 1254 at 4.3 and 1260 at 4.2 ug/Kg in sample RH5W55; PCB 1254 at 5.0 and PCB 1260 at 6.7 ug/Kg in sample RH5W65 and PCBs 1254 at 5.7 and 1260 at 5.0 ug/Kg in sample RH5W75. The PCB 1254 and 1260 %RSDs are 14% and 24%, respectively. The % lipids were 9, 9.4 and 9.7, yielding a %RSD of 4%. There was not much variability between the lipids and Aroclor triplicate values. None of the hatchery rainbow trout PCB or % lipid data was qualified on this basis.

BB3W35 (05424062), BB3W65 (5424065) and BB3W75 (05424066) are the burbot field triplicate samples collected from FSCA 3. Combined Aroclors 1254/1260 was detected in all three samples at the following concentrations: 31 ug/Kg in BB3W35; 38 ug/Kg in sample BB3W65 and 37 ug/Kg in sample BB#W75. The PCB 1254/1260 %RSD is 11 %. The % lipids were 1.2, 1.3 and 1.1, yielding a %RSD of 8 %. There was not much variability between the lipids and Aroclor triplicate values. None of the burbot PCB or % lipid data was qualified on this basis.

LS6W35 (05424093), LS6W65 (05424095) and LS6W75 (05424096) are the large scale sucker (whole body) field triplicate samples collected from FSCA 6 submitted blind to MEL. Combined Aroclors 1254/1260 was detected in all three samples at the following concentrations: 87 ug/Kg in sample LS6W35; 71 ug/Kg in sample LS6W65 and 80 ug/Kg in sample LS6W75. The PCB 1254/1260 %RSD is 10%. The % lipids were 6.9, 6.3 and 6.6, yielding a %RSD of 5%. There was not much variability between the lipids and Aroclor triplicate values. None of the large scale sucker PCB or % lipid data was qualified on this basis.

Compound Identification

Aroclors 1254 and 1260 were evidently present in all of the samples. The chromatogram overlays for the Aroclors 1260, 1254 and sample runs were mapped and evaluated by this reviewer. In instances where concentrations of Aroclors 1254 and 1260 were very low that only the major co-eluting peaks are quantifiable, the Aroclor detections were reported by the analysts as a combined Aroclor 1254/1260 with an estimated qualification. Where Aroclor 1254 and 1260 peaks could be isolated, separate concentrations for each Aroclor was reported. All of the extracts went through additional clean-ups when needed. Other than the usual low-level baseline noise, there were no other interferences observed with the chromatograms. All of the Aroclors reported somewhat matched the standard fingerprint patterns. All of the Aroclors identified were verified and are acceptable.

Laboratory Contact

The laboratory was not contacted for this review.

Overall Assessment

All of the samples were analyzed in accordance with the method specifications. There were no significant problems found with the data. The data, as qualified, are acceptable and can be used for all purposes.

Data Qualifiers	
U	The analyte was not detected at or above the reported numeric result.
J	The analyte was positively identified. The associated numerical result is an estimate.
UJ	The analyte was not detected at or above the reported estimated result. The associated numerical value is an estimate of the quantitation limit of the analyte in this sample.
R	The data are unusable for all purposes.
N	There is evidence the analyte is present in this sample.
JN	There is evidence that the analyte is present. The associated numerical result is an estimate.

TABLE 1- SUMMARY OF HOLDING TIMES

Sample Number	Region 10 Sample Tracking Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	Extraction Date	% Lipid Analysis Date	PCB Analysis Date
WE1F15	05364201	09/06/05	9/14/05	11/07/05	11/16/05	01/06/06	01/06/06	01/31/06
WE1F25	05364202	09/06/05	9/14/05	11/07/05	11/16/05	01/06/06	01/06/06	01/31/06
WE1F35	05364203	09/06/05	9/4/05	11/07/05	11/16/05	01/06/06	01/06/06	01/31/06
WE1F45	05364204	09/06/05	9/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE1F55	05364205	09/06/05	9/4/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE1O15	05364206	09/06/05	9/14/05	11/07/05	11/16/05	01/06/06	01/06/06	01/31/06
WE1O25	05364207	09/06/05	9/14/05	11/07/05	11/16/05	01/06/06	01/06/06	01/31/06
WE1O35	05364208	09/06/05	9/14/05	11/07/05	11/16/05	01/06/06	01/06/06	01/31/06
WE1O45	05364209	09/06/05	9/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE1O55	05364210	09/06/05	9/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
LS1W25	05364221	09/06/05	09/14/05	01/06/06	02/02/06	04/27/06	04/27/06	04/27/06
LS2W35	05364222	09/06/05	09/14/05	01/06/06	02/02/06	04/25/06	04/25/06	04/25/06
WE3F15	05374200	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3F25	05374201	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3F35	05374202	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3F45	05374203	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3F55	05374204	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3O15	05374205	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/23/06
WE3O25	05374206	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE3O35	05374207	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE3O45	05374208	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE3O55	05374209	09/10/05	09/14/05	11/08/05	11/16/05	01/06/06	01/06/06	01/31/06
WE4W15	05374210	09/10/05	09/14/05	11/09/05	11/16/05	01/09/06	01/09/06	01/23/06
WE4W25	05374213	09/12/05	09/14/05	11/09/05	11/16/05	01/09/06	01/09/06	01/23/06
WE4W35	05374214	09/12/05	09/14/05	11/09/05	11/16/05	01/09/06	01/09/06	01/31/06
WE4W45	05374215	09/12/05	09/14/05	11/09/05	11/16/05	01/09/06	01/09/06	01/31/06
WE4W55	05374216	09/12/05	09/14/05	11/09/05	11/16/05	01/09/06	01/09/06	01/23/06
WE6F15	05374217	09/14/05	09/15/05	11/11/05	11/16/05	01/09/06	01/09/06	01/23/06
WE6F25	05374218	09/14/05	09/15/05	11/11/05	11/16/05	05/02/06	01/10/06	02/16/06
WE6F35	05374219	09/14/05	09/15/05	11/11/05	11/16/05	01/10/06	01/10/06	02/16/06
WE6F45	05374220	09/14/05	09/15/05	11/13/05	11/16/05	01/10/06	01/10/06	02/16/06

Sample Number	Region 10 Sample Tracking Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	Extraction Date	% Lipid Analysis Date	PCB Analysis Date
WE6F55	05374221	09/14/05	09/15/05	11/13/05	11/16/05	01/10/06	01/10/06	02/16/06
WE6O15	05374222	09/14/05	09/15/05	11/11/05	11/16/05	01/10/06	01/10/06	02/17/06
WE6O25	05374223	09/14/05	09/15/05	11/11/05	11/16/05	01/10/06	01/10/06	02/17/06
WE6O35	05374224	09/14/05	09/15/05	11/11/05	11/16/05	01/10/06	01/10/06	02/17/06
WE6O45	05374225	09/14/05	09/15/05	11/13/05	11/16/05	01/10/06	01/10/06	02/17/06
WE6O55	05374226	09/14/05	09/15/05	11/13/05	11/16/05	01/10/06	01/10/06	02/17/06
LW3W15	05374227	09/13/05	09/14/05	11/22/05	12/08/05	01/10/06	01/10/06	02/16/06
LW3W55	05374228	09/13/05	09/14/05	11/22/05	12/08/05	01/17/06	01/17/06	02/16/06
LW3W25	05374229	09/14/05	09/15/05	11/21/05	12/08/05	01/17/06	01/17/06	02/16/06
LW3W35	05374230	09/14/05	09/15/05	10/14/05	12/08/05	01/17/06	01/17/06	02/17/06
LW3W45	05374231	09/14/05	09/15/05	11/21/05	12/08/05	01/17/06	01/17/06	02/17/06
LW3W65	05374232	09/14/05	09/15/05	11/21/05	12/08/05	03/21/06	03/21/06	04/18/06
LW3W75	05374233	09/14/05	09/15/05	11/21/05	12/08/05	01/17/06	01/17/06	02/17/06
LW5W25	05374234	09/14/05	09/15/05	11/21/05	12/08/05	03/21/06	03/21/06	04/18/06
LW5W15	05374235	09/15/05	09/16/05	11/16/05	12/08/05	01/31/06	01/31/06	03/07/06
LW5W35	05374236	09/15/05	09/16/05	11/16/05	12/08/05	01/31/06	01/31/06	03/07/06
LW5W45	05374237	09/15/05	09/16/05	11/22/05	12/08/05	01/31/06	01/31/06	03/07/06
LW5W55	05374238	09/15/05	09/16/05	11/28/05	12/08/05	01/31/06	01/31/06	03/07/06
LW4W25	05374239	09/17/05	09/18/05	11/22/05	12/08/05	01/31/06	01/31/06	03/07/06
LW4W35	05374240	09/17/05	09/18/05	11/22/05	12/08/05	01/31/06	01/31/06	03/08/06
LW4W45	05374241	09/17/05	09/18/05	11/28/05	12/08/05	01/31/06	01/31/06	03/08/06
LW4W55	05374242	09/17/05	09/18/05	11/28/05	12/08/05	02/01/06	02/01/06	03/08/06
LW6W13	05374243	09/17/05	09/18/05	11/18/05	12/08/05	02/01/06	02/01/06	03/08/06
LW4W15	05374244	09/17/05	09/18/05	11/21/05	12/08/05	02/01/06	02/01/06	03/08/06
RW1F15	05374245	09/13/05	09/14/05	12/20/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1F25	05374246	09/13/05	09/14/05	12/22/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1F35	05374247	09/13/05	09/14/05	12/22/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1F45	05374248	09/13/05	09/14/05	12/23/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1F55	05374249	09/13/05	09/14/05	12/21/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1F65	05374250	09/13/05	09/14/05	12/21/05	01/06/06	02/01/06	02/01/06	03/08/06

Sample Number	Region 10 Sample Tracking Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	Extraction Date	% Lipid Analysis Date	PCB Analysis Date
RW1O15	05374251	09/13/05	09/14/05	12/20/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1O25	05374252	09/13/05	09/14/05	12/22/05	01/06/06	02/01/06	02/01/06	03/08/06
RW1O35	05374253	09/13/05	09/14/05	12/22/05	01/06/06	02/07/06	02/07/06	03/08/06
RW1O45	05374254	09/13/05	09/14/05	12/23/05	01/06/06	02/07/06	02/07/06	03/08/06
RW1O55	05374255	09/13/05	10/22/05	12/21/05	01/06/06	02/07/06	02/07/06	03/08/06
RW1O65	05374256	09/13/05	10/22/05	12/21/05	01/06/06	02/07/06	02/07/06	03/08/06
MW1W45	05374257	09/13/05	09/14/05	11/29/05	01/12/06	02/07/06	02/07/06	03/08/06
MW1W55	05374258	09/13/05	09/14/05	11/30/05	01/12/06	02/07/06	02/07/06	03/08/06
MW1W65	05374261	09/14/05	09/14/05	11/29/05	01/12/06	02/15/06	02/15/06	03/08/06
MW1W75	05374262	09/14/05	09/14/05	11/29/05	01/12/06	02/15/06	02/15/06	03/08/06
MW1W15	05374263	09/13/05	09/14/05	11/30/05	01/12/06	02/15/06	02/15/06	03/08/06
MW1W25	05374264	09/13/05	09/14/05	11/29/05	01/12/06	02/15/06	02/15/06	03/08/06
MW1W35	05374265	09/13/05	09/14/05	11/30/05	01/12/06	02/15/06	02/15/06	03/08/06
WE2W15	05414001	10/12/05	10/18/05	11/10/05	11/16/05	03/15/06	03/15/06	04/19/06
WE2W25	05414002	10/12/05	10/18/05	11/10/05	11/16/05	03/15/06	03/15/06	04/19/06
WE2W35	05414003	10/12/05	10/18/05	11/10/05	11/16/05	03/15/06	03/15/06	04/19/06
WE2W45	05414004	10/12/05	10/18/05	11/10/05	11/16/05	03/15/06	03/15/06	04/19/06
WE2W55	05414005	10/12/05	10/18/05	11/10/05	11/16/05	03/15/06	03/15/06	04/19/06
WE2W65	05414006	10/12/05	10/18/05	11/10/05	11/16/05	03/15/06	03/15/06	04/19/06
WE2W75	05414007	10/12/05	10/18/05	11/10/05	11/16/05	03/15/06	03/15/06	04/19/06
RW2W53	05414008	10/13/05	10/19/05	01/11/06	02/02/06	03/17/06	03/17/06	04/17/06
LS2W15	05414009	10/13/05	10/19/05	01/11/06	02/02/06	03/17/06	03/17/06	04/17/06
LS2W25	05414010	10/13/05	10/19/05	01/09/06	02/02/06	03/17/06	03/17/06	04/17/06
LS2W35	05414011	10/12/05	10/19/05	01/11/06	02/02/06	05/02/06	05/02/06	05/11/06
LS2W45	05414012	10/12/05	10/19/05	01/10/06	02/02/06	03/17/06	03/17/06	04/18/06
LS2W65	05414013	10/13/05	10/19/05	01/09/06	02/02/06	03/17/06	03/17/06	04/18/06
LS3W75	05414014	10/14/05	10/19/05	01/09/06	02/02/06	03/17/06	03/17/06	04/17/06
LS3W15	05414015	10/14/05	10/19/05	01/19/06	02/02/06	03/17/06	03/17/06	04/17/06
LS3W25	05414016	10/14/05	10/19/05	01/20/06	02/02/06	03/17/06	03/17/06	04/18/06
LS3W35	05414017	10/14/05	10/19/05	01/23/06	02/02/06	04/03/06	04/03/06	04/27/06

Sample Number	Region 10 Sample Tracking Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	Extraction Date	% Lipid Analysis Date	PCB Analysis Date
LS3W55	05414018	10/14/05	10/19/05	01/23/06	02/02/06	04/03/05	04/03/05	04/27/06
LS4W25	05414019	10/14/05	10/19/05	01/23/06	02/02/06	03/21/06	03/21/06	04/18/06
WE5W15	05424001	10/17/05	10/13/05	11/10/05	12/08/05	03/21/06	03/21/06	04/18/06
WE5W25	05424002	10/17/05	10/18/05	11/09/05	12/08/05	03/21/06	03/21/06	04/18/06
WE5W35	05424005	10/17/05	10/18/05	11/11/05	11/16/05	03/21/06	03/21/06	04/18/06
WE6W65	05424006	10/20/05	10/23/05	11/09/05	11/16/05	03/21/06	03/21/06	04/18/06
WE6W75	05424009	10/20/05	10/23/05	11/09/05	11/16/05	03/21/06	03/21/06	04/18/06
LW2W15	05424010	10/18/05	10/19/05	11/17/05	12/08/05	03/21/06	03/21/06	04/18/06
LW2W25	05424011	10/18/05	10/19/05	11/16/05	12/08/05	03/27/06	03/27/06	04/19/06
LW2W45	05424012	10/18/05	10/19/05	11/28/05	12/08/05	03/27/06	03/27/06	04/19/06
LW2W55	05424013	10/20/05	10/23/05	11/28/05	12/08/05	03/27/06	03/27/06	04/19/06
LW2W35	05424014	10/19/05	10/20/05	11/15/05	12/08/05	03/27/06	03/27/06	04/19/06
LW6W23	05424015	10/22/05	10/23/05	11/18/05	12/08/05	03/28/06	03/28/06	04/25/06
RH3F15	05424016	10/18/05	10/22/05	12/01/05	12/22/05	03/27/06	03/27/06	04/19/06
RH3F25	05424017	10/18/05	10/22/05	12/02/05	12/22/05	03/27/06	03/27/06	04/19/06
RH3F35	05424018	10/18/05	10/22/05	12/05/05	12/22/05	03/27/06	03/27/06	04/19/06
RH3O15	05424019	10/18/05	10/22/05	12/01/05	12/22/05	03/27/06	03/27/06	04/19/06
RH3O25	05424020	10/18/05	10/22/05	12/02/05	12/22/05	03/27/06	03/27/06	04/19/06
RH3O35	05424021	10/18/05	10/22/05	12/05/05	12/22/05	03/27/06	03/27/06	04/19/06
RH4W15	05424022	10/18/05	10/22/05	12/13/05	12/22/05	03/27/06	03/27/06	04/19/06
RH4W25	05424023	10/18/05	10/22/05	12/12/05	12/22/05	03/27/06	03/27/06	04/19/06
RH4W35	05424024	10/18/05	10/22/05	12/14/05	12/22/05	03/27/06	03/27/06	04/19/06
RH4W45	05424025	10/18/05	10/22/05	12/13/05	12/22/05	03/27/06	03/27/06	04/19/06
RH4W55	05424026	10/18/05	10/22/05	12/13/05	12/22/05	03/27/06	03/27/06	04/19/06
RH5W15	05424027	10/20/05	10/22/05	12/08/05	12/22/05	03/27/06	03/27/06	04/19/06
RH5W25	05424028	10/20/05	10/22/05	12/09/05	12/22/05	03/27/06	03/27/06	04/19/06
RH5W35	05424029	10/20/05	10/22/05	12/08/05	12/22/05	03/28/06	03/28/06	04/25/06
RH5W45	05424030	10/21/05	10/22/05	12/09/05	12/22/05	03/28/06	03/28/06	04/25/06
RH5W55	05424031	10/21/05	10/22/05	12/06/05	12/22/05	03/28/06	03/28/06	04/25/06
RH5W65	05424032	10/20/05	10/22/05	12/07/05	12/22/05	03/28/06	03/28/06	04/25/06

Sample Number	Region 10 Sample Tracking Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	Extraction Date	% Lipid Analysis Date	PCB Analysis Date
RH5W75	05424033	10/20/05	10/22/05	12/07/05	12/22/05	03/28/06	03/28/06	04/25/06
RH6F15	05424034	10/21/05	10/22/05	12/14/05	12/22/05	03/28/06	03/28/06	04/25/06
RH6F25	05424035	10/21/05	10/22/05	12/15/05	12/22/05	03/28/06	03/28/06	04/25/06
RH6F35	05424036	10/21/05	10/22/05	12/15/05	12/22/05	03/28/06	03/28/06	04/25/06
RH6F45	05424037	10/21/05	10/22/05	12/15/05	12/22/05	03/28/06	03/28/06	04/25/06
RH6F55	05424038	10/21/05	10/22/05	12/15/05	12/22/05	03/28/06	03/28/06	04/25/06
RH6O15	05424039	10/21/05	10/22/05	12/15/05	12/22/05	03/28/06	03/28/06	04/25/06
RH6O25	05424040	10/21/05	10/22/05	12/15/05	12/22/05	03/28/06	03/28/06	04/25/06
RH6O35	05424041	10/21/05	10/22/05	12/15/05	12/22/05	03/28/06	03/28/06	04/25/06
RH6O45	05424042	10/21/05	10/22/05	12/15/05	12/22/05	03/28/06	03/28/06	04/25/06
RH6O55	05424043	10/21/05	10/22/05	12/15/05	12/22/05	03/28/06	03/28/06	04/25/06
RW2W15	05424044	10/18/05	10/22/05	12/16/05	01/06/06	03/28/06	03/28/06	04/25/06
RW2W25	05424045	10/19/05	10/22/05	12/17/05	01/06/06	03/28/06	03/28/06	04/25/06
RW2W35	05424046	10/18/05	10/22/05	12/17/05	01/06/06	03/28/06	03/28/06	04/25/06
RW2W45	05424047	10/18/05	10/22/05	12/16/05	01/06/06	03/29/06	03/29/06	04/25/06
RW2W65	05424048	10/18/05	10/22/05	12/17/05	01/06/06	03/29/06	03/29/06	04/25/06
RW2W75	05424049	10/18/05	10/22/05	12/17/05	01/06/06	03/29/06	03/29/06	04/25/06
RW3F15	05424050	10/19/05	10/22/05	12/19/05	01/06/06	03/29/06	03/29/06	04/25/06
RW3F25	05424051	10/19/05	10/22/05	12/19/05	01/06/06	03/29/06	03/29/06	04/25/06
RW3O15	05424052	10/19/05	10/22/05	12/19/05	01/06/06	03/29/06	03/29/06	04/26/06
RW3O25	05424053	10/19/05	10/22/05	12/19/05	01/06/06	03/29/06	03/29/06	04/26/06
RW5W15	05424054	10/20/05	10/22/05	12/16/05	01/06/06	03/29/06	03/29/06	04/26/06
RW6F14	05424055	10/21/05	10/22/05	12/19/05	01/06/06	03/29/06	03/29/06	04/26/06
RW6O14	05424056	10/21/05	10/22/05	12/20/05	01/06/06	03/29/06	03/29/06	04/26/06
BB2W13	05424057	10/18/05	10/22/05	12/27/05	01/20/06	03/29/06	03/29/06	04/26/06
BB2W23	05424058	10/18/05	10/22/05	12/27/05	01/20/06	03/29/06	03/29/06	04/26/06
BB2W33	05424059	10/18/05	10/22/05	12/27/05	01/20/06	03/29/06	03/29/06	04/26/06
BB3W15	05424060	10/18/05	10/22/05	12/30/05	01/20/06	03/29/06	03/29/06	04/26/06
BB3W25	05424061	10/18/05	10/22/05	01/03/06	01/20/06	03/29/06	03/29/06	04/26/06
BB3W35	05424062	10/18/05	10/22/05	01/04/06	01/20/06	03/29/06	03/29/06	04/26/06

Sample Number	Region 10 Sample Tracking Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	Extraction Date	% Lipid Analysis Date	PCB Analysis Date
BB3W45	05424063	10/18/05	10/22/05	01/04/06	01/20/06	03/29/06	03/29/06	04/26/06
BB3W55	05424064	10/18/05	10/22/05	01/04/06	01/20/06	03/29/06	03/29/06	04/26/06
BB3W65	05424065	10/18/05	10/22/05	01/04/06	01/20/06	03/29/06	03/29/06	04/26/06
BB3W75	05424066	10/18/05	10/22/05	01/04/06	01/20/06	03/29/06	03/29/06	04/26/06
BB4W15	05424067	10/19/05	10/22/05	01/04/06	01/20/06	03/29/06	03/29/06	04/26/06
BB4W25	05424068	10/18/05	10/22/05	01/05/06	01/20/06	03/29/06	03/29/06	04/26/06
BB4W35	05424069	10/19/05	10/22/05	01/05/06	01/20/06	03/29/06	03/29/06	04/26/06
BB4W45	05424070	10/19/05	10/22/05	01/05/06	01/20/06	03/29/06	03/29/06	04/26/06
BB5W15	05424071	10/20/05	10/24/06	12/27/06	01/20/06	03/31/06	03/31/06	04/26/06
BB5W25	05424072	10/22/05	10/24/06	12/28/06	01/20/06	03/31/06	03/31/06	04/26/06
BB5W35	05424073	10/22/05	10/24/06	12/28/06	01/20/06	03/31/06	03/31/06	04/26/06
BB5W45	05424074	10/22/05	10/24/06	12/28/06	01/20/06	03/31/06	03/31/06	04/26/06
BB5W55	05424075	10/22/05	10/24/06	12/28/06	01/20/06	03/31/06	03/31/06	04/26/06
BB6W15	05244076	10/22/06	10/24/06	12/30/05	01/20/06	03/31/06	03/31/06	04/26/06
BB6W25	05424077	10/22/06	10/24/06	12/29/05	01/20/06	03/31/06	03/31/06	04/26/06
BB6W35	05424078	10/22/06	10/24/06	12/29/05	01/20/06	03/31/06	03/31/06	04/26/06
BB6W45	05424079	10/22/06	10/24/06	12/30/05	01/20/06	03/31/06	03/31/06	04/26/06
BB6W55	05424080	10/22/05	10/24/05	12/30/05	01/20/06	03/29/06	03/29/06	04/27/06
LS1W45	05424081	10/19/05	10/22/05	01/09/06	02/02/06	04/03/06	04/03/06	04/27/06
LS4W15	05424082	10/16/05	10/19/05	01/16/06	02/02/06	04/03/06	04/03/06	05/05/06
LS4W35	05424083	10/16/05	10/19/05	01/16/06	02/02/06	04/03/06	04/03/06	05/11/06
LS4W45	05424084	10/16/05	10/19/05	01/14/06	02/02/06	04/03/06	04/03/06	04/27/06
LS4W55	05424085	10/16/05	10/19/05	01/14/06	02/02/06	04/03/06	04/03/06	05/11/06
LS5W15	05424086	10/17/05	10/22/05	01/12/06	02/02/06	04/03/06	04/03/06	04/27/06
LS5W25	05424087	10/17/05	10/22/05	01/08/06	02/02/06	04/03/06	04/03/06	05/11/06
LS5W35	05424088	10/17/05	10/22/05	01/12/06	02/02/06	04/03/06	04/03/06	05/11/06
LS5W45	05424089	10/17/05	10/22/05	01/11/06	02/02/06	04/03/06	04/03/06	05/11/06
LS5W55	05424090	10/17/05	10/22/05	01/13/06	02/02/06	04/03/06	04/03/06	04/27/06
LS6W15	05424091	10/18/05	10/22/05	01/18/06	02/02/06	04/03/06	04/03/06	04/27/06
LS6W25	05424092	10/18/05	10/22/05	01/17/06	02/02/06	04/03/06	04/03/06	04/27/06

Sample Number	Region 10 Sample Tracking Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	Extraction Date	% Lipid Analysis Date	PCB Analysis Date
LS6W35	05424093	10/18/05	10/22/05	01/17/06	02/02/06	04/03/06	04/03/06	04/27/06
LS6W45	05424094	10/18/05	10/22/05	01/19/06	02/02/06	04/03/06	04/03/06	04/27/06
LS6W65	05424095	10/18/05	10/22/05	01/17/06	02/02/06	04/03/06	04/03/06	04/27/06
LS6W75	05424096	10/18/05	10/22/05	01/17/06	02/02/06	04/03/06	04/03/06	04/27/06
LS1G50769	05424097	10/19/05	10/22/05	01/06/06	02/02/06	NA *	04/18/06	NA *
LS1G50771	05424099	10/19/05	10/22/05	01/06/06	02/02/06	NA	04/18/06	NA
LS1W50769	05424253	10/19/05	10/22/05	01/06/06	02/02/06	NA	04/18/06	NA
LS1W50770	05424254	10/19/05	10/22/05	01/06/06	02/02/06	NA	04/18/06	NA
LS1W50771	05424255	10/19/05	10/22/05	01/06/06	02/02/06	NA	04/18/06	NA
LS1W50775	05424256	10/19/05	10/22/05	01/06/06	02/02/06	NA	04/18/06	NA
LS1W50778	05424257	10/19/05	10/22/05	01/06/06	02/02/06	NA	04/13/06	NA
LS1W60778	05424258	10/19/05	10/22/05	01/19/06	02/02/06	NA	04/13/06	NA
LS6W50727	05424265	10/18/05	10/22/05	01/19/06	02/02/06	NA	04/13/06	NA
LS6W50732	05424266	10/18/05	10/22/05	01/19/06	02/02/06	NA	04/18/06	NA
LS6W50734	05424267	10/18/05	10/22/05	01/14/06	02/02/06	NA	04/13/06	NA
LS6W50744	05424268	10/18/05	10/22/05	01/19/06	02/02/06	NA	04/13/06	NA
LS6W50747	05424269	10/18/05	10/22/05	01/19/06	02/02/06	NA	04/18/06	NA
LS6W60734	05424270	10/18/05	10/22/05	01/19/06	02/02/06	NA	04/18/06	NA

* NA – Not Analyzed.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 8, 2006

Reply to
Attn of: **MGREPOGR**
OEA-095

MEMORANDUM

Subject: Data Validation Report for the Polychlorinated Dibenzodioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) Analysis of the Fish Tissue Samples Collected for the Phase I Upper Columbia River (UCR) Remedial Investigation/Feasibility Study (RI/FS) September 2005

From: Ginna Grepo-Grove, Senior Chemist
Technical Support Unit, OEA

To: Sally Thomas, RPM, UCR, Fish Tissue Study
USEPA, ECL

Marc Stifelman, Human Health Risk Assessment, USEPA, OEA
Burt Shephard, Ecological Risk Assessment, USEPA, OEA
Jim Stefanoff, Project Manager, CH2MHill
Artemis Antipas, QA Manager, CH2MHill

The quality assurance (QA) review of 186 fish tissue samples collected from the above referenced site has been completed. These samples were analyzed for PCDD/PCDF in accordance with the Contract Laboratory Program's (CLP) Statement of Work (SOW) for the Multi-Media, Multi-Concentration Dioxins and Furans Analysis (DLM02.0) and the Project - Modified Analysis and Flexibility Clause. The analyses were performed by Paradigm Analytical of Wilmington, NC. The following samples were evaluated in this validation report:

Field Sample Number	Sample Description	Region Tracking Number	CLP Tag Number	Paradigm Laboratory Number
WE1F15	Walleye FSCA#1 Comp# 1 - 5 Fish Fillets Skin-on (R & L)	05364201	274154	G619-9-1
WE1F25	Walleye FSCA#1 Comp# 2 - 5 Fish Fillet Skin-on (R & L)	05364 202	274162	G619-9-2
WE1F35	Walleye FSCA#1 Comp# 3 - 5 Fish Fillet Skin-on (R & L)	05364203	274174	G619-9-3
WE1F45	Walleye FSCA#1 Comp# 4 - 5 Fish Fillet Skin-on (R & L)	05364204	274186	G619-9-4
WE1F55	Walleye FSCA#1 Comp# 5 - 5 Fish Fillet Skin-on (R & L)	05364205	274199	G619-9-5
WE1O15	Walleye FSCA#1 Comp# 1 - 5 Offals	05364206	274158	G619-9-6

Field Sample Number	Sample Description	Region Tracking Number	CLP Tag Number	Paradigm Laboratory Number
WE1O25	Walleye FSCA#1 Comp# 2 - 5 Offals	05364207	274168	G619-9-7
WE1O35	Walleye FSCA#1 Comp# 3 - 5 Offals	05364208	274180	G619-9-8
WE1O45	Walleye FSCA#1 Comp# 4 - 5 Offals	05364209	274193	G619-9-9
WE1O55	Walleye FSCA#1 Comp# 5 - 5 Offals	05364210	274785	G619-9-10
WE3F15	Walleye FSCA#3 Comp# 1 - 5 Fish Fillet Skin-on (R & L)	05374200	274782	G619-9-11
WE3F25	Walleye FSCA#3 Comp# 2 - 5 Fish Fillet Skin-on (R & L)	05374201	274374	G619-9-12
WE3F35	Walleye FSCA#3 Comp# 3 - 5 Fish Fillet Skin-on (R & L)	05374202	274264	G619-9-13
WE3F45	Walleye FSCA#3 Comp# 4 - 5 Fish Fillet Skin-on (R & L)	05374203	274769	G619-9-14
WE3F55	Walleye FSCA#3 Comp# 5 - 5 Fish Fillet Skin-on (R & L)	05374204	274252	G619-9-15
WE3O15	Walleye FSCA#3 Comp# 1 - 5 Offals	05374205	274756	G619-9-16
WE3O35	Walleye FSCA#3 Comp# 3 - 5 Offals	05374206	274762	G619-9-17
WE3O45	Walleye FSCA#3 Comp# 3 -5 Offals	05374207	274791	G619-8-18
WE3O55	Walleye FSCA#3 Comp# 5 -5 Offals	05374208	274257	G619-9-19
WE4W15	Walleye FSCA#4 Wholebody Comp #1 - 5 Fish	05374209	NA **	G619-9-20
LW2W15	Lake White Fish FSCA#2 Whole body Comp# 1 - 5 Fish	05424010	274051	G619-10-1
LW2W25	Lake White Fish FSCA#2 Whole body Comp# 2 - 5 Fish	05424011	274072	G619-10-2
LW2W35	Lake White Fish FSCA#2 Whole body Comp# 3 - 5 Fish	05424014	274079	G619-10-3
LW2W45	Lake White Fish FSCA#2 Whole body Comp# 4 - 5 Fish	05424012	274086	G619-10-4
LW3W25	Lake White Fish FSCA#3 Whole body Comp# 2 - 5 Fish *	05374229	274574	G619-10-5
LW3W55	Lake White Fish FSCA#3 Whole body Comp# 5 - 5 Fish	05374228	274675	G619-10-6
LW4W15	Lake White Fish FSCA#4 Whole body Comp# 1 - 5 Fish	05374244	274392	G619-10-7
LW4W45	Lake White Fish FSCA#4 Whole body Comp# 4 - 5 Fish	05374241	274702	G619-10-8
LW5W15	Lake White Fish FSCA#5 Whole body Comp# 1 - 5 Fish	05374235	274717	G619-10-9
LW5W25	Lake White Fish FSCA#5 Whole body Comp# 2 - 5 Fish	05374234	274724	G619-10-10
LW5W35	Lake White Fish FSCA#5 Whole body Comp# 3 - 5 Fish	05374236	274731	G619-10-11
LW5W45	Lake White Fish FSCA#5 Whole body Comp# 4 - 5 Fish	05374237	274737	G619-10-12
LW5W55	Lake White Fish FSCA#5 Whole body Comp# 5 - 5 Fish	05374238	274745	G619-10-13
LW6W13	Lake White Fish FSCA#6 Whole body Comp# 1 - 3 Fish	05374243	274099	G619-10-14
LW6W23	Lake White Fish FSCA#6 Whole body Comp# 2 - 3 Fish	05424015	274620	G619-10-15
WE2W55	Walleye FSCA#2 Whole body Comp# 5 - 5 fish*	05414005	274368	G619-10-16
WE2W65	Walleye FSCA#2 Whole body Comp# 6 - 5 fish*	05414006	274372	G619-10-17
WE2W75	Walleye FSCA#2 Whole body Comp# 7 - 5 fish*	05414007	274496	G619-10-18
WE5W15	Walleye FSCA#5 Whole body Comp# 1 - 5 fish	05424001	274481	G619-10-19
WE5W25	Walleye FSCA#5 Whole body Comp# 2 - 5 fish	05424002	274615	G619-10-20
WE2W15	Walleye FSCA#2 Whole body Comp# 1 - 5 fish	05414001	274359	G619-11-1
WE2W25	Walleye FSCA#2 Whole body Comp# 2 - 5 fish	05414002	274382	G619-11-2
WE2W35	Walleye FSCA#2 Whole body Comp# 3 - 5 fish	05414003	274459	G619-11-3
WE2W45	Walleye FSCA#2 Whole body Comp# 4 - 5 fish	05414004	274631	G619-11-4
WE3O25	Walleye FSCA#3 Comp# 2 - 5 Offals	05374206	274451	G619-11-5
WE4W25	Walleye FSCA#4 Whole body Comp# 2 -5 fish	05374213	274292	G619-11-6
WE4W35	Walleye FSCA#4 Whole body Comp# 3 -5 fish	05374214	274352	G619-11-7
WE4W45	Walleye FSCA#4 Whole body Comp# 4 -5 fish	05374215	274474	G619-11-8
WE4W55	Walleye FSCA#4 Whole body Comp# 5 -5 fish	05374216	274468	G619-11-9
WE5W35	Walleye FSCA#5 Whole body Comp# 3 -5 fish	05424005	274603	G619-11-10
WE6F15	Walleye FSCA#6 Comp# 1 - 5 Fish Fillet Skin-on (R & L)	05374217	274504	G619-11-11
WE6F25	Walleye FSCA#6 Comp# 2 - 5 Fish Fillet Skin-on (R & L)	05374218	274663	G619-11-12
WE6F35	Walleye FSCA#6 Comp# 3 - 5 Fish Fillet Skin-on (R & L)	05374219	274518	G619-11-13
WE6F45	Walleye FSCA#6 Comp# 4 - 5 Fish Fillet Skin-on (R & L)	05374220	274533	G619-11-14
WE6F55	Walleye FSCA#6 Comp# 5 - 5 Fish Fillet Skin-on (R & L)	05374221	274543	G619-11-15
WE6O15	Walleye FSCA#6 Comp# 1 -5 Offals	05374222	274511	G619-11-16
WE6O25	Walleye FSCA#6 Comp# 2 - 5 Offals	05374223	274666	G619-11-17

Field Sample Number	Sample Description	Region Tracking Number	CLP Tag Number	Paradigm Laboratory Number
WE6O35	Walleye FSCA#6 Comp# 3 - 5 Offals	05374224	274524	G619-11-18
WE6O45	Walleye FSCA#6 Comp# 4 - Offals	05374225	274537	G619-11-19
WE6O55	Walleye FSCA#6 Comp# 5 - Offals	05374226	274657	G619-11-20
WE6W65	Walleye FSCA#6 Whole body Comp# 6 - 5 fish*	05424006	274488	G619-12-1
WE6W75	Walleye FSCA#6 Whole body Comp# 7 - 5 fish	05424009	274354	G619-12-2
LW2W55	Lake White Fish FSCA#2 Whole body Comp# 5 - 5 Fish	05424013	274982	G619-12-3
LW3W15	Lake White Fish FSCA#3 Whole body Comp# 1 - 5 Fish	05374227	274553	G619-12-4
LW3W35	Lake White Fish FSCA#3 Whole body Comp# 3 - 5 Fish	05374230	274588	G619-12-5
LW3W45	Lake White Fish FSCA#3 Whole body Comp# 4 - 5 Fish	05374231	274595	G619-12-6
LW3W65	Lake White Fish FSCA#3 Whole body Comp# 6 - 5 Fish*	05374232	274567	G619-12-7
LW3W75	Lake White Fish FSCA#3 Whole body Comp# 7 - 5 Fish*	05374233	274581	G619-12-8
LW4W25	Lake White Fish FSCA#4 Whole body Comp# 2 - 5 Fish	05374239	274560	G619-12-9
LW4W35	Lake White Fish FSCA#4 Whole body Comp# 3 - 5 Fish	05374240	274696	G619-12-10
LW4W55	Lake White Fish FSCA#4 Whole body Comp# 4 - 5 Fish	05374242	274710	G619-12-11
WE6W65	Walleye FSCA#6 Whole body Comp# 6 - 5 fish * (analytical dup)	05424007	274609	G619-12-12
RH6F35	Rainbow Trout Hatchery FSCA#6 Comp# 3 5 -Fish Fillets Skin-on (L&R)	05424036	284838	G619-12-13
RH6F45	Rainbow Trout Hatchery FSCA#6 Comp# 4 5 -Fish Fillets Skin-on (L&R)	05424037	273449	G619-12-14
RH6F65	Rainbow Trout Hatchery FSCA#6 Comp# 6 5 - Fish Fillets Skin-on (L&R) *	05424038	274443	G619-12-15
RH6O15	Rainbow Trout Hatchery FSCA#6 Comp# 1 -5 Offals	05424039	274817	G619-12-16
RH6O25	Rainbow Trout Hatchery FSCA#6 Comp# 2 -5 Offals *	05424040	274830	G619-12-17
RH6O35	Rainbow Trout Hatchery FSCA#6 Comp# 3 -5 Offals	05424041	274844	G619-12-18
RH6O45	Rainbow Trout Hatchery FSCA#6 Comp# 3 -5 Offals	05424042	273443	G619-12-18
RH6O65	Rainbow Trout Hatchery FSCA#6 Comp# 6 -5 Offals *	05424043	274448	G619-12-20
RH3F15	Rainbow Trout Hatchery FSCA#3 Comp# 1 -5 Fish Fillets Skin-on (L&R)	05424016	274955	G619-13-1
RH3F25	Rainbow Trout Hatchery FSCA#3 Comp# 2 -5 Fish Fillets Skin-on (L&R)	05424017	274969	G619-13-2
RH3F35	Rainbow Trout Hatchery FSCA#3 Comp# 3 -5 Fish Fillets Skin-on (L&R)	05424018	274981	G619-13-3
RH3O15	Rainbow Trout Hatchery FSCA#3 Comp# 1 -5 Offals	05424019	274962	G619-13-4
RH3O25	Rainbow Trout Hatchery FSCA#3 Comp# 2 -5 Offals	05424020	274976	G619-13-5
RH3O35	Rainbow Trout Hatchery FSCA#3 Comp# 3 -5 Offals	05424021	274988	G619-13-6
RH4W15	Rainbow Trout Hatchery FSCA#4 Whole body Comp # 1 - 5 Fish	05424022	274995	G619-13-7
RH4W25	Rainbow Trout Hatchery FSCA#4 Whole body Comp # 2 - 5 Fish	05424023	274855	G619-13-8
RH4W35	Rainbow Trout Hatchery FSCA#4 Whole body Comp # 3 - 5 Fish	05424024	274864	G619-13-9
RH4W45	Rainbow Trout Hatchery FSCA#4 Whole body Comp # 4 - 5 Fish	05424025	274882	G619-13-10
RH4W55	Rainbow Trout Hatchery FSCA#4 Whole body Comp # 5 - 5 Fish	05424026	274890	G619-13-11
RH5W15	Rainbow Trout Hatchery FSCA#5 Whole body Comp # 1 - 5 Fish	05424027	274918	G619-13-12
RH5W25	Rainbow Trout Hatchery FSCA#5 Whole body Comp # 2 - 5 Fish	05424028	274925	G619-13-13
RH5W35	Rainbow Trout Hatchery FSCA#5 Whole body Comp # 3 - 5 Fish	05424029	274931	G619-13-14
RH5W45	Rainbow Trout Hatchery FSCA#5 Whole body Comp # 4 - 5 Fish	05424031	274938	G619-13-15
RH5W55	Rainbow Trout Hatchery FSCA#5 Whole body Comp # 5 - 5 Fish *	05424032	274947	G619-13-16
RH5W65	Rainbow Trout Hatchery FSCA#5 Whole body Comp # 6 - 5 Fish*	05424033	274904	G619-13-17
RH5W75	Rainbow Trout Hatchery FSCA#5 Whole body Comp # 6 - 5 Fish*	05424034	274803	G619-13-18
RH6F15	Rainbow Trout Hatchery FSCA#6 Comp#1 -5 Fish Fillets Skin-on (L& R)	05424035	274810	G619-13-19
RH6F25	Rainbow Trout Hatchery FSCA#6 Comp# 2 -5 Fish Fillets Skin-on (L& R) *	05424033	274823	G619-13-20
RW1F15	Rainbow Trout Wild FSCA#1 Comp#1 -5 Fish Fillets Skin-on (L &R)	05374245	274439	G619-16-1
RW1F25	Rainbow Trout Wild FSCA#1 Comp#2 -5 Fish Fillets Skin-on (L &R)	05374246	274419	G619-16-2
RW1F35	Rainbow Trout Wild FSCA#1 Comp#3 -5 Fish Fillets Skin-on (L &R)	05374247	274431	G619-16-3
RW1F45	Rainbow Trout Wild FSCA#1 Comp#4 -5 Fish Fillets Skin-on (L &R)	05374248	274315	G619-16-4
RW1F55	Rainbow Trout Wild FSCA#1 Comp#5 -5 Fish Fillets Skin-on (L &R) *	05374249	274323	G619-16-5
RW1F65	Rainbow Trout Wild FSCA#1 Comp#6 -5 Fish Fillets Skin-on (L &R) *	05374250	274344	G619-16-6
RW1O15	Rainbow Trout Wild FSCA#1 Comp# 1 -5 Offals	05374251	274301	G619-16-7
RW1O25	Rainbow Trout Wild FSCA#1 Comp# 2 -5 Offals	05374252	274410	G619-16-8

Field Sample Number	Sample Description	Region Tracking Number	CLP Tag Number	Paradigm Laboratory Number
RW1O35	Rainbow Trout Wild FSCA#1 Comp# 3 -5 Offals	05374253	274429	G619-16-9
RW1O45	Rainbow Trout Wild FSCA#1 Comp# 4 -5 Offals	05374254	274309	G619-16-10
MW1W15	Mountain Whitefish FSCA#1 Whole body Comp# 1 - 5 Fish	05374263	273720	G619-16-11
MW1W25	Mountain Whitefish FSCA#1 Whole body Comp# 2 - 5 Fish	05374264	273726	G619-16-12
MW1W35	Mountain Whitefish FSCA#1 Whole body Comp# 3 - 5 Fish	05374265	273732	G619-16-13
MW1W45	Mountain Whitefish FSCA#1 Whole body Comp# 4 - 5 Fish *	05374257	273739	G619-16-14
MW1W55	Mountain Whitefish FSCA#1 Whole body Comp# 5 - 5 Fish	05374258	276706	G619-16-15
MW1W65	Mountain Whitefish FSCA#1 Whole body Comp# 6 - 5 Fish *	05374261	273742	G619-16-16
MW1W75	Mountain Whitefish FSCA#1 Whole body Comp# 7 - 5 Fish *	05374262	276726	G619-16-17
RW2W15	Rainbow Trout Wild FSCA # 2 Whole body Comp# 1 - 5 Fish	05424044	273435	G619-17-1
RW2W25	Rainbow Trout Wild FSCA # 2 Whole body Comp# 2 - 5 Fish	05424045	274349	G619-17-2
RW2W35	Rainbow Trout Wild FSCA # 2 Whole body Comp# 3 - 5 Fish *	05424046	273707	G619-17-3
RW2W45	Rainbow Trout Wild FSCA # 2 Whole body Comp# 4 - 5 Fish	05424047	273560	G619-17-4
RW2W65	Rainbow Trout Wild FSCA # 2 Whole body Comp# 6 - 5 Fish *	05424048	273414	G619-17-5
RW2W75	Rainbow Trout Wild FSCA # 2 Whole body Comp# 5 - 5 Fish *	05424049	273413	G619-17-6
RW3F15	Rainbow Trout Wild FSCA #3 Comp# 1 - 5 Fish Fillets Skin-on (L&R)	05424050	273599	G619-17-7
RW3F25	Rainbow Trout Wild FSCA #3 Comp# 2 -5 Fish Fillets Skin-on (L&R)	05424051	273572	G619-17-8
RW3O15	Rainbow Trout Wild FSCA #3 Comp# 1 -5 Offals	05424052	273564	G619-17-9
RW3O25	Rainbow Trout Wild FSCA #3 Comp #2 - 5 Offals	05424053	273585	G619-17-10
RW5W15	Rainbow Trout Wild FSCA #5 Whole body Comp# 1 - 5 Fish	05424054	273580	G619-17-11
RW6F14	Rainbow Trout Wild FSCA #6 Comp# 1 - 5 Fish Fillets Skin-on (L&R)	05424055	273591	G619-17-12
RW6O14	Rainbow Trout Wild FSCA #6 Comp# 1 - 5 Offals	05424056	273597	G619-17-13
RW1O55	Rainbow Trout Wild FSCA #1 Comp# 1 - 5 Offals *	05374255	274325	G619-17-14
RW1O65	Rainbow Trout Wild FSCA #1 Comp# 6 - 5 Offals *	05374256	274337	G619-17-15
BB2W13	Burbot FSCA # 1 Comp# 1 - 3 Fish	05424057	276720	G619-18-1
BB2W23	Burbot FSCA # 1 Comp# 2 - 3 Fish	05424058	276747	G619-18-2
BB2W33	Burbot FSCA # 1 Comp# 3 - 3 Fish	05424059	276712	G619-18-3
BB3W15	Burbot FSCA# 3 Whole body Comp# 1 - 5 Fish	05424060	276741	G619-18-4
BB3W25	Burbot FSCA# 3 Whole body Comp# 2 - 5 Fish	05424061	277006	G619-18-5
BB3W35	Burbot FSCA# 3 Whole body Comp# 3 - 5 Fish *	05424062	277013	G619-18-6
BB3W45	Burbot FSCA# 3 Whole body Comp# 4 - 5 Fish	05424063	277032	G619-18-7
BB3W55	Burbot FSCA# 3 Whole body Comp# 5 - 5 Fish	05424064	277039	G619-18-8
BB3W65	Burbot FSCA# 3 Whole body Comp# 6 - 5 Fish *	05424065	277019	G619-18-9
BB3W75	Burbot FSCA# 3 Whole body Comp# 7 - 5 Fish *	05424066	277028	G619-18-10
BB4W15	Burbot FSCA# 4 Whole body Comp# 1 - 5 Fish	05424067	276904	G619-18-11
BB4W25	Burbot FSCA# 4 Whole body Comp# 2 - 5 Fish	05424068	276932	G619-18-12
BB4W35	Burbot FSCA# 4 Whole body Comp# 3 - 5 Fish	05424069	276939	G619-19-1
BB4W45	Burbot FSCA# 4 Whole body Comp# 4 - 5 Fish	05424070	276946	G619-19-2
BB5W15	Burbot FSCA# 5 Whole body Comp# 1 - 5 Fish	05424071	277125	G619-19-3
BB5W25	Burbot FSCA# 5 Whole body Comp# 2 - 5 Fish	05424072	277111	G619-19-4
BB5W35	Burbot FSCA# 5 Whole body Comp# 3 - 5 Fish	05424073	276911	G619-19-5
BB5W45	Burbot FSCA# 5 Whole body Comp# 4 - 5 Fish	05424074	276918	G619-19-6
BB5W55	Burbot FSCA# 5 Whole body Comp# 5 - 5 Fish	05424075	277104	G619-19-7
BB6W15	Burbot FSCA# 6 Whole body Comp# 1 - 5 Fish	05424076	277134	G619-19-8
BB6W25	Burbot FSCA# 6 Whole body Comp# 2 - 5 Fish	05424077	277148	G619-19-9
BB6W35	Burbot FSCA# 6 Whole body Comp# 3 - 5 Fish	05424078	277450	G619-19-10
BB6W45	Burbot FSCA# 6 Whole body Comp# 4 - 5 Fish	05424079	277442	G619-19-11
BB6W55	Burbot FSCA# 6 Whole body Comp# 5 - 5 Fish	05424080	277141	G619-19-12
LS1W25	Large Scale Sucker FSCA# 1 Whole body Comp# 2 - 5 Fish	05364221	276661	G619-20-1
LS1W35	Large Scale Sucker FSCA# 1 Whole body Comp# 3 - 5 Fish	05364222	276684	G619-20-2
LS1W45	Large Scale Sucker FSCA# 1 Whole body Comp# 4 - 5 Fish	05424081	276692	G619-20-3

Field Sample Number	Sample Description	Region Tracking Number	CLP Tag Number	Paradigm Laboratory Number
LS2W15	Large Scale Sucker FSCA# 2 Whole body Comp# 1 – 5 Fish	05414009	276697	G619-20-4
LS2W25	Large Scale Sucker FSCA# 2 Whole body Comp# 2 – 5 Fish *	05414010	277406	G619-20-5
LS2W35	Large Scale Sucker FSCA# 2 Whole body Comp# 3 – 5 Fish	05414011	277420	G619-20-6
LS2W45	Large Scale Sucker FSCA# 2 Whole body Comp# 4 – 5 Fish	05414012	277434	G619-20-7
LS2W65	Large Scale Sucker FSCA# 2 Whole body Comp# 6 – 5 Fish *	05414013	277416	G619-20-8
LS3W75	Large Scale Sucker FSCA# 3 Whole body Comp# 1 – 5 Fish *	05414014	277424	G619-20-9
LS3W15	Large Scale Sucker FSCA# 3 Whole body Comp# 2 – 5 Fish	05414015	276451	G619-20-10
LS3W25	Large Scale Sucker FSCA# 3 Whole body Comp# 3 – 5 Fish	05414016	276456	G619-20-11
LS3W35	Large Scale Sucker FSCA# 3 Whole body Comp# 4 – 5 Fish	05414017	276465	G619-20-12
LS3W55	Large Scale Sucker FSCA# 3 Whole body Comp# 5 – 5 Fish	05414018	276470	G619-20-13
LS4W15	Large Scale Sucker FSCA# 4 Whole body Comp# 1 – 5 Fish	05424080	276477	G619-20-14
LS4W25	Large Scale Sucker FSCA# 4 Whole body Comp# 2 – 5 Fish	05414019	276482	G619-20-15
LS4W35	Large Scale Sucker FSCA# 4 Whole body Comp# 3 – 5 Fish	05424083	276757	G619-21-1
LS4W45	Large Scale Sucker FSCA# 4 Whole body Comp# 4 – 5 Fish	05424084	**	G619-21-2
LS4W55	Large Scale Sucker FSCA# 4 Whole body Comp# 5 – 5 Fish	05424085	276763	G619-21-3
LS5W15	Large Scale Sucker FSCA# 5 Whole body Comp# 1 – 5 Fish	05424086	276776	G619-21-4
LS5W25	Large Scale Sucker FSCA# 5 Whole body Comp# 2 – 5 Fish	05424087	276786	G619-21-5
LS5W35	Large Scale Sucker FSCA# 5 Whole body Comp# 3 – 5 Fish	05424088	276569	G619-21-6
LS5W45	Large Scale Sucker FSCA# 5 Whole body Comp# 4 – 5 Fish	05424089	276593	G619-21-7
LS5W55	Large Scale Sucker FSCA# 5 Whole body Comp# 5 – 5 Fish	05424090	276574	G619-21-8
LS6W15	Large Scale Sucker FSCA# 6 Whole body Comp# 1 – 5 Fish	05424091	276581	G619-21-9
LS6W25	Large Scale Sucker FSCA# 6 Whole body Comp# 2 – 5 Fish	05424092	276589	G619-21-10
LS6W35	Large Scale Sucker FSCA# 6 Whole body Comp# 3 – 5 Fish *	05424093	276793	G619-21-11
LS6W45	Large Scale Sucker FSCA# 6 Whole body Comp# 4 – 5 Fish	05424094	276800	G619-21-12
LS6W65	Large Scale Sucker FSCA# 6 Whole body Comp# 6 – 5 Fish *	05424095	277219	G619-21-13
LS6W75	Large Scale Sucker FSCA# 6 Whole body Comp# 7 – 5 Fish *	05424096	277204	G619-21-14
RW2W53	Rainbow Trout Wild FSCA #2 Whole body Comp# 5 – 3 Fish	05414008	276555	G619-21-15

* Duplicate/Triplicate

** - Not Received by Paradigm Lab

NA – Not Analyzed

A holding time summary cross-checking the chain or custody, fish processing and sample integrity traceability listing the fish tissue samples collected and analyzed for dioxins/furans with the corresponding Region 10 sample tracking numbers, field sample numbers and laboratory identification numbers and dates of collection, homogenization and verified time of sample receipt (VTSR) at the different laboratories, extraction and analyses are summarized in Table 1 and Appendix B at the end of this validation report.

DATA QUALIFICATIONS

The following comments refer to the laboratory performance in meeting the Quality Control specifications outlined in the Phase 1 Fish Tissue Sampling Quality Assurance Project Plan (QAPP) for the Upper Columbia River Site CERCLA RI/FS, the Contract Laboratory Program’s (CLP) Statement of Work (SOW) for the Multi-Media, Multi-Concentration Dioxins and Furans Analysis (DLM02.0) and the Project - Modified Analysis and Flexibility Clause. Some of the data quality elements were qualified using the reviewer’s professional judgment.

The conclusions presented herein are based on the information provided for the review.

Field Sample Collection

The fish tissue sample collection was accomplished through a multi-agency/tribal effort with the CH2MHill team as the overall lead in the field. Sample vessels and vessel operators were provided by the following tribal and federal agencies under an interagency or sub-contracting agreement with EPA and/or CH2MHill: Spokane Tribe of Indians, Confederated Tribes of the Colville Reservation, US Fish and Wildlife Services and the USEPA Investigation and Engineering Unit of the Office of Environmental Assessment.

The sample collection dates were based on the fish availability and fish species' spawning season. There were two sample collection events conducted, the first one was conducted in September 2005 and the second one was in October 2005. The fish species that were collected from the designated fish sample collection areas (FSCA 1 through 6) were Walleye (*Sander vitreus*), Rainbow trout (*Oncorhynchus mykiss*), Lake white fish (*Coregonus clupeaformis*), Large-scale sucker (*Catostomas macrocheilus*), and Burbot (*Lota lota*). Long-nose suckers and Mountain whitefish were not originally listed in the QAPP as target fish species but were also collected and added to the target fish species due to their availability in the FSCAs. The mountain white fish were analyzed while the long-nose suckers were archived. The rainbow trout samples were grouped into three categories – wild, hatchery and mixed wild and hatchery. Only the wild and hatchery rainbow trouts were analyzed for the compounds of concern. The mixed wild and hatchery rainbow trouts were archived for future analysis, if needed.

The fish samples were generally collected using gill nets, electro-fishing, burbot traps and angling, if necessary. The field sample collection process was audited by the project's EPA and CH2MHill QA Managers. There were no significant problems encountered during sample collection, on-site processing, sampling documentation and sample shipment.

Sample Processing and Chain-of-Custody Documentation

CH2MHill set-up a trailer dedicated for the on-site fish sample processing which included visual inspection of the fish, sex determination, conducting field measurements (fish length and weight) and otolith, scale and opercular covers (large scale suckers only) removal for subsequent fish age determination by the Washington State Department of Fish and Wildlife (WSDFW). All of the field forms generated for these measurements and determination were evaluated and cross-checked with the homogenization forms and chain-of-custody (COC) documentation. All of the field measurements, field sampling documentation (COCs) and sample preservation (freezing to -20C) were conducted by CH2MHill within 24 hours of sample collection.

Frozen whole fish samples were shipped to CH2MHill laboratory - Applied Science Laboratory (ASL) located in Corvallis, OR for filleting (if needed), homogenization, compositing, aliquot distribution and storage. There were four types of tissues prepared and analyzed for the compounds of potential concern (COPCs) for the sites, namely: fillets, left and right side with skin-on, offals (remaining tissue, internal organs and fish bones after filleting), guts (for large scale sucker only) and whole body (includes fish head, skin and entrails).

As specified in the EPA approved site QAPP, the following tissue types and homogenates were prepared by ASL and shipped to USEPA Manchester Environmental Laboratory (MEL) for subsequent PCB Aroclor, metals, percent lipids and speciated arsenic analyses and/or archival:

- Walleye – fillets and offals at three FSCAs and whole body composites from three FSCAs
- Rainbow trout (wild and hatchery) – fillets and offals at three FSCAs and whole body composites from three FSCAs
- Lake whitefish – whole body composites
- Mountain whitefish – whole body composites
- Large scale sucker – whole body composites for organics; guts/internal organs composites-
metals
- Burbot – whole body composites
- Long-nose suckers – whole body composites (archived)

Some discrepancies and missing information were noted on the composite sample numbers listed on the COCs and the fish processing forms. ASL (represented by Mr. Robert Wong) and CH2MHill QA Manager, (Ms. Artemis Antipas) were contacted to clarify and correct these discrepancies on April 26, 2006. An explanation and reasons for the discrepancies were immediately sent to the reviewer. See the attached communication logs at the end of this validation memo.

Sample Homogenization and Compositing

Each individual fish collected was given identification number, tagged and individually homogenized at ASL using a commercial grade stainless steel blender/grinder (Robo-Coupe Blixer 6) with liquid nitrogen. Equal amounts of homogenized whole body, fillet or offal tissue samples were mixed and composited to form a single sample. The homogenization forms and the resulting fish sample composites were evaluated by this reviewer. There were no discrepancies noted between the sample collection forms, homogenization forms and the sample composite COCs. Fillet and whole body samples included the fish skin. Care was taken to prevent cross-contamination between sample homogenates. Prior to the start of the project samples, the homogenization process was audited by the project's EPA and CH2MHill QA Managers. To monitor processing cross-contamination, proof blanks were collected at the QAPP specified frequency and sent to the Contract Laboratory program (CLP) for the analysis of the project target compound.

Corrective Action: Deviation from the QAPP as a result of field and sample processing assessment: In a mock sample processing and homogenization conducted during the EPA's and CH2MHill's QA lab audit, it was found out that otoliths were very hard to remove when the fish samples were already frozen. In addition, subjecting the fish to freezing and defrosting ruptures the internal organs, make the fish muscles mushy and thus made the separation of fillets from the offals quite a challenge. To avoid cross-contamination of the fish tissue samples with the offals and to better preserve the otoliths, it was agreed

by the project team that the removal of otolith will be conducted on-site after field measurements and before sample preservation (freezing to -20C) and if bench space and resources will allow, filleting of fish samples will also be performed on-site prior to freezing the samples.

Fish Age Determination and Range

The following methods were used to determine the age of the fish: otoliths (inner ear of a fish) were used to determine the age of Lake whitefish, burbot and mountain whitefish. Both otoliths and scales were used to determine the age of the walleye, wild and hatchery rainbow trouts. The opercular covers (also called opercula) were used determine the age of large-scale suckers.

Otolith, scales and operculas were read with the knowledge of the place of capture, sex and size of the fish. The readings were performed by only one individual Mr. John Sneva of WSDFW. Precision and consistency of readings were checked through the comparison of annuli (otoliths) and the occuli (scales) readings when both specimens are available. The fish age are based on visual readings (duplicate readings, if additional otoliths or scales were available) and maybe estimated range. There was no second party independent readings or validation conducted with these age estimates.

Fish age logs indicated the following: age of lake whitefish ranged from 1-3 years old; hatchery rainbow trouts ranged mostly 1-2 years; wild rainbow trouts from 1-4 years old; mountain whitefish ranged from 0-15 years old; large-scale suckers nine of which were <10 years old; the rest ranged from >10-36 years of age; walleye and burbot from 2-5 years old.

Sample Receipt and Storage

All of the samples were received frozen and intact at the USEPA MEL from CH2MHill lab. After inspection, inventory and logging-in, the sample homogenates were stored in a freezer at -20C while waiting for extraction and analysis. The samples evaluated in this validation report were shipped by MEL to Paradigm Lab from January 26, 2006 to March 24, 2006. All of the fish tissue samples were received intact and still frozen at the verified time of sample receipt (VTSR) at Paradigm Labs.

Holding Times - Acceptable

All of the fish tissue samples were frozen at -20 °C while on storage at MEL and samples were still frozen when received at Paradigm Lab. The integrity of the samples was preserved while on storage at MEL and during shipment. All of the sample analyses met the contractual extraction and contractual analytical holding times of 10 days from the VTSR and 30 days from the extraction date, respectively. All of the samples also met the method and project extraction and analytical holding times of one year from the date of sample collection. None of the dioxin or furan data were qualified on this basis. The list of samples, cross-referenced to the fish species, station location, and the dates of sample collection, VTSR at the lab, extraction, extract clean-up and analysis dates are listed in Table 1 at the end of this report. The list of individual fish comprising a composite sample can be found in Appendix B of this report.

Sample Preparation - Acceptable

All of the samples were extracted following the specifications of the flexibility clause and the DLM02.0 SOW. The primary extracts went through special clean-up processes specified in the SOW to remove the chlorinated diphenyl ethers (CDPEs) and other organic material interferences in the extract. The efficiency of the clean-up process was monitored by the recoveries of the clean-up standard, ³⁷Cl₄-2,3,7,8-TCDD.

Instrument Performance Checks - Acceptable

The primary sample analyses were conducted using the DB-5 column. For tetrachlorodibenzofuran (TCDF) confirmation, additional runs were performed on a DB-225 column. Both columns met the isomer specificity requirements for 2, 3, 7, 8- tetrachlorodibenzo-p-dioxin (TCDD), TCDF and other PCDD/PCDF isomers. The frequency of analysis, minimum resolving power of >10,000, signal-to-noise (S/N) ratios, ion abundance ratios, retention times and the % valley criteria were also met at the beginning and end of each analytical sequence. The appropriate switching times for the Selected Ion Current Profile (SICP) descriptors and the chromatographic resolutions were established from the first and last eluting isomers per descriptor. The chromatographic separations between the isomer eluting closest to 2,3,7, 8-TCDD and 2,3,7,8-TCDF as expressed by percent (%) valley were all less than 25% for both columns used. The absolute retention times (RTs) of the internal standard ¹³C₁₂- 2, 3, 7, 8-TCDD were greater than 25 minutes. Homologues do not overlap between homologue descriptors switching times. The instrument used remained stable throughout each analytical sequence. None of the data were qualified on this basis.

Initial Calibrations - Acceptable

The frequency of analysis of the initial calibrations (ICALs) in both columns DB5 and DB225 were met. The instruments' resolutions of >10,000 resolving power were maintained throughout the course of all the analytical sequences. The percent relative standard deviations (%RSDs) for all the native and isotope-labeled compounds in all ICALs were less than 20%. The chromatographic separations between the isomer eluting closest to 2, 3, 7, 8-TCDD and 2, 3, 7, 8-TCDF as expressed by percent (%) valley were all less than 25%. The absolute retention times (RTs) of the internal standard ¹³C₁₂- 1,2,3,4-TCDD were greater than 25 minutes in the primary column, DB-5 and >15 minutes in the confirmatory column, DB225. All of the calibration standards were analyzed at the concentrations specified by the flexibility clause, the S/N ratios are >10 including the lowest standard (CS0), the ion abundance ratios and relative retention times (RRTs) in reference to both ¹³C-1,2,3,4-TCDD and ¹³C-1,2,3,7,8,9-HxCDD as internal standards were within the control limits. None of the reported results were qualified on this basis.

Continuing Calibrations - Acceptable

All of the continuing calibration verification standards (CCVs) associated with the samples met the criteria for frequency of analysis, mass resolutions, ion abundance ratios, isomer specificity, absolute RTs, RRTs in reference to both ¹³C-1,2,3,4-TCDD and ¹³C-1,2,3,7,8,9-HxCDD as internal standards, the chromatographic resolutions, the S/N ratios >10 and the percent differences (%D) of the daily response factors (RF) as compared to the mean RF from the ICALs. None of the data were qualified on this basis.

On-Going Precision and Recovery (OPR) - Acceptable

The frequency of analysis and recovery criteria were met by all OPRs extracted and analyzed with the samples. None of the data were qualified on this basis.

Compound Quantitation and Detection Limits

All of the samples were analyzed at the project-required concentration levels. All of the samples were extracted at the project specified amount. All of the target compounds detected in the samples were calculated off the primary column using the mean relative response factors (RRF) from the initial calibrations and were at concentrations within the instrument's calibration range. All of the 2,3,7,8-TCDFs initially detected in the DB-5 primary column were re-analyzed on a second confirmatory column (DB-225). All 2, 3, 7, 8-TCDF detections were calculated off the DB-5 column because of the chlorinated diphenyl ether (CDPE) interference in the DB225 column.

Target compounds detected at concentrations less than the laboratory specified reporting limits were qualified estimated, "J". The reporting limits for some of the PCDDs/PCDFs detected in the samples were elevated and qualified non-detects due to interferences and contamination in the laboratory blank(s).

Detected compounds that met all of the identification criteria except for the mass ion abundance ratios were reported at the level of concentration detected (estimated maximum potential concentration-EMPC) and were qualified non-detects, "U", by this reviewer.

Compound Identification

Majority of the reported PCDDs/PCDFs met the technical acceptance criteria for compound identifications, i.e., S/N ratios greater than 2.5, ion abundance ratios, RRTs using both $^{13}\text{C}_{12}$ -1,2,3,4-TCDD and $^{13}\text{C}_{12}$ - 1,2,3,7,8,9-HxCDD as injection standards within the method specified limits and chromatographic resolutions. All of the reported results did not co-elute with CDPE's. Where co-elution with CDPEs were identified, the results were qualified as non-detects, "U", by this reviewer and reported at the level of detection due to interferences. Some of the PCDD/PCDF target compounds identified did not meet the ion abundance ratio criteria and were flagged as non-detects at the EMPC. Most of these compounds were also identified as contaminants in the laboratory blank (s).

Method Blanks

The frequency of analysis of laboratory blank was met. Trace levels of some of the target compounds were detected in the method blank and were qualified as follows in the associated samples: detections at concentrations >5x the value in their respective blank(s) were qualified non-detects, "U"; detection >5x the value in the blank(s) were not qualified on this basis. The following samples were qualified based on the contamination in the blank:

Method Blank	Extraction date	Compounds Detected	Amount Detected (pg/g)	Affected samples
LMB12330	01/29/06	1,2,3,7,8-PeCDD	0.192	All samples in the SDG: G619-9.
		2,3,7,8-TCDF	0.08	
		1,2,3,7,8-PeCDF	0.204	
		2,3,4,7,8-PeCDF	0.190	
		1, 2,3,6,7,8-HxCDF	0.150	
		2,3,4,6,7,8-HxCDF	0.174	
		Total TCDFs	0.08	
		Total PeCDDs	0.192	
		Total PeCDFs	0.394	
		Total HxCDFs	0.324	
		LMB12351	02/09/06	
OCDD	2.41			
2,3,7,8-TCDF	0.132			
1,2,3,7,8-PeCDF	0.0760			
2,3,4,7,8-PeCDF	0.0960			
1,2,3,4,7,8-HxCDF	0.0760			
1,2,3,6,7,8-HxCDF	0.0760			
2,3,4,6,7,8-HxCDF	0.0780			
1,2,3,7,8,9-HxCDF	0.0800			
1,2,3,4,6,7,8-HpCDF	0.0680			
OCDF	0.446			
Total TCDFs	0.290			
Total PeCDFs	0.132			
Total HxCDDs	0.172			
Total HxCDFs	0.310			
Total HpCDDs	0.156			
LMB 12353	02/12/06	1,2,3,4,6,7,8-HpCDD	0.222	All sample in the SDG: G19-11
		OCDD	1.70	
		2,3,7,8-TCDF	0.150	
		1,2,3,7,8-PeCDF	0.114	
		2,3,4,7,8-PeCDF	0.102	
		1,2,3,4,6,7,8- HpCDF	0.060	
		OCDF	0.140	
		Total HpCDDs	0.134	
		Total TCDFs	0.150	
		Total PeCDFs	0.216	
		Total HpCDFs	0.180	
LMB12366	03/14/06	OCDD	1.19	All samples in the SDG: G619-16.
		2,3,4,7,8-PeCDD	0.108	
		Total PeCDD	0.108	
LMB12395	03/21/06	1,2,3,7,8- PeCDD	0.210	All samples in the SDG: G619-17
		1,2,3,7,8 – PeCDF	0.250	
		2,3,4,7,8-PeCDF	0.192	
		1,2,3,4,7,8-HxCDF	0.164	
		1,2,3,6,7,8-HxCDF	0.206	
		2,3,4,6,7,8 – HxCDF	0.222	
		1,2,3,7,8,9 - HxCDF	0.302	
		Total PeCDDs	0.210	
		Total PeCDFs	0.442	
Total HxCDFs	0.894			
LMB12400	03/22/06	1,2,3,7,8-PeCDD	0.176	All samples in the SDG: G619-18
		1,2,3,7,8,9 - HxCDD	0.208	

Method Blank	Extraction date	Compounds Detected	Amount Detected (pg/g)	Affected samples
		1,2,3,4,6,7,8 – HpCDD	0.210	
		OCDD	0.944	
		2,3,7,8- TCDF	0.170	
		1,2,3,7,8-PeCDF	0.194	
		2,3,4,7,8-PeCDF	0.238	
		1,2,3,4,7,8 – HxCDF	0.196	
		1,2,3,6,7,8-HxCDF	0.176	
		2,3,4,6,7,8- HxCDF	0.142	
		1,2,3,7,8,9 – HxCDF	0.212	
		1,2,3,4,6,7,8 – HpCDF	0.178	
		1,2,3,4,7,8,9 – HpCDF	0.188	
		OCDF	0.518	
		Total PeCDDs	0.176	
		Total HxCDDs	0.560	
		Total HpCDDs	0.210	
		Total PeCDFs	0.432	
		Total HxCDFs	0.726	
		Total HpCDFs	0.366	
LMB12403	03/24/06	1,2,3,7,8-PeCDD	0.144	All samples in the SDG: G619-19
		1,2,3,7,8,9 - HxCDD	0.192 (EMPC)	
		OCDD	0.910	
		2,3,7,8- TCDF	0.252 (EMPC)	
		1,2,3,7,8-PeCDF	0.238	
		2,3,4,7,8-PeCDF	0.200 (EMPC)	
		1,2,3,4,7,8 – HxCDF	0.140	
		1,2,3,6,7,8-HxCDF	0.164	
		2,3,4,6,7,8- HxCDF	0.136	
		1,2,3,4,6,7,8 – HpCDF	0.116	
		1,2,3,4,7,8,9 – HpCDF	0.124	
		Total PeCDDs	0.144	
		Total PeCDFs	0.238	
		Total HxCDFs	0.576	
Total HpCDFs	0.240			
LMB12410	03/30/06	1,2,3,7,8-PeCDF	0.188	All samples in the SDG: G619-20
		Total PeCDFs	0.188	
LMB12434	04/2/106	1,2,3,4,6,7,8-HpCDD	0.240	All samples in SDG: G619-21
		OCDD	1.11	
		1,2,3,7,8-PeCDF	0.088	
		2,3,4,7,8-PeCDF	0.124	

Field Duplicates/Triplicates

Field duplicates/triplicates were submitted blind to the laboratory for analysis. The decision to submit a duplicate or triplicate depends on the availability of fish tissue or offal homogenates. The following sample pairs or triplicates were analyzed for dioxins and furans:

Sample Name	Paradigm Lab Sample No	Detected Compounds	Concentrations (picogram/gram)	RPD/RSDs	Validation Qualifiers
LW3W25	G619-10-15	2,3,7,7-TCDF	3.67	7.2%	No qualifiers
LW3W65	G619-12-7		3.95		
LW3W75	G619-12-8		3.42		

Sample Name	Paradigm Lab Sample No	Detected Compounds	Concentrations (picogram/gram)	RPD/RSDs	Validation Qualifiers
WE2W55	G619-10-1	2,3,7,8- TCDF	1.13	5.8%	No qualifiers
WE2W65	G619-10-2		1.08		
WE2W75	G619-10-3		1.21		
MW1W45	G619-16-14	2,3,7,8- TCDF	3.39	2.6%	No qualifiers
MW1W65	G619-16-16		3.57		
MW1W75	G619-16-17		3.48		
LS2W25	G619-20-5				No qualifiers
LS2W65	G619-20-8				
LS2W75	G619-20-9				
LS6W35	G619-21-11	2,3,7,8-TCDF	3.86	3.5%	No qualifiers
LS6W65	G619-21-13		3.65		
LS6W75	G619-21-14		3.62		
RW2W35	G619-17-4	2,3,7,8- TCDF	3.95	10.8%	No qualifiers
RW2W65	G619-17-5		3.28		
RW2W75	G619-17-6		3.31		
RH5W55	G619-13-16	2,3,7,8-TCDF	1.42	7.5%	No qualifiers
RH5W65	G619-13-17		1.58		
RH5W75	G619-13-18		1.49		
RH6F25	G619-13-20	Trace 2,3,7,8-TCDF & OCDD	NA	NA	No qualifiers
RH6F65	G619-12-15	All NDs			
RH6O25	G619-12-17	2,3,7,8 – TCDF	2.07		
RH6O65	G619-12-20		2.24		
RW1F55	G619-16-5	2,3,7,8-TCDF	0.631 J	0.2 RPD	No qualifiers
RW1F65	G619-16-6		0.632 J		
RW1O55	G619-17-14	2,3,7,8-TCF	1.67	5.3 RPD	No qualifiers
RW1O65	G619-17-15		1.76		
BB3W65	G619-18-9	2,3,7,8-TCDF	3.09	7.1 %	No qualifiers
BB3W75	G619-18-10		3.21		
BB3W35	G619-18-6		2.79		

Toxicity Equivalence Quotients (TEQs)

TEQs were calculated and reported by the laboratory using the World Health Organization Toxicity Equivalent Factors (WHO TEFs) for the detected compounds and a 0 multiplier for the non-detected compounds. The detected dioxins and furans that were qualified as non-detect by the reviewer were not included in the re-calculated total TEQs. Report the re-calculated TEQs.

Analytical Sequence - Acceptable

All of the standards, blanks, samples and QC samples were analyzed in accordance with the method specified analytical sequence. Mass ion locks and resolution and window defining mix were analyzed and checked at the beginning and end of each analytical sequence. All of the analytical sequences were also bracketed by the continuing calibration check standards. None of the data were qualified on this basis.

Internal Standards Recoveries - Acceptable

All of the applicable technical acceptance criteria for the internal standards were met by all analyses performed. None of the data were qualified on this basis.

Surrogate Recoveries - Not Applicable

Surrogate was not required for this method. However, clean-up standard 37Cl-2, 3, 7, 8-TCDD was added to all samples and QC samples. The clean-up standard recoveries were acceptable for all analyses.

Laboratory Contact

The laboratory was contacted for this review to re-submit deliverable data forms that needed corrections. Paradigm responded immediately. All of the corrected forms were received prior to completion of this review.

Overall Assessment

All of the samples were analyzed in accordance with the method specifications. There were no significant problems found with the data. The data, as qualified, are acceptable and can be used for all purposes.

Data Qualifiers	
U	The analyte was not detected at or above the reported numeric result.
J	The analyte was positively identified. The associated numerical result is an estimate.
UJ	The analyte was not detected at or above the reported estimated result. The associated numerical value is an estimate of the quantitation limit of the analyte in this sample.
R	The data are unusable for all purposes.
N	There is evidence the analyte is present in this sample.
JN	There is evidence that the analyte is present. The associated numerical result is an estimate.

TABLE 1- SUMMARY OF HOLDING TIMES



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

May 15, 2006

Reply to
Attn of: **MGREPOGR**
OEA-095

MEMORANDUM

Subject: Data Validation Report for the Polychlorinated Biphenyl (PCB) Congener Analysis of the Fish Tissue Samples Collected for the Phase I Upper Columbia River (UCR) Remedial Investigation/Feasibility Study (RI/FS) September 2005

From: Ginna Grepo-Grove, Senior Chemist
Technical Support Unit, OEA

To: Sally Thomas, RPM, UCR, Fish Tissue Study
USEPA, ECL

Marc Stifelman, Human Health Risk Assessment, USEPA, OEA
Burt Shephard, Ecological Risk Assessment, USEPA, OEA
Jim Stefanoff, Project Manager, CH2MHill
Artemis Antipas, QA Manager, CH2MHill

The quality assurance (QA) review of 38 fish tissue samples collected from the above referenced site has been completed. These samples were analyzed for 209 full PCB congener list in accordance with the Contract Laboratory Program's (CLP) *Statement of Work (SOW) for the Analysis of Chlorinated Biphenyl (CB) Congeners, Multi-Media, Multi-Concentration (CBC01.0 Revision May, 2005)* and the Method 1668A, "Chlorinated Biphenyl Congeners in Water, Soil and Tissue by HRGC/HRMS, December 1999" by Paradigm Analytical Laboratory of Wilmington NC.

The following fish tissue samples were evaluated in this report:

Field Sample Number	Sample Description	Region Tracking Number	CLP Tag Number	Paradigm Laboratory Number
WE1F45	Walleye FSCA#1 Comp# 4 - 5 Fish Fillet Skin-on (R & L)	05364204	274186	G619-9-4
WE1F55	Walleye FSCA#1 Comp# 5 - 5 Fish Fillet Skin-on (R & L)	05364205	274199	G619-9-5
WE1O45	Walleye FSCA#1 Comp# 4 - 5 Offals	05364209	274193	G619-9-9
WE3F25	Walleye FSCA#3 Comp# 2 - 5 Fish Fillet Skin-on (R & L)	05374201	274374	G619-9-12
RH5W55	Rainbow Trout Hatchery FSCA#5 Whole body Comp # 5 - 5 Fish	05424032	274949	G619-14-2
RH6F35	Rainbow Trout Hatchery FSCA#6 Comp# 3 5 -Fish Fillets Skin-on (L&R)	05424036	274841	G619-14- 3
RH6O35	Rainbow Trout Hatchery FSCA#6 Comp# 3 -5 Offals	05424041	274848	G619-14- 4
RW1F25	Rainbow Trout Wild FSCA#1 Comp#2 -5 Fish Fillets Skin-on (L &R)	05374246	274418	G619-14- 5

Field Sample Number	Sample Description	Region Tracking Number	CLP Tag Number	Paradigm Laboratory Number
RW1O25	Rainbow Trout Wild FSCA#1 Comp# 2 -5 Offals	05374252	274412	G619-14- 6
RW2W45	Rainbow Trout Wild FSCA # 2 Whole body Comp# 4 - 5 Fish	05424047	273401	G619-14- 7
RW3F15	Rainbow Trout Wild FSCA #3 Comp# 1 - 5 Fish Fillets Skin-on (L&R)	05424050	273405	G619-14- 8
RW3O15	Rainbow Trout Wild FSCA #3 Comp# 1 -5 Offals	05424052	273556	G619-14- 9
RW5W15	Rainbow Trout Wild FSCA #5 Whole body Comp# 1 - 5 Fish	05424054	273578	G619-14- 10
WE2W55	Walleye FSCA#2 Whole body Comp# 5 - 5 fish*	05414005	284371	G619-14- 11
BB2W33	Burbot FSCA # 1 Comp# 3 - 3 Fish	05424059	276713	G619-14- 12
BB3W35	Burbot FSCA# 3 Whole body Comp# 3 – 5 Fish	05424062	277012	G619-14- 13
BB4W25	Burbot FSCA# 4 Whole body Comp# 2 – 5 Fish	05424068	276933	G619-14- 14
BB5W55	Burbot FSCA# 5 Whole body Comp# 5 – 5 Fish	05424075	277103	G619-14- 15
BB6W55	Burbot FSCA# 6 Whole body Comp# 5 – 5 Fish	05424080	277142	G619-14- 16
LS1W35	Large Scale Sucker FSCA# 1 Whole body Comp# 3 – 5 Fish	05364222	276680	G619-14- 17
LS2W45	Large Scale Sucker FSCA# 2 Whole body Comp# 4 – 5 Fish	05414012	277427	G619-14- 18
LS3W25	Large Scale Sucker FSCA# 3 Whole body Comp# 3 – 5 Fish	05414016	276455	G619-14- 19
LS4W55	Large Scale Sucker FSCA# 4 Whole body Comp# 5 – 5 Fish	05424085	26761	G619-14- 20
LS5W25	Large Scale Sucker FSCA# 5 Whole body Comp# 2 – 5 Fish	05424087	276783	G619-15-1
LS6W25	Large Scale Sucker FSCA# 6 Whole body Comp# 2 – 5 Fish	05424092	276587	G619-15-2
LW2W55	Lake White Fish FSCA#2 Whole body Comp# 5 - 5 Fish	05424013	274093	G619-15- 3
LW3W25	Lake White Fish FSCA#3 Whole body Comp# 2 - 5 Fish	05374229	274575	G619-15- 4
LW4W25	Lake White Fish FSCA#4 Whole body Comp# 2 - 5 Fish	05374239	274561	G619-15- 5
LW5W45	Lake White Fish FSCA#5 Whole body Comp# 4 - 5 Fish	05374237	274738	G619-15- 6
LW6W13	Lake White Fish FSCA#6 Whole body Comp# 1 - 3 Fish	05374243	274100	G619-15- 7
MW1W15	Mountain Whitefish FSCA#1 Whole body Comp# 1 - 5 Fish	05374263	273718	G619-15- 8
RH3F35	Rainbow Trout Hatchery FSCA#3 Comp# 3 -5 Fish Fillets Skin-on (L&R)	05424018	274985	G619-15- 9
RH3O35	Rainbow Trout Hatchery FSCA#3 Comp# 3 -5 Offals	05424021	274992	G619-15- 10
RH4W45	Rainbow Trout Hatchery FSCA#4 Whole body Comp # 4 - 5 Fish	05424025	274886	G619-15- 11
WE3O25	Walleye FSCA#3 Comp# 2 - 5 Offals	05374206	274955	G619-15- 12
WE4W45	Walleye FSCA#4 Whole body Comp# 4 -5 fish	05374215	274477	G619-15- 13
WE5W25	Walleye FSCA#5 Whole body Comp# 2 - 5 fish	05424002	274617	G619-15- 14
WE6F35	Walleye FSCA#6 Comp# 3 - 5 Fish Fillet Skin-on (R & L)	05374219	274522	G619-15- 15
WE6O35	Walleye FSCA#6 Comp# 3 - 5 Offals	05374224	274529	G619-15- 16

DATA QUALIFICATIONS

The following comments refer to the laboratory performance in meeting the Quality Control specifications outlined in the Phase 1 Fish Tissue Sampling Quality Assurance Project Plan (QAPP) for the Upper Columbia River Site CERCLA RI/FS, the Contract Laboratory Program’s (CLP) Statement of Work (SOW) for the Multi-Media, Multi-Concentration CB Congener Analysis (CB01.0) and the Project - Modified Analysis and Flexibility Clause. Some of the data quality elements were qualified using the reviewer’s professional judgment.

The conclusions presented herein are based on the information provided for the review.

Field Sample Collection

The fish tissue sample collection was accomplished through a multi-agency/tribal effort with the CH2MHill team as the overall lead in the field. Sample vessels and vessel operators were provided by the following tribal and federal agencies under an interagency or sub-contracting agreement with EPA and/or CH2MHill: Spokane Tribe of Indians, Confederated Tribes of the Colville Reservation, US Fish and

Wildlife Services and the USEPA Investigation and Engineering Unit of the Office of Environmental Assessment.

The sample collection dates were based on the fish availability and fish species' spawning season. There were two sample collection events conducted, the first one was conducted in September 2005 and the second one was in October 2005. The fish species that were collected from the designated fish sample collection areas (FSCA 1 through 6) were Walleye (*Sander vitreus*), Rainbow trout (*Oncorhynchus mykiss*), Lake white fish (*Coregonus clupeaformis*), Large-scale sucker (*Catostomas macrocheilus*), and Burbot (*Lota lota*). Long-nose suckers and Mountain whitefish were not originally listed in the QAPP as target fish species but were also collected and added to the target fish species due to their availability in the FSCAs. The mountain white fish were analyzed while the long-nose suckers were archived. The rainbow trout samples were grouped into three categories – wild, hatchery and mixed wild and hatchery. Only the wild and hatchery rainbow trouts were analyzed for the compounds of concern. The mixed wild and hatchery rainbow trouts were archived for future analysis, if needed.

The fish samples were generally collected using gill nets, electro-fishing, burbot traps and angling, if necessary. The field sample collection process was audited by the project's EPA and CH2MHill QA Managers. There were no significant problems encountered during sample collection, on-site processing, sampling documentation and sample shipment.

Sample Processing and Chain-of-Custody Documentation

CH2MHill set-up a trailer dedicated for the on-site fish sample processing which included visual inspection of the fish, sex determination, conducting field measurements (fish length and weight) and otolith, scale and opercular covers (large scale suckers only) removal for subsequent fish age determination by the Washington State Department of Fish and Wildlife (WSDFW). All of the field forms generated for these measurements and determination were evaluated and cross-checked with the homogenization forms and chain-of-custody (COC) documentation. All of the field measurements, field sampling documentation (COCs) and sample preservation (freezing to -20C) were conducted by CH2MHill within 24 hours of sample collection.

Frozen whole fish samples were shipped to CH2MHill laboratory - Applied Science Laboratory (ASL) located in Corvallis, OR for filleting (if needed), homogenization, compositing, aliquot distribution and storage. There were four types of tissues prepared and analyzed for the compounds of potential concern (COPCs) for the sites, namely: fillets, left and right side with skin-on, offals (remaining tissue, internal organs and fish bones after filleting), guts (for large scale sucker only) and whole body (includes fish head, skin and entrails).

As specified in the EPA approved site QAPP, the following tissue types and homogenates were prepared by ASL and shipped to USEPA Manchester Environmental Laboratory (MEL) for subsequent PCB Aroclor, metals, percent lipids and speciated arsenic analyses and/or archival:

- Walleye – fillets and offals at three FSCAs and whole body composites from three FSCAs
- Rainbow trout (wild and hatchery) – fillets and offals at three FSCAs and whole body composites from three FSCAs
- Lake whitefish – whole body composites

- Mountain whitefish – whole body composites
- Large scale sucker – whole body composites for organics; guts/internal organs composites-
metals
- Burbot – whole body composites
- Long-nose suckers – whole body composites (archived)

Some discrepancies and missing information were noted on the composite sample numbers listed on the COCs and the fish processing forms. ASL (represented by Mr. Robert Wong) and CH2MHill QA Manager, (Ms. Artemis Antipas) were contacted to clarify and correct these discrepancies on April 26, 2006. An explanation and reasons for the discrepancies were immediately sent to the reviewer. See the attached communication logs at the end of this validation memo.

Sample Homogenization and Compositing

Each individual fish collected was given identification number, tagged and individually homogenized at ASL using a commercial grade stainless steel blender/grinder (Robo-Coupe Blixer 6) with liquid nitrogen. Equal amounts of homogenized whole body, fillet or offal tissue samples were mixed and composited to form a single sample. The homogenization forms and the resulting fish sample composites were evaluated by this reviewer. There were no discrepancies noted between the sample collection forms, homogenization forms and the sample composite COCs. Fillet and whole body samples included the fish skin. Care was taken to prevent cross-contamination between sample homogenates. Prior to the start of the project samples, the homogenization process was audited by the project's EPA and CH2MHill QA Managers. To monitor processing cross-contamination, proof blanks were collected at the QAPP specified frequency and sent to the Contract Laboratory program (CLP) for the analysis of the project target compound.

Corrective Action: Deviation from the QAPP as a result of field and sample processing assessment: In a mock sample processing and homogenization conducted during the EPA's and CH2MHill's QA lab audit, it was found out that otoliths were very hard to remove when the fish samples were already frozen. In addition, subjecting the fish to freezing and defrosting ruptures the internal organs, make the fish muscles mushy and thus made the separation of fillets from the offals quite a challenge. To avoid cross-contamination of the fish tissue samples with the offals and to better preserve the otoliths, it was agreed by the project team that the removal of otolith will be conducted on-site after field measurements and before sample preservation (freezing to -20C) and if bench space and resources will allow, filleting of fish samples will also be performed on-site prior to freezing the samples.

Fish Age Determination and Range

The following methods were used to determine the age of the fish: otoliths (inner ear of a fish) were used to determine the age of Lake whitefish, burbot and mountain whitefish. Both otoliths and scales were used to determine the age of the walleye, wild and hatchery rainbow trouts. The opercular covers (also called opercula) were used determine the age of large-scale suckers.

Otolith, scales and operculas were read with the knowledge of the place of capture, sex and size of the fish. The readings were performed by only one individual Mr. John Sneva of WSDFW. Precision and

consistency of readings were checked through the comparison of annuli (otoliths) and the occuli (scales) readings when both specimens are available. The fish age are based on visual readings (duplicate readings, if additional otoliths or scales were available) and maybe estimated range. There was no second party independent readings or validation conducted with these age estimates.

Fish age logs indicated the following: age of lake whitefish ranged from 1-3 years old; hatchery rainbow trouts ranged mostly 1-2 years; wild rainbow trouts from 1-4 years old; mountain whitefish ranged from 0-15 years old; large-scale suckers nine of which were <10 years old; the rest ranged from >10-36 years of age; walleye and burbot from 2-5 years old.

Sample Receipt and Storage

All of the samples were received frozen and intact at the USEPA MEL from CH2MHill lab. After inspection, inventory and logging-in, the sample homogenates were stored in a freezer at -20C while waiting for extraction and analysis. The samples evaluated in this validation report were shipped by MEL to Paradigm Lab from January 26, 2006 to March 24, 2006. All of the fish tissue samples were received intact and still frozen at the verified time of sample receipt (VTSR) at Paradigm Labs.

Holding Times - Acceptable

All of the fish tissue samples were frozen at -20 °C while on storage at MEL and samples were still frozen when received at Paradigm Lab. The integrity of the samples was preserved while on storage at MEL and during shipment. All of the sample analyses met the contractual extraction and analytical holding times of 10 days from the VTSR and 30 days from the extraction date, respectively.

All of the samples also met the method and project extraction and analytical holding times of one year from the date of sample collection. None of the CB congener data were qualified on this basis. The list of samples, cross-referenced to the fish species, station location, and the dates of sample collection, VTSR at the lab, extraction, extract clean-up and analysis dates are listed in Table 1 at the end of this report. The list of individual fish comprising a composite sample can be found in Appendix B of this report.

Sample Preparation and Clean-up - Acceptable

Appropriate clean-up techniques (silica gel, acid/base back extraction, florisil columns) were used by the lab to remove the interfering organic materials during analysis. The sample chromatograms indicated that all of the chlorodiphenyl ethers (CDPEs) and other organic material in the samples were removed prior to analysis. None of the data were qualified on this basis.

Instrument Performance - Acceptable

The frequency of analysis, minimum resolving power of >10,000, the signal-to-noise (S/N) ratio, mass/ion (m/z) abundance ratios and the appropriate switching times for the Selected Ion Monitoring (SIM) descriptors and the chromatographic resolutions were met by the DB-1 column used in the analysis. Homologues do not overlap between homologue descriptors' switching times. The retention times (RTs) and relative retention times (RRTs) were within the retention time windows established during initial calibration. None of the data were qualified on this basis.

Initial Calibrations - Acceptable

All of the initial calibration (ICAL) curves met the technical acceptance criteria, i.e., percent relative standard deviations (%RSDs) of all native and deuterated CB congeners, signal-to-noise (S/N) ratio of >10 including the lowest standard (CS1), retention times, the instruments' resolving power >10,000 and the ion abundance criteria. The instruments' resolving power were maintained and remained stable throughout the course of the analytical sequences. None of the reported results were qualified on this basis.

Continuing Calibration Verifications (VERS) - Acceptable

The frequency of analysis, mass resolutions, S/N and m/z abundance ratios, native and labeled isotope standard recoveries, the RTs of the native compounds relative to the RRTs of the labeled isotopes, injection internal standard and clean-up standard recoveries were met by all VERs analyzed on both GC/MS systems. None of the data were qualified on this basis.

On-Going Precision and Recovery (OPR) - Acceptable

The frequency of analysis, mass resolutions, S/N and m/z abundance ratios, native and labeled isotope recoveries, the RRTs of the native compounds relative to the labeled isotopes and injection internal standard and clean-up standard recoveries were met by all OPRs extracted and analyzed with the samples and QC samples. None of the data were qualified on this basis.

Compound Quantitation and Detection Limits

All of the samples were analyzed at the project- required detection limits. The following CBs co-eluted in the primary column and were reported as combined CBs: CB12 & 13, CB18 & CB30, CB20 & CB28, CB21 & CB33, CB26 & CB29, CB40 & CB71, CB44, CB65 & CB47, CB45 & CB51, CB49 & CB69, CB50 & CB53, CB59 & CB62, CB61, CB70, CB74 & CB76, CB85, CB116 & CB117, CB86, CB87, CB108, CB119 & CB125, CB88 & CB91, CB90, CB101 & CB113, CB110 & CB115, CB107 & CB124, CB128 & CB166, CB129, CB138 & CB163, CB139 & CB140, CB135 & CB151, CB147 & CB149, CB153 & CB168, CB156 & CB157, CB171 & CB173, CB180 & CB193, CB183 & CB185, CB197 & CB200, CB198 & CB199. CB detections with interferences were qualified estimated, "J", by this reviewer.

Some CB congeners were detected at concentrations that were over the calibration range. These samples were not analyzed at dilutions and the reported results were qualified estimated, "J". Data users should consider these flagged results as biased low and may be higher than was actually reported.

The PCB congener detection limits for some of the samples were elevated due to the detection of trace levels of some of the PCB congeners in their associated method blank(s). Even with elevated detection limits, the project-required detection limits for all CB congeners were met.

Compound Identification

All of the reported results met the technical acceptance criteria for PCB congener identification, i.e., S/N ratios greater than 2.5 and >10 for the labeled isotopes, RTs within 0.5% of the mean retention times calculated from the ICALs, ratios between the integrated areas of the method specified m/z pairs were within the method specified limits. There were no CDPE chromatographic interferences in the sample

analyses.

Some of the CB results identified and reported did not meet the method specified mass-ion abundance ratio criteria and were given the “K” qualifier by the lab. The “K” qualifiers were crossed by this reviewer and the results were qualified as non-detects, “U”, at the level of detection, if > reporting limits (RLs) or elevated at RLs when detected at concentrations <RLs.

Method Blanks

The frequency of analysis of laboratory blanks was met. Trace levels of CBs were detected in the method blanks.

Method Blank	Extraction Date	Detected Compounds	Conc. (pg/g)	5x Conc. (pg/g)	Validation Qualifier	Affected Samples
	01/30/06	PCB 1	0.520	2.60	<5x conc. LMB = U	All samples in the SDG: G619-9
		PCB 2	0.804	4.02		
		PCB 3	0.882	4.41		
		PCB 4	0.944	4.72	>5x conc. LMB- No flag	
		PCB 6	0.500	1.00		
		PCB 8	2.64	13.2		
		PCB 11	23.9	119		
		PCB 15	3.25	16.3		
		PCB 16	1.55	7.75		
		PCB 17	1.42	7.10		
		PCB18	2.70	13.5		
		PCB 19	0.244	1.22		
		PCB 20	10.2	51.0		
		PCB 21	6.01	30.0		
		PCB 22	4.73	23.7		
		PCB 24	0.186	0.930		
		PCB 25	0.624	3.12		
		PCB 26	1.28	6.40		
		PCB 27	0.304	1.52		
		PCB 31	8.27	41.4		
		PCB 32	1.08	5.40		
		PCB 35	0.284	1.42		
		PCB 37	3.67	17.4		
		PCB 40	2.06	10.3		
		PCB 41	0.682	3.41		
		PCB 42	1.14	5.70		
		PCB 44	4.12	20.6		
		PCB 45	0.942	4.71		
		PCB 48	0.966	4.83		
		PCB 49	2.16	10.8		
PCB 50	0.466	2.33				
PCB 52	4.61	23.1				
PCB 56	1.16	5.80				
PCB 59	0.374	1.87				
PCB 60	0.840	4.20				
PCB 61	5.39	26.9				
PCB 64	1.90	9.50				
PCB 66	2.15	10.8				
PCB 67	0.146	0.730				
PCB 77	0.542	2.71				
PCB 79	0.174	0.870				

Method Blank	Extraction Date	Detected Compounds	Conc. (pg/g)	5x Conc. (pg/g)	Validation Qualifier	Affected Samples
		PCB 82	0.854	4.27		
		PCB 83	0.470	2.35		
		PCB 84	1.21	6.05		
		PCB 85	1.06	5.30		
		PCB 86	4.45	22.3		
		PCB 88	0.530	2.65		
		PCB 90	4.15	20.8		
		PCB 92	0.840	4.20		
		PCB 95	2.94	14.7		
		PCB 98	0.304	1.52		
		PCB 99	1.58	7.90		
		PCB 105	2.48	12.4		
		PCB 107	0.360	1.80		
		PCB 109	0.342	1.71		
		PCB 110	6.16	30.8		
		PCB 112	0.270	1.35		
		PCB 118	4.46	22.3		
		PCB 126	0.346	1.73		
		PCB 128	0.914	4.57		
		PCB 129	5.72	28.6		
		PCB 132	2.28	11.4		
		PCB 135	1.60	8.00		
		PCB 136	0.734	2.67		
		PCB 141	0.862	4.31		
		PCB 146	0.582	2.91		
		PCB 147	3.79	18.9		
		PCB 153	3.40	17.0		
		PCB 156	0.734	3.676		
		PCB 158	0.506	2.53		
		PCB 164	0.376	1.88		
		PCB 167	0.264	1.32		
		PCB 170	0.650	3.25		
		PCB 171	0.420	2.10		
		PCB 174	0.766	3.83		
		PCB 177	0.466	2.33		
		PCB 179	0.372	1.86		
		PCB 180	1.42	7.10		
		PCB 183	0.680	3.40		
		PCB 187	1.05	5.25		
		PCB 189	0.184	0.920		
		PCB 198	0.446	2.23		
LMB12375	03/05/06	PCB 1	0.578	2.89		All samples in SDG: G619-14
		PCB 2	0.978	4.89		
		PCB 3	0.758	3.79		
		PCB 8	2.76	13.8		
		PCB 11	17.4	87.0		
		PCB 15	2.42	12.1		
		PCB 16	1.24	6.20		
		PCB 17	1.03	5.15		
		PCB 18	1.72	8.60		
		PCB 20	4.92	24.6		
		PCB 21	3.09	15.5		
		PCB 22	2.56	12.8		
		PCB 26	0.660	3.30		
		PCB 31	3.90	19.5		

Method Blank	Extraction Date	Detected Compounds	Conc. (pg/g)	5x Conc. (pg/g)	Validation Qualifier	Affected Samples
		PCB 32	0.712	3.56		
		PCB 37	2.13	10.7		
		PCB 40	0.910	4.55		
		PCB 44	2.24	11.2		
		PCB 49	1.00	5.00		
		PCB 52	1.84	9.20		
		PCB 61	2.96	14.8		
		PCB 64	0.962	4.81		
		PCB 66	1.34	6.70		
		PCB 86	1.64	8.20		
		PCB 90	1.76	8.80		
		PCB 95	1.38	6.90		
		PCB 110	2.13	10.7		
		PCB 118	1.35	6.75		
		PCB 129	2.14	10.7		
PCB 153	1.34	6.70				
LMB12381	03/08/06	PCB 1	0.346	1.73		All samples in the SDG: G619-15
		PCB 2	0.492	2.46		
		PCB 3	0.630	3.15		
		PCB 4	0.498	2.49		
		PCB 6	0.364	1.82		
		PCB 8	1.58	7.90		
		PCB 9	0.148	0.740		
		PCB 11	7.70	38.5		
		PCB15	1.33	6.65		
		PCB 16	0.918	4.59		
		PCB 17	0.666	3.33		
		PCB 18	1.13	5.65		
		PCB 20	3.61	18.1		
		PCB 21	2.23	11.2		
		PCB 22	1.70	8.50		
		PCB 25	0.280	1.40		
		PCB 26	0.562	2.81		
		PCB 31	3.00	15.0		
		PCB 32	0.462	2.31		
		PCB 35	0.250	1.25		
		PCB 37	1.40	7.00		
		PCB 40	0.740	3.70		
		PCB 42	0.490	2.45		
		PCB 44	1.72	8.60		
		PCB 45	0.484	2.42		
		PCB 49	0.870	4.35		
		PCB 52	1.63	8.15		
		PCB 56	0.610	3.05		
		PCB 60	0.496	2.48		
		PCB 61	2.76	13.8		
PCB 64	0.864	4.32				
PCB 66	1.56	7.80				
PCB 79	0.290	1.45				
PCB 85	0.590	2.95				
PCB 86	1.61	8.05				
PCB 90	1.76	8.80				
PCB 95	1.12	5.60				
PCB 99	0.914	4.57				
PCB 105	1.05	5.25				

Method Blank	Extraction Date	Detected Compounds	Conc. (pg/g)	5x Conc. (pg/g)	Validation Qualifier	Affected Samples
		PCB 107	0.408	2.04		
		PCB 109	0.222	1.11		
		PCB 110	2.12	10.6		
		PCB 118	2.00	10.0		
		PCB 128	0.588	2.94		
		PCB 129	2.25	11.25		
		PCB 132	0.672	3.36		
		PCB 141	0.518	2.59		
		PCB 147	1.18	5.90		
		PCB 153	2.25	11.25		
		PCB 156	0.660	3.30		
		PCB 180	1.13	5.65		
		PCB 187	0.754	3.77		
		PCB 209	0.464	2.32		

Toxicity Equivalence (TEQs)

The total PCB homologues and TEQ values were not calculated and reported by the laboratory.

Standard Reference Material (SRM)

Two aliquots of Lake Superior Fish Tissue SRM 1946 were submitted with the samples in two sample delivery groups (SDGs: G618-14 and G619-15). This SRM is a frozen tissue homogenate prepared from lake trout (*Salvelinus namaycush namaycush*) collected from Lake Superior (US/Canada) with certified concentrations of selected PCB Congeners and other organic and inorganic compounds.

The SRMs were received in Paradigm Labs with a cooler temperature of 12C and the homogenates were no longer frozen. The SRM 1946 has certified concentrations for the following PCB congeners: PCB 44, PCB 49, PCB 52, PCB 66, PCB 70, PCB 74, PCB 77, PCB 87, PCB95, PCB 99, PCB 101, PCB 105, PCB 110, PCB 118, PCB 126, PCB 128, PCB 138, PCB 146, PCB 149, PCB 153, PCB 156, PCB 169, PCB 170, PCB180, PCB 183, PCB 187, PCB 194, PCB 195, PCB 206 and PCB 209. The SRM also has reference values for the following PCB congeners: PCB 18, PCB 28, PCB 31, PCB 56, PCB 63, PCB 107, PCB 132, PCB 158, PCB 163, PCB 174, PCB 193 and PCB201.

The analysis of the SRMs yielded % recoveries that were acceptable for all certified and reference concentrations with the exception of the recoveries for PCBs 56 and 107 indicating low bias in the associated results. In addition, the low level PCB 169 was only recovered in one of the SRM run. Due to possible bias in the associated results, the reported results for PCBs 56, 107 and 169 were qualified estimated, “J”. Data users should consider the values reported for these three congeners as the lowest amount present in the samples.

Analytical Sequence - Acceptable

All of the standards, blanks, samples and QC samples were analyzed in accordance with the method specified analytical sequence. Mass ion locks and resolutions were checked every sequence. A window defining mix, calibration verifications, OPRs and method blanks were analyzed at the method required frequency. All of the analytical sequences were within an acceptable the 12- hour QC period and were bracketed by the resolution and continuing calibration check standards. None of the data were qualified on this basis.

Internal Standards Recoveries - Acceptable

Injection internal standards (IS), isotope-labeled PCB congeners and clean-up standards were added to all samples, QC samples to monitor the stability of the GC/MS systems, the exact amount of extract/standard injected and for concentration quantitation of native PCB congeners. All of the analyses met the internal and labeled standards' recovery criteria (25% to 150%). None of the data were qualified on this basis.

Laboratory Contact

The laboratory was not contacted for this review.

Overall Assessment

All of the samples were analyzed in accordance with the project and method specifications. The data, as qualified, are acceptable and can be used for all purposes.

Data Qualifiers	
U	The analyte was not detected at or above the reported result.
J	The analyte was positively identified. The associated numerical result is an estimate.
UJ	The analyte was not detected at or above the reported estimated result. The associated numerical value is an estimate of the quantitation limit of the analyte in this sample.
R	The data are unusable for all purposes.
N	There is evidence the analyte is present in this sample.
JN	There is evidence that the analyte is present. The associated numerical result is an estimate.

Table 1- Holding Times Summary

Sample Number	Region Tracking Number	Paradigm Laboratory Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	VTSR at Paradigm Lab	Date Extraction	Date Extract Clean-up	Date Analysis
WE1F45	05364204	G619-9-4	09/06/05	9/14/05	11/08/05	11/16/05	01/26/06	01/30/06	02/01/06	02/03/06
WE1F55	05364205	G619-9-5	09/06/05	9/14/05	11/08/05	11/16/05	01/26/06	01/30/06	02/01/06	02/03/06
WE1O45	05364209	G619-9-9	09/06/05	9/14/05	11/08/05	11/16/05	01/26/06	01/30/06	02/01/06	02/03/06
WE3F25	05374201	G619-9-12	09/10/05	9/14/05	11/08/05	11/16/05	01/26/06	01/30/06	02/01/06	02/03/06
RH5W55	05424031	G619-14-2	10/21/05	10/22/05	12/06/05	12/22/05	02/24/06	03/05/05	03/06/06	03/25/06
RH6F35	05424036	G619-14-3	10/21/05	10/22/05	12/15/05	12/22/05	02/24/06	03/05/05	03/06/06	03/25/06
RH6O35	05424041	G619-14-4	10/21/05	10/22/05	12/15/05	12/22/05	02/24/06	03/05/05	03/06/06	03/25/06
RW1F25	05374246	G619-14-5	09/13/05	09/14/05	12/22/05	01/06/06	02/24/06	03/05/05	03/06/06	03/29/06
RW1O25	05374252	G619-14-6	09/13/05	09/14/05	12/22/05	01/06/06	02/24/06	03/05/05	03/06/06	03/30/06
RW2W45	05424047	G619-14-7	10/18/05	10/22/05	12/16/05	01/06/06	02/24/06	03/05/05	03/06/06	04/05/06
RW3F15	05424050	G619-14-8	10/19/05	10/22/05	12/19/05	01/06/06	02/24/06	03/05/05	03/06/06	04/05/06
RW3O15	05424052	G619-14-9	10/19/05	10/22/05	12/19/05	01/06/06	02/24/06	03/05/05	03/06/06	03/28/06
RW5W15	05424054	G619-14-10	10/20/05	10/22/05	12/16/05	01/06/06	02/24/06	03/05/05	03/06/06	04/05/06
WE2W55	05414005	G619-14-11	10/12/05	10/13/05	11/10/05	12/08/05	02/24/06	03/05/05	03/06/06	03/28/06
BB2W33	05424059	G619-14-12	10/18/05	10/22/05	12/27/05	01/20/06	02/24/06	03/05/05	03/06/06	03/28/06
BB3W35	05424062	G619-14-13	10/18/05	10/22/05	01/04/06	01/20/06	02/24/06	03/05/05	03/06/06	03/29/06
BB4W25	05424068	G619-14-14	10/18/05	10/22/05	01/05/06	01/20/06	02/24/06	03/05/05	03/06/06	03/29/06
BB5W55	05424075	G619-14-15	10/22/05	10/24/05	12/28/05	01/20/06	02/24/06	03/05/05	03/06/06	03/29/06
BB6W55	05424080	G619-14-16	10/22/05	10/24/05	12/30/05	01/20/06	02/24/06	03/05/05	03/06/06	03/29/06
LS1W35	05364222	G619-14-17	09/06/05	09/14/05	01/06/06	02/02/06	02/24/06	03/05/05	03/06/06	04/05/06
LS2W45	05414012	G619-14-18	10/12/05	10/19/05	01/10/06	02/02/06	02/24/06	03/05/05	03/06/06	03/30/06
LS3W25	05414016	G619-14-19	10/14/05	10/19/05	01/20/06	02/02/06	02/24/06	03/05/05	03/06/06	03/29/06
LS4W55	05424085	G619-14-20	10/16/05	10/19/05	01/14/06	02/02/06	02/24/06	03/05/05	03/06/06	03/29/06
LS5W25	05424087	G619-15-1	10/17/05	10/22/05	01/08/06	02/02/06	02/24/06	03/09/06	03/10/06	03/30/06
LS6W25	05424092	G619-15-2	10/18/05	10/22/05	01/17/06	02/02/06	02/24/06	03/09/06	03/10/06	03/31/06
LW2W55	05424013	G619-15-3	10/20/05	10/23/05	11/28/05	12/08/05	02/24/06	04/09/06	04/10/06	04/13/06
LW3W25	05374229	G619-15-4	09/14/05	09/15/05	11/21/05	12/08/05	02/24/06	04/09/06	04/10/06	04/13/06
LW4W25	05374239	G619-15-5	09/17/05	09/18/05	11/22/05	12/08/05	02/24/06	04/09/06	04/10/06	04/13/06
LW5W45	05374237	G619-15-6	09/15/05	09/16/05	11/22/05	12/08/05	02/24/06	03/09/06	03/10/06	04/07/06
LW6W13	05374243	G619-15-7	09/17/05	09/18/05	11/18/05	12/08/05	02/24/06	04/09/06	04/10/06	04/13/06
MW1W15	05374263	G619-15-8	09/13/05	09/14/05	11/30/05	01/12/06	02/24/06	04/09/06	04/10/06	04/13/06
RH3F35	05424018	G619-15-9	10/18/05	10/22/05	12/05/05	12/22/05	02/24/06	03/09/06	03/10/06	04/02/06
RH3O35	05424021	G619-15-10	10/18/05	10/22/05	12/05/05	12/22/05	02/24/06	04/09/06	04/10/06	04/14/06
RH4W45	05424025	G619-15-11	10/18/05	10/22/05	12/13/05	12/22/05	02/24/06	03/09/06	03/10/06	04/02/06

Sample Number	Region Tracking Number	Paradigm Laboratory Number	Date Sample Collection	VTSR at CH2M Lab	CH2MHill Homogenization Date	VTSR at MEL	VTSR at Paradigm Lab	Date Extraction	Date Extract Clean-up	Date Analysis
WE3025	05374206	G619-15-12	09/10/05	09/14/05	11/08/05	11/16/05	02/24/06	04/09/06	04/10/06	04/14/06
WE4W45	05374215	G619-15-13	09/12/05	09/14/05	11/09/05	11/16/05	02/24/06	03/09/06	03/10/06	04/07/06
WE5W25	05424002	G619-15-14	10/17/05	10/18/05	11/09/05	12/08/05	02/24/06	03/09/06	03/10/06	04/06/06
WE6F35	05374219	G619-15-15	09/14/05	09/15/05	11/11/05	11/16/05	02/24/06	03/09/06	03/10/06	04/07/06
WE6O35	05374224	G619-15-16	09/14/05	09/15/05	11/11/05	11/16/05	02/24/06	04/09/06	04/10/06	04/14/06



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10 LABORATORY
7411 Beach Dr. East
Port Orchard, Washington 98366

MEMORANDUM

DATE: April 26, 2006

TO: Sally Thomas, Project Manager, EPA Region 10
Office of Environmental Cleanup, Unit 3 Site Cleanup

From: Katie Adams, Chemist, EPA Region 10 Laboratory
Office of Environmental Assessment

SUBJECT: Data Review of the Arsenic Speciation Analyses for Upper Columbia River fish tissues
Project Code: TEC-774G
Account Code: 06T10P302DD2C106XLA00

The following is a data review of the arsenic speciation analyses of 10 fish tissue samples (Set #1) from the Upper Columbia River project. The analyses were done following an extraction and ion chromatography/ICP-MS procedure developed by NERL, ORD-Cincinnati. The work was performed by EPA chemists at the EPA Manchester Environmental Laboratory in Port Orchard, WA.

This review was conducted for the following samples:

05364201 05364206 05374204 05374209 05374214 05374220 05374225
05414005 05414006 05424005

Data Qualifications

The following comments refer to the quality control specifications outlined in the Laboratory's current Quality Assurance Plan, and the QAPP. For those tests for which the USEPA Region 10 Laboratory has been NELAP accredited, all requirements of the current NELAC Standard have been met. The qualifications recommended herein are based on the information provided for the review.

1.0 Timeliness - Acceptable

A specific holding time for the analysis of arsenic species in tissue samples has not been established. The samples were collected from 09/06/2005 to 10/17/2005, and were received by the laboratory on 11/16/2005. The analyses were completed on 03/01/2006. No data qualification was required based on holding time criteria.

2.0 Sample Preparation - Acceptable

The samples arrived at the laboratory already ground and homogenized; they were stored at -20°C until further sample preparation could begin. The samples were freeze-dried prior to extraction.

A portion of each freeze-dried sample was treated with 0.83% tetramethyl ammonium hydroxide (TMAOH), and then neutralized with acetic acid, in order to extract the arsenic species from the tissue. An aliquot of this extract was analyzed for total arsenic. The efficiency of the TMAOH extraction was determined by comparing the amount of arsenic present in the extract to the amount of arsenic present in the original sample. A different portion of the TMAOH extract was analyzed by Ion Chromatography – Inductively Coupled Plasma – Mass Spectrometry (IC-ICP-MS) to separate and quantitate the ionic arsenic species. No qualification of the data was required based on sample preparation.

3.0 Total Arsenic Analysis - Acceptable

The total arsenic determination for the tissue samples was reviewed in the memorandum “Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05364201 – 05424009),” March 16, 2006.

The total arsenic determination for the TMAOH extracts was performed by ICP-MS on 02/24/2006, following laboratory procedures. Each sample digestate was analyzed along with a post-spike, and the resulting information was used to perform a single point Method of Standard Additions correction in order to compensate for the effects of the TMAOH matrix on the analysis.

All procedures met laboratory requirements; therefore, no qualification was necessary based on the analysis of the total arsenic in the sample extracts.

4.0 Ion Chromatography Determination - Acceptable

The anion chromatography analysis determines As^{+3} (Arsenite), As^{+5} (Arsenate), DMA (Dimethylarsinic acid), and MMA (Monomethylarsonic acid). Further characterization of the sample by cation chromatography was not an objective for this project.

Results for As^{+3} and As^{+5} are summed and reported as “inorganic arsenic.” This is because the sample preparation and handling processes in this method do not always preserve the individual inorganic species; As^{+3} and As^{+5} interconvert over time.

Arsenobetaine (AsB) and other cationic species are not separated by this column, but elute together as one peak at the beginning of each chromatogram. Therefore, these results are reported together as “AsB + Cations.” The “AsB+Cations” concentration has been estimated based on calibration standards containing AsB. Because other cations eluting with this peak may have different response factors than AsB, all results reported as “AsB+Cations” are qualified “J”, estimated.

The “unknown species” listed in the report are likely to be anionic arsenosugars. An estimated concentration for these species is provided using a predetermined response factor, and the results are qualified “J”. Further identification and quantitation of the arsenosugars isn’t possible, as standards are not available. Note that in this case, the samples which contained unknown species all had one unidentified peak, with a similar retention time. Therefore it is likely that these samples each contained the same, single, arsenosugar.

5.0 Ion Chromatography Quality Control

All chromatography results were corrected for instrument drift by the analysis of a reference arsenic standard injected post-column, and measured prior to measurement of the species that have undergone the chromatographic separation.

The chromatographic analyses were calibrated with standards containing at least five different concentrations of each of the species being determined. The calibration curves were linear with an $R^2 > 0.995$ for each species. The lowest point on the calibration curve was at the quantitation limit. All points on the calibration curve were within the acceptance range of the true value (10% for all points except the lowest standard, 30% for the lowest standard).

Calibration verification standards were analyzed before and after sample analysis. Second source standards were used for As^{+3} and As^{+5} ; second source standards are not available for the remainder of the arsenic species. The recoveries of the calibration verification standards for each species met the 90 - 100% concentration acceptance criteria.

Laboratory control samples (spike blanks) are extracted and analyzed along with the tissue samples to verify the efficiency

of laboratory procedures. The results met the recovery acceptance criterion (85 – 115% of the standard's true value).

Procedural blanks (extraction blanks) were extracted and analyzed with the samples to show potential contamination from the extraction or analytical procedure. The blank did not contain detectable levels of any of the arsenic species, except for trace levels of As^{+3} . We believe that the autosampler vials are responsible for this arsenic contamination. The only sample with As^{+3} levels above our reporting limit was the reference material DORM. The inorganic arsenic result for this sample was qualified "J" to indicate that the result may be biased high due to contamination.

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed on sample 05364206 to provide information about the effect of the sample matrix on extraction and measurement methods. All matrix spike recoveries were within the acceptance limits of 75-125%.

Duplicate analyses for the chromatography were performed on samples 05364206 and 05424005 to evaluate the reproducibility of the procedure. All results which were above the quantitation limit were within the $\pm 20\%$ RPD criterion.

6.0 Reporting Limit/Quantitation Limits

The reporting limits and quantitation limits used for these sample results are based on our evaluation of the sensitivity of the chromatographic determination. We have established that standards at the quantitation limit can be measured within established limits of accuracy and precision, over the entire course of a chromatographic analysis. The reporting limit was established at a level at which the peaks are consistently distinguished from the background.

Sample results that are greater than the quantitation limit are reported with three significant figures.

Sample results that are greater than the reporting limit, but less than the quantitation limit, are reported with two significant figures and qualified "J", estimated.

Sample results for which a distinct peak is present, but below the reporting limit, are also reported with two significant figures and qualified "J", estimated.

Sample results for which no distinct peak is discernable, are given the value of the reporting limit, and qualified "U", undetected.

Final sample results are calculated based on the chromatography result, the weight of the TMAOH extract aliquot, and the weight of the dried sample that was taken for analysis. Results for the undetected species are calculated using the reporting limit concentration with weights mentioned above.

7.0 Overall Assessment of the Data

The efficiency of the analytical procedure is measured at two stages during the analysis. First, the extraction efficiency is determined by comparing the total concentration of arsenic in the sample extract, to the total concentration present in the tissue.

The second measure of efficiency is the chromatographic recovery. The chromatographic recovery is calculated by summing the individual chromatographic arsenical concentrations and dividing this by the total arsenic concentration present in the sample extract. This compares the quantity of arsenic injected onto the column, to the quantity eluting from the column.

The overall speciation recovery combines these two efficiencies, by comparing the total arsenic eluting from the column, to the amount present in the tissue samples.

Table A (attached) provides the efficiency results for each sample. Criteria for efficiency have not been established for the tissue matrix at this time.

Below are the definitions for the qualifiers used in the Inorganic area when qualifying data from Inorganic analysis.

DATA QUALIFIERS

- U - The analyte was not detected at or above the reported value.
- J - The identification of the analyte is acceptable; the reported value is an estimate.
- JK - The identification of the analyte is acceptable; the reported value is an estimate and may be biased high. The actual value is expected to be less than the reported value.
- JL - The identification of the analyte is acceptable; the reported value is an estimate and may be biased low. The actual value is expected to be greater than the reported value.
- UJ - The analyte was not detected at or above the reported value. The reported value is an estimate.
- NA - Not Applicable. The parameter was not analyzed for, or other is no analytical result for this parameter. No value is reported with this qualification.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10 LABORATORY
7411 Beach Dr. East
Port Orchard, Washington 98366

MEMORANDUM

DATE: May 25, 2006

TO: Sally Thomas, Project Manager
Office of Environmental Cleanup, Unit 3 Site Cleanup, EPA Region 10

From: Katie Adams, Chemist
Office of Environmental Assessment, EPA Region 10

SUBJECT: Data Review of the Arsenic Speciation Analyses for Upper Columbia River Fish Tissues
Set/Shipment #2

Project Code: TEC-774G
Account Code: 06T10P302DD2C106XLA00

The following is a data review of the arsenic speciation analyses of 10 fish tissue samples from the Upper Columbia River project. The analyses were done following an extraction and ion chromatography/ICP-MS procedure developed by NERL, ORD-Cincinnati. The work was performed by EPA chemists at the EPA Manchester Environmental Laboratory in Port Orchard, WA.

This review was conducted for the following samples:

05374229 05374232 05374241 05424012 05424017 05424020 05424024
05424028 05424037 05424042

Data Qualifications

The following comments refer to the quality control specifications outlined in the Laboratory's current Quality Assurance Plan, and the QAPP. For those tests for which the USEPA Region 10 Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The qualifications recommended herein are based on the information provided for the review.

1.0 Timeliness - Acceptable

A specific holding time for the analysis of arsenic species in tissue samples has not been established. The samples were collected from 09/14/2005 to 10/21/2005, and were received by the laboratory on 12/22/2005. The analyses were completed on 04/10/2006. No data qualification was required based on holding time criteria.

2.0 Sample Preparation - Acceptable

The samples arrived at the laboratory already ground and homogenized; they were stored at -20°C until further sample preparation could begin. The samples were freeze-dried prior to extraction.

A portion of each freeze-dried sample was treated with 0.83% tetramethyl ammonium hydroxide (TMAOH), and then neutralized with acetic acid, in order to extract the arsenic species from the tissue. An aliquot of this extract was analyzed for total arsenic. The efficiency of the TMAOH extraction was determined by comparing the amount of arsenic present in the extract to the amount of arsenic present in the original sample. A different portion of the TMAOH extract was analyzed by Ion Chromatography – Inductively Coupled Plasma – Mass Spectrometry (IC-ICP-MS) to separate and quantitate the ionic arsenic species. No qualification of the data was required based on sample preparation.

3.0 Total Arsenic Analysis - Acceptable

The total arsenic determination for the tissue samples was reviewed in the memorandum “Data Review of the Upper Columbia River RI-FS Total Metals in Fish Data, Shipment #2,” May 10, 2006.

The total arsenic determination for the TMAOH extracts was performed by ICP-MS on 03/14/2006, following laboratory procedures. Each sample digestate was analyzed along with a post-spike, and the resulting information was used to perform a single point Method of Standard Additions correction in order to compensate for the effects of the TMAOH matrix on the analysis.

All procedures met laboratory requirements; therefore, no qualification was necessary based on the analysis of the total arsenic in the sample extracts.

4.0 Ion Chromatography Determination - Acceptable

The anion chromatography analysis determines As^{+3} (Arsenite), As^{+5} (Arsenate), DMA (Dimethylarsonic acid), MMA (Monomethylarsonic acid). Further characterization of the sample by cation chromatography was not an objective for this project.

Results for As^{+3} and As^{+5} are summed and reported as “inorganic arsenic.” This is because the sample preparation and handling processes in this method do not always preserve the individual inorganic species; As^{+3} and As^{+5} interconvert over time.

Arsenobetaine (AsB) and other cationic species are not separated by this column, but elute together as one peak at the beginning of each chromatogram. Therefore, these results are reported together as “AsB + Cations.” The “AsB + Cations” concentration has been estimated based on calibration standards containing AsB. Because other cations eluting with this peak may have different response factors than AsB, all results reported as “AsB + Cations” are qualified “J”, estimated.

The “unknown species” listed in the report are likely to be anionic arsenosugars. An estimated concentration for these species is provided using a predetermined response factor, and the results are qualified “J”. Further identification and quantitation of the arsenosugars isn’t possible, as standards are not available. Note that in this case, arsenosugar peaks with several different retention times were observed in different samples.

5.0 Ion Chromatography Quality Control

All chromatography results were corrected for instrument drift by the analysis of a reference arsenic standard injected post-column, and measured prior to measurement of the species that have undergone the chromatographic separation.

The chromatographic analyses were calibrated with standards containing at least five different concentrations of each of the species being determined. The calibration curves were linear with an $R^2 > 0.995$ for each species. The lowest point on the calibration curve was at the quantitation limit. All points on the calibration curve were within the acceptance range of the true value (10% for all points, 30% for the lowest standard) with the exception of MMA at 31% and 33%, respectively, on the two days of analysis. However, MMA results for all samples are below the minimum reporting limit; therefore, no high bias is evident, and no results required qualification.

Calibration verification standards were analyzed before and after sample analysis. Second source standards were used for As^{+3} and As^{+5} ; second source standards are not available for the remainder of the arsenic species. The recoveries of the calibration verification standards for each species met the 90 - 100% concentration acceptance criteria with one exception. The recoveries of all species in a continuing verification standard on 04/12/2006 were biased low, with the recoveries for

the species ranging from 81% to 85%. Therefore, all results for sample 05374241 were qualified (JL) indicating a possible low bias (this is the only sample from this set that was affected by the low recoveries of this control).

Laboratory control samples (spike blanks) are extracted and analyzed along with the tissue samples to verify the efficiency of laboratory procedures. The results met the recovery acceptance criterion (85 – 115% of the standard's true value).

Procedural blanks (extraction blanks) were extracted and analyzed with the samples to show potential contamination from the extraction or analytical procedure. The blank did not contain detectable levels of any of the arsenic species.

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed on samples 05374229 and 05424017 to provide information about the effect of the sample matrix on extraction and measurement methods. All matrix spike recoveries were within the acceptance limits of 75-125%.

Duplicate analyses for the chromatography were performed on samples 05374229 and 05424017 to evaluate the reproducibility of the procedure. All results which were above the quantitation limit were within the $\pm 20\%$ RPD criterion.

6.0 Reporting Limit/Quantitation Limits

Arsenic species results are only reported for samples which had detectable levels of total arsenic. Samples 05374017, 05374017DU, and 05424037 did not contain measurable levels of total arsenic. Arsenic species results for these samples are reported "NA", not applicable.

The reporting limits and quantitation limits used for these samples results are based on our evaluation of the sensitivity of the chromatographic determination. We have established that standards at the quantitation limit can be measured within established limits of accuracy and precision, over the entire course of a chromatographic analysis. The reporting limit was established at a level at which the peaks are consistently distinguished from the background.

Sample results that are greater than the quantitation limit are reported with three significant figures.

Sample results that are greater than the reporting limit, but less than the quantitation limit, are reported with two significant figures and qualified "J", estimated.

Sample results for which no distinct peak is discernable, are given the value of the reporting limit, and qualified "U", undetected.

Final sample results are calculated based on the chromatography result, the weight of the TMAOH extract aliquot, and the weight of the dried sample that was taken for analysis. Results for the undetected species are calculated using the reporting limit concentration with weights mentioned above.

7.0 Overall Assessment of the Data

The efficiency of the analytical procedure is measured at two stages during the analysis. First, the extraction efficiency is determined by comparing the total concentration of arsenic in the sample extract, to the total concentration present in the tissue.

The second measure of efficiency is the chromatographic recovery. The chromatographic recovery is calculated by summing the individual chromatographic arsenical concentrations and dividing this by the total arsenic concentration present in the sample extract. This compares the quantity of arsenic injected onto the column, to the quantity eluting from the column.

The overall speciation recovery combines these two efficiencies, by comparing the total arsenic eluting from the column, to the amount present in the tissue samples.

Table A (attached) provides the efficiency results for each sample. Criteria for efficiency have not been established for the tissue matrix at this time.

Below are the definitions for the qualifiers used in the Inorganic area when qualifying data from Inorganic analysis.

DATA QUALIFIERS

- U - The analyte was not detected at or above the reported value.
- J - The identification of the analyte is acceptable; the reported value is an estimate.
- JK - The identification of the analyte is acceptable; the reported value is an estimate and may be biased high. The actual value is expected to be less than the reported value.
- JL - The identification of the analyte is acceptable; the reported value is an estimate and may be biased low. The actual value is expected to be greater than the reported value.
- UJ - The analyte was not detected at or above the reported value. The reported value is an estimate.
- NA - Not Applicable. The parameter was not analyzed for, or other is no analytical result for this parameter. No value is reported with this qualification.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10 LABORATORY
7411 Beach Dr. East
Port Orchard, Washington 98366

MEMORANDUM

DATE: May 16, 2006

TO: Sally Thomas, Project Manager, EPA Region 10
Office of Environmental Cleanup, Unit 3 Site Cleanup

From: Katie Adams, Chemist, EPA Region 10 Laboratory
Office of Environmental Assessment

SUBJECT: Data Review of the Arsenic Speciation Analyses for Upper Columbia River fish tissues
Shipment #4
Project Code: TEC-774G
Account Code: 06T10P302DD2C106XLA00

The following is a data review of the arsenic speciation analyses of 13 fish tissue samples from the Upper Columbia River project. The analyses were done following an extraction and ion chromatography/ICP-MS procedure developed by NERL, ORD-Cincinnati. The work was performed by EPA chemists at the EPA Manchester Environmental Laboratory in Port Orchard, WA.

This review was conducted for the following samples:

05374249	05374250	05374255	05374256	05374257	05374261	05374263
05374265	05424046	05424048	05424051	05424053	05424054	

Data Qualifications

The following comments refer to the quality control specifications outlined in the Laboratory's current Quality Assurance Plan, and the QAPP. For those tests for which the USEPA Region 10 Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The qualifications recommended herein are based on the information provided for the review.

1.0 Timeliness - Acceptable

A specific holding time for the analysis of arsenic species in tissue samples has not been established. The samples were collected between 09/13/2005 to 10/20/2005, and were received by the laboratory on 01/12/2006. The analyses were completed on 04/12/2006. No data qualification was required based on holding time criteria.

2.0 Sample Preparation - Acceptable

The samples arrived at the laboratory already ground and homogenized; they were stored at -20°C until further sample preparation could begin. The samples were freeze-dried prior to extraction.

A portion of each freeze-dried sample was treated with 0.83% tetramethyl ammonium hydroxide (TMAOH), then neutralized with acetic acid, in order to extract the arsenic species from the tissue. An aliquot of this extract was analyzed for total arsenic. The efficiency of the TMAOH extraction was determined by comparing the amount of arsenic present in the extract to the amount of arsenic present in the original sample. A different portion of the TMAOH extract was analyzed by Ion Chromatography – Inductively Coupled Plasma – Mass Spectrometry (IC-ICP-MS) to separate and quantitate the ionic arsenic species. No qualification of the data was required based on sample preparation.

3.0 Total Arsenic Analysis - Acceptable

The total arsenic determination for the tissue samples was reviewed in the memorandum “Data Validation for Upper Columbia River RI-FS, Analysis of Total elements in Fish Tissue (05374245 – 05374256, 05414008, 05424044 – 05424056),” May 16, 2006.

The total arsenic determination for the TMAOH extracts was performed by ICP-MS on 03/24/2006, following laboratory procedures. Each sample digestate was analyzed along with a post-spike, and the resulting information was used to perform a single point Method of Standard Additions correction in order to compensate for the effects of the TMAOH matrix on the analysis.

All procedures met laboratory requirements; therefore, no qualification was necessary based on the analysis of the total arsenic in the sample extracts.

4.0 Ion Chromatography Determination - Acceptable

The anion chromatography analysis determines As^{+3} (Arsenite), As^{+5} (Arsenate), DMA (Dimethylarsonic acid), MMA (Monomethylarsonic acid). Further characterization of the sample by cation chromatography was not an objective for this project.

Results for As^{+3} and As^{+5} are summed and reported as “inorganic arsenic.” This is because the sample preparation and handling processes in this method do not always preserve the individual inorganic species; As^{+3} and As^{+5} interconvert over time.

Arsenobetaine (AsB) and other cationic species are not separated by this column, but elute together as one peak at the beginning of each chromatogram. Therefore, these results are reported together as “AsB + Cations.” The “AsB + Cations” concentration has been estimated based on calibration standards containing AsB. Because other cations eluting with this peak may have different response factors than AsB, all results reported as “AsB + Cations” are qualified “J”, estimated.

The “unknown species” listed in the report are likely to be anionic arsenosugars. An estimated concentration for these species is provided using a predetermined response factor, and the results are qualified “J”. Further identification and quantitation of the arsenosugars isn’t possible, as standards are not available. Note that in this case, arsenosugar peaks with several different retention times were observed in different samples.

5.0 Ion Chromatography Quality Control

All chromatography results were corrected for instrument drift by the analysis of a reference arsenic standard injected post-column, and measured prior to measurement of the species that have undergone the chromatographic separation.

The chromatographic analyses were calibrated with standards containing at least five different concentrations of each of the species being determined. The calibration curves were linear with an $R^2 > 0.995$ for each species. The lowest point on the calibration curve was at the quantitation limit. All points on the calibration curve were within the acceptance range of the true value (10% for all points, 30% for the lowest standard), with the following exceptions:

As^{+3} and MMA were at 113% for a standard at 1 $\mu\text{g/L}$, and MMA was at 162% for the lowest standard (0.5 $\mu\text{g/L}$). However, results for As^{+3} and MMA for all samples are below the minimum reporting limit; therefore, no high bias is evident, and no results required qualification.

Calibration verification standards were analyzed before and after sample analysis. Second source standards were used for

As⁺³ and As⁺⁵; second source standards are not available for the remainder of the arsenic species. The recoveries of the calibration verification standards for each species met the 90 - 100% concentration acceptance criteria.

Laboratory control samples (spike blanks) are extracted and analyzed along with the tissue samples to verify the efficiency of laboratory procedures. The results met the recovery acceptance criterion (85 – 115% of the standard's true value).

Procedural blanks (extraction blanks) were extracted and analyzed with the samples to show potential contamination from the extraction or analytical procedure. The blank did not contain detectable levels of any of the arsenic species, except for trace levels of As⁺³. We believe that the autosampler vials are responsible for this arsenic contamination. None of the samples for which inorganic arsenic was reported had an As⁺³ component (all the inorganic arsenic in these cases was measured as As⁺⁵). Therefore the As⁺³ contamination was not a contributing factor to the results, and no qualification was required.

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed on samples 05374249 and 05374257 to provide information about the effect of the sample matrix on extraction and measurement methods. All matrix spike recoveries were within the acceptance limits of 75-125%.

Duplicate analyses for the chromatography were performed on samples 05374249 and 05374257 to evaluate the reproducibility of the procedure. All results which were above the quantitation limit were within the $\pm 20\%$ RPD criterion.

6.0 Reporting Limit/Quantitation Limits

The reporting limits and quantitation limits used for these samples results are based on our evaluation of the sensitivity of the chromatographic determination. We have established that standards at the quantitation limit can be measured within established limits of accuracy and precision, over the entire course of a chromatographic analysis. The reporting limit was established at a level at which the peaks are consistently distinguished from the background.

Sample results that are greater than the quantitation limit are reported with three significant figures.

Sample results that are greater than the reporting limit, but less than the quantitation limit, are reported with two significant figures and qualified "J", estimated.

Sample results for which no distinct peak is discernable, are given the value of the reporting limit, and qualified "U", undetected.

Final sample results are calculated based on the chromatography result, the weight of the TMAOH extract aliquot, and the weight of the dried sample that was taken for analysis. Results for the undetected species are calculated using the reporting limit concentration with weights mentioned above.

7.0 Overall Assessment of the Data

The efficiency of the analytical procedure is measured at two stages during the analysis. First, the extraction efficiency is determined by comparing the total concentration of arsenic in the sample extract, to the total concentration present in the tissue.

The second measure of efficiency is the chromatographic recovery. The chromatographic recovery is calculated by summing the individual chromatographic arsenical concentrations and dividing this by the total arsenic concentration present in the sample extract. This compares the quantity of arsenic injected onto the column, to the quantity eluting from the column.

The overall speciation recovery combines these two efficiencies, by comparing the total arsenic eluting from the column, to the amount present in the tissue samples.

Table A (attached) provides the efficiency results for each sample. Criteria for efficiency have not been established for the tissue matrix at this time.

Below are the definitions for the qualifiers used in the Inorganic area when qualifying data from Inorganic analysis.

DATA QUALIFIERS

- U - The analyte was not detected at or above the reported value.
- J - The identification of the analyte is acceptable; the reported value is an estimate.
- JK - The identification of the analyte is acceptable; the reported value is an estimate and may be biased high. The actual value is expected to be less than the reported value.
- JL - The identification of the analyte is acceptable; the reported value is an estimate and may be biased low. The actual value is expected to be greater than the reported value.
- UJ - The analyte was not detected at or above the reported value. The reported value is an estimate.
- NA - Not Applicable. The parameter was not analyzed for, or other is no analytical result for this parameter. No value is reported with this qualification.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10 LABORATORY

7411 Beach Dr. East
Port Orchard, Washington 98366

MEMORANDUM

DATE: May 22, 2006

TO: Sally Thomas, Project Manager, EPA Region 10
Office of Environmental Cleanup, Unit 3 Site Cleanup

From: Katie Adams, Chemist, EPA Region 10 Laboratory
Office of Environmental Assessment

SUBJECT: Data Review of the Arsenic Speciation Analyses for Upper Columbia River fish tissues
Shipments 6 and 7
Project Code: TEC-774G
Account Code: 06T10P302DD2C106XLA00

The following is a data review of the arsenic speciation analyses of 8 fish tissue samples from the Upper Columbia River project. The analyses were done following an extraction and ion chromatography/ICP-MS procedure developed by NERL, ORD-Cincinnati. The work was performed by EPA chemists at the EPA Manchester Environmental Laboratory in Port Orchard, WA.

This review was conducted for the following samples:

05364217 05364218 05414011 05414013 05424062 05424065 05424069

05424079

Data Qualifications

The following comments refer to the quality control specifications outlined in the Laboratory's current Quality Assurance Plan, and the QAPP. For those tests for which the USEPA Region 10 Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The qualifications recommended herein are based on the information provided for the review.

1.0 Timeliness - Acceptable

A specific holding time for the analysis of arsenic species in tissue samples has not been established. The samples were collected from 09/07/2005 to 10/22/2005, and were received by the laboratory on 01/20/2006 and 02/02/2006. The analyses were completed on 04/18/2006. No data qualification was required based on holding time criteria.

2.0 Sample Preparation - Acceptable

The samples arrived at the laboratory already ground and homogenized; they were stored at -20°C until further sample preparation could begin. The samples were freeze-dried prior to extraction.

A portion of each freeze-dried sample was treated with 0.83% tetramethyl ammonium hydroxide (TMAOH), then neutralized with acetic acid, in order to extract the arsenic species from the tissue. An aliquot of this extract was analyzed for total arsenic. The efficiency of the TMAOH extraction was determined by comparing the amount of arsenic present in the extract to the amount of arsenic present in the original sample. A different portion of the TMAOH extract was analyzed by Ion Chromatography – Inductively Coupled Plasma – Mass Spectrometry (IC-ICP-MS) to separate and quantitate the ionic arsenic species. No qualification of the data was required based on sample preparation.

3.0 Total Arsenic Analysis - Acceptable

The total arsenic determination for the tissue samples was reviewed in the memoranda, “Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05424057 – 05424080)” and “Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05364216 – 05364222, 05414009 – 05414018, 05414025, 05414026).”

The total arsenic determination for the TMAOH extracts was performed by ICP-MS on 03/31/2006, following laboratory procedures. Each sample digestate was analyzed along with a post-spike, and the resulting information was used to perform a single point Method of Standard Additions correction in order to compensate for the effects of the TMAOH matrix on the analysis.

All procedures met laboratory requirements; therefore, no qualification was necessary based on the analysis of the total arsenic in the sample extracts.

4.0 Ion Chromatography Determination - Acceptable

The anion chromatography analysis determines As^{+3} (Arsenite), As^{+5} (Arsenate), DMA (Dimethylarsonic acid), MMA (Monomethylarsonic acid). Further characterization of the sample by cation chromatography was not an objective for this project.

Results for As^{+3} and As^{+5} are summed and reported as “inorganic arsenic.” This is because the sample preparation and handling processes in this method do not always preserve the individual inorganic species; As^{+3} and As^{+5} interconvert over time.

Arsenobetaine (AsB) and other cationic species are not separated by this column, but elute together as one peak at the beginning of each chromatogram. Therefore, these results are reported together as “AsB + Cations.” The “AsB + Cations” concentration has been estimated based on calibration standards containing AsB. Because other cations eluting with this peak may have different response factors than AsB, all results reported as “AsB + Cations” are qualified “J”, estimated.

The “unknown species” listed in the report are likely to be anionic arsenosugars. An estimated concentration for these species is provided using a predetermined response factor, and the results are qualified “J”. Further identification and quantitation of the arsenosugars isn’t possible, as standards are not available.

5.0 Ion Chromatography Quality Control

All chromatography results were corrected for instrument drift by the analysis of a reference arsenic standard injected post-column, and measured prior to measurement of the species that have undergone the chromatographic separation.

The chromatographic analyses were calibrated with standards containing at least five different concentrations of each of the species being determined. The calibration curves were linear with an $R^2 > 0.995$ for each species. The lowest point on the calibration curve was at the quantitation limit. All points on the calibration curve were within the acceptance range of the true value (10% for all points, 30% for the lowest standard), with the following exceptions:

As^{+3} and MMA were at 113% for a standard at 1 $\mu\text{g/L}$, and MMA was at 162% for the lowest standard (0.5 $\mu\text{g/L}$). However, results for As^{+3} and MMA for all samples are below the minimum reporting limit; therefore, no high bias is evident, and no results required qualification.

Calibration verification standards were analyzed before and after sample analysis. Second source standards were used for As⁺³ and As⁺⁵; second source standards are not available for the remainder of the arsenic species. The recoveries of the calibration verification standards for each species met the 90 - 100% concentration acceptance criteria, with the following exceptions:

The recoveries of all species in a continuing verification standard on 04/12/2006 were biased low, with the recoveries for the species ranging from 81% to 85%. Therefore, all results for samples MEF032906ACO, 05364217, 05364217DU, 05364218, 05414011, and 05414013 were qualified (JL) indicating a possible low bias.

Also, the recoveries of MMA and As⁺⁵ in one of the calibration verification standards on 04/18/2006 were biased low at 89% and 88% respectively. MMA and Inorganic As results for samples 05424266 and 05424086 were qualified (JL) indicating a possible low bias.

Laboratory control samples (spike blanks) are extracted and analyzed along with the tissue samples to verify the efficiency of laboratory procedures. The results met the recovery acceptance criterion (85 – 115% of the standard's true value).

Procedural blanks (extraction blanks) were extracted and analyzed with the samples to show potential contamination from the extraction or analytical procedure. The blank did not contain detectable levels of any of the arsenic species, except for trace levels of As⁺³. We believe that the autosampler vials are responsible for this arsenic contamination. No samples contained detectable levels of As⁺³.

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed on samples 05364217 and 05424062 to provide information about the effect of the sample matrix on extraction and measurement methods. All matrix spike recoveries were within the acceptance limits of 75-125%.

Duplicate analyses for the chromatography were performed on samples 05364217 and 05424062 to evaluate the reproducibility of the procedure. All results which were above the quantitation limit were within the ±20% RPD criterion.

6.0 Reporting Limit/Quantitation Limits

The reporting limits and quantitation limits used for these samples results are based on our evaluation of the sensitivity of the chromatographic determination. We have established that standards at the quantitation limit can be measured within established limits of accuracy and precision, over the entire course of a chromatographic analysis. The reporting limit was established at a level at which the peaks are consistently distinguished from the background.

Sample results that are greater than the quantitation limit are reported with three significant figures.

Sample results that are greater than the reporting limit, but less than the quantitation limit, are reported with two significant figures and qualified "J", estimated.

Sample results for which no distinct peak is discernable, are given the value of the reporting limit, and qualified "U", undetected.

Final sample results are calculated based on the chromatography result, the weight of the TMAOH extract aliquot, and the weight of the dried sample that was taken for analysis. Results for the undetected species are calculated using the reporting limit concentration with weights mentioned above.

7.0 Overall Assessment of the Data

The efficiency of the analytical procedure is measured at two stages during the analysis. First, the extraction efficiency is determined by comparing the total concentration of arsenic in the sample extract, to the total concentration present in the tissue.

The second measure of efficiency is the chromatographic recovery. The chromatographic recovery is calculated by summing the individual chromatographic arsenical concentrations and dividing this by the total arsenic concentration present in the sample extract. This compares the quantity of arsenic injected onto the column, to the quantity eluting from the column.

The overall speciation recovery combines these two efficiencies, by comparing the total arsenic eluting from the column, to the amount present in the tissue samples.

Table A (attached) provides the efficiency results for each sample. Criteria for efficiency have not been established for the tissue matrix at this time.

Below are the definitions for the qualifiers used in the Inorganic area when qualifying data from Inorganic analysis.

DATA QUALIFIERS

- U - The analyte was not detected at or above the reported value.
- J - The identification of the analyte is acceptable; the reported value is an estimate.
- JK - The identification of the analyte is acceptable; the reported value is an estimate and may be biased high. The actual value is expected to be less than the reported value.
- JL - The identification of the analyte is acceptable; the reported value is an estimate and may be biased low. The actual value is expected to be greater than the reported value.
- UJ - The analyte was not detected at or above the reported value. The reported value is an estimate.
- NA - Not Applicable. The parameter was not analyzed for, or other is no analytical result for this parameter. No value is reported with this qualification.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10 LABORATORY
7411 Beach Dr. East
Port Orchard, Washington 98366

MEMORANDUM

DATE: May 23, 2006

TO: Sally Thomas, Project Manager, EPA Region 10
Office of Environmental Cleanup, Unit 3 Site Cleanup

From: Katie Adams, Chemist, EPA Region 10 Laboratory
Office of Environmental Assessment

SUBJECT: Data Review of the Arsenic Speciation Analyses for Upper Columbia River fish tissues
Shipment #7
Project Code: TEC-774G
Account Code: 06T10P302DD2C106XLA00

The following is a data review of the arsenic speciation analyses of 12 fish tissue samples from the Upper Columbia River project. The analyses were done following an extraction and ion chromatography/ICP-MS procedure developed by NERL, ORD-Cincinnati. The work was performed by EPA chemists at the EPA Manchester Environmental Laboratory in Port Orchard, WA.

This review was conducted for the following samples:

05364212	05364213	05414024	05414029	05424081	05424086	05424251
05424252	05424257	05424258	05424260	05424266		

Data Qualifications

The following comments refer to the quality control specifications outlined in the Laboratory's current Quality Assurance Plan, and the QAPP. For those tests for which the USEPA Region 10 Laboratory has been NELAC accredited, all requirements of the current NELAC Standard have been met. The qualifications recommended herein are based on the information provided for the review.

1.0 Timeliness - Acceptable

A specific holding time for the analysis of arsenic species in tissue samples has not been established. The samples were collected from 09/07/2005 to 10/19/2005, and were received by the laboratory on 02/02/2006. The analyses were completed on 04/18/2006. No data qualification was required based on holding time criteria.

2.0 Sample Preparation - Acceptable

The samples arrived at the laboratory already ground and homogenized; they were stored at -20°C until further sample preparation could begin. The samples were freeze-dried prior to extraction.

A portion of each freeze-dried sample was treated with 0.83% tetramethyl ammonium hydroxide (TMAOH), then neutralized with acetic acid, in order to extract the arsenic species from the tissue. An aliquot of this extract was analyzed for total arsenic. The efficiency of the TMAOH extraction was determined by comparing the amount of arsenic present in the extract to the amount of arsenic present in the original sample. A different portion of the TMAOH extract was analyzed by Ion Chromatography – Inductively Coupled Plasma – Mass Spectrometry (IC-ICP-MS) to separate and quantitate the ionic arsenic species. No qualification of the data was required based on sample preparation.

3.0 Total Arsenic Analysis - Acceptable

The total arsenic determination for the tissue samples was reviewed in a series of memoranda, “Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05414019, 05414027 - 05414029, 05424081 - 05424094)”, “Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05424095, 05424096, 05424253 - 05424258, 05424265 - 05424270)”, and “Data Validation for Upper Columbia RI-FS, Analysis of Total Elements in Fish Tissue (05364211 – 05364215, 05414020 – 05414022, 05414024, 05424097 – 05424099, 05424250 – 05424252, 05424259, 05424260, 05424262, 05424263).”

The total arsenic determination for the TMAOH extracts was performed by ICP-MS on 04/17/2006, following laboratory procedures. Each sample digestate was analyzed along with a post-spike, and the resulting information was used to perform a single point Method of Standard Additions correction in order to compensate for the effects of the TMAOH matrix on the analysis.

All procedures met laboratory requirements; therefore, no qualification was necessary based on the analysis of the total arsenic in the sample extracts.

4.0 Ion Chromatography Determination - Acceptable

The anion chromatography analysis determines As^{+3} (Arsenite), As^{+5} (Arsenate), DMA (Dimethylarsonic acid), MMA (Monomethylarsonic acid). Further characterization of the sample by cation chromatography was not an objective for this project.

Results for As^{+3} and As^{+5} are summed and reported as “inorganic arsenic.” This is because the sample preparation and handling processes in this method do not always preserve the individual inorganic species; As^{+3} and As^{+5} interconvert over time.

Arsenobetaine (AsB) and other cationic species are not separated by this column, but elute together as one peak at the beginning of each chromatogram. Therefore, these results are reported together as “AsB + Cations.” The “AsB + Cations” concentration has been estimated based on calibration standards containing AsB. Because other cations eluting with this peak may have different response factors than AsB, all results reported as “AsB + Cations” are qualified “J”, estimated.

The “unknown species” listed in the report are likely to be anionic arsenosugars. An estimated concentration for these species is provided using a predetermined response factor, and the results are qualified “J”. Further identification and quantitation of the arsenosugars isn’t possible, as standards are not available.

5.0 Ion Chromatography Quality Control

All chromatography results were corrected for instrument drift by the analysis of a reference arsenic standard injected post-column, and measured prior to measurement of the species that have undergone the chromatographic separation.

The chromatographic analyses were calibrated with standards containing at least five different concentrations of each of the species being determined. The calibration curves were linear with an $R^2 > 0.995$ for each species. The lowest point on the calibration curve was at the quantitation limit. All points on the calibration curve were within the acceptance range of the true value (10% for all points, 30% for the lowest standard).

Calibration verification standards were analyzed before and after sample analysis. Second source standards were used for As^{+3} and As^{+5} ; second source standards are not available for the remainder of the arsenic species. The recoveries of the calibration verification standards for each species met the 90 - 100% concentration acceptance criteria with the exceptions

of the recoveries of MMA and As⁺⁵ in one of the calibration verification standards on 04/18/2006, which were biased low at 89% and 88% respectively. MMA and Inorganic As results for samples 05424266 and 05424086 were qualified (JL) indicating a possible low bias.

Laboratory control samples (spike blanks) are extracted and analyzed along with the tissue samples to verify the efficiency of laboratory procedures. The results met the recovery acceptance criterion (85 – 115% of the standard's true value).

Procedural blanks (extraction blanks) were extracted and analyzed with the samples to show potential contamination from the extraction or analytical procedure. The blank did not contain detectable levels of any of the arsenic species.

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed on sample 05424257 to provide information about the effect of the sample matrix on extraction and measurement methods. All matrix spike recoveries were within the acceptance limits of 75-125%.

Duplicate analyses for the chromatography were performed on sample 05424257 to evaluate the reproducibility of the procedure. All results which were above the quantitation limit were within the $\pm 20\%$ RPD criterion.

6.0 Reporting Limit/Quantitation Limits

The reporting limits and quantitation limits used for these samples results are based on our evaluation of the sensitivity of the chromatographic determination. We have established that standards at the quantitation limit can be measured within established limits of accuracy and precision, over the entire course of a chromatographic analysis. The reporting limit was established at a level at which the peaks are consistently distinguished from the background.

Sample results that are greater than the quantitation limit are reported with three significant figures.

Sample results that are greater than the reporting limit, but less than the quantitation limit, are reported with two significant figures and qualified "J", estimated.

Sample results for which no distinct peak is discernable, are given the value of the reporting limit, and qualified "U", undetected.

Final sample results are calculated based on the chromatography result, the weight of the TMAOH extract aliquot, and the weight of the dried sample that was taken for analysis. Results for the undetected species are calculated using the reporting limit concentration with weights mentioned above.

7.0 Overall Assessment of the Data

The efficiency of the analytical procedure is measured at two stages during the analysis. First, the extraction efficiency is determined by comparing the total concentration of arsenic in the sample extract, to the total concentration present in the tissue.

The second measure of efficiency is the chromatographic recovery. The chromatographic recovery is calculated by summing the individual chromatographic arsenical concentrations and dividing this by the total arsenic concentration present in the sample extract. This compares the quantity of arsenic injected onto the column, to the quantity eluting from the column.

The overall speciation recovery combines these two efficiencies, by comparing the total arsenic eluting from the column, to the amount present in the tissue samples.

Table A (attached) provides the efficiency results for each sample. Criteria for efficiency have not been established for the tissue matrix at this time.

Below are the definitions for the qualifiers used in the Inorganic area when qualifying data from Inorganic analysis.

DATA QUALIFIERS

- U - The analyte was not detected at or above the reported value.
- J - The identification of the analyte is acceptable; the reported value is an estimate.
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- JL - The identification of the analyte is acceptable; the reported value is an estimate and may be biased low. The actual value is expected to be greater than the reported value.
- UJ - The analyte was not detected at or above the reported value. The reported value is an estimate.
- NA - Not Applicable. The parameter was not analyzed for, or other is no analytical result for this parameter. No value is reported with this qualification.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10 LABORATORY
7411 Beach Dr. East
Port Orchard, Washington 98366

April 24, 2006

MEMORANDUM

SUBJECT: Data Review for Percent Lipids in fish tissue for Upper Columbia River
Project Code: TEC-774G Account Code: 05T10P302DD2C106XLA00

FROM: Peggy Knight, Chemist, USEPA Region 10 Laboratory
Office of Environmental Assessment

TO: Sally Thomas, Project Manager, USEPA Region 10
Office of Environmental Cleanup

CC: Kevin Rochlin, USEPA Region 10
Office of Environmental Cleanup

Monica Tonel, USEPA Region 10
Office of Environmental Cleanup

Jim Stefanoff, CH2M Hill

The data review of the lipid analysis results for twenty-five fish tissue samples has been completed. The samples were prepared and analyzed by the USEPA Region 10 Laboratory staff located in Manchester, WA using EPA methods SW-846 3541 with gravimetric analysis.

Reviewed in this report are data for sample numbers:

05364216 05364217 05364218 05364219 05364220 05414025 05414026 05414027
05414028 05414029 05424097 05424099 05424251 05424253 05424254 05424255
05424256 05424257 05424258 05424265 05424266 05424267 05424268 05424269
05424270

DATA QUALIFICATIONS

The following comments refer to laboratory performance in meeting the quality control specifications outlined in the analytical method, the Manchester Laboratory Quality Assurance Manual, standard operating procedures, and professional judgment. For those tests for which the USEPA Region 10 Laboratory has been NELAC accredited, all requirements of the current

NELAC Standard have been met.

The conclusions presented herein are based on the information provided for the review.

Percent Lipid Determination – Acceptable

Percent lipids were determined from a portion of the extract generated for the PCB analysis. This procedure determines non-polar lipids.

Overall Assessment

All requirements for data qualifiers from the preceding sections were accumulated. Each sample data summary sheet and each compound was checked for positive or negative results. From this, the overall need for data qualifiers for each analysis was determined. In cases where more than one of the preceding sections required data qualifiers, the most restrictive qualifier has been added to the data.

In general, all unqualified data can be used without restriction. The usefulness of qualified data should be treated according to the severity of the qualifier. Should questions arise regarding the qualification of data and its relation to the usefulness, the reader is encouraged to contact Peggy Knight at the Region 10 Laboratory, phone number (360)871-8713.

LABORATORY QUALIFIER/REMARK CODE DEFINITIONS

Qualifier/ Remark Code	Definition (Codes Assigned to Values)
<	<p>Microbiology – Level of target organism present in the sample is less than detection limit. The reported value is the detection limit.</p> <p>Flash Point – The expected flash point temperature is less than the reported value.</p>
>	<p>Microbiology – Level of target organism exceeds upper limit for acceptable range of countable colonies (MF only) or exceeds MPN indices based on number of positive tubes (MPN only). The reported value is the upper limit.</p> <p>Flash Point – If the sample has a flashpoint, it is greater than the reported value.</p>
J	The identification of the analyte is acceptable; the reported value is an estimate.
JK	The identification of the analyte is acceptable; the reported value is an estimate and may be <u>biased high</u> . The actual value is expected to be less than the reported value.
JL	The identification of the analyte is acceptable; the reported value is an estimate and may be <u>biased low</u> . The actual value is expected to be greater than the reported value.
K	The identification of the analyte is acceptable; the reported value may be <u>biased high</u> . The actual value is expected to be less than the reported value.
L	The identification of the analyte is acceptable; the reported value may be <u>biased low</u> . The actual value is expected to be greater than the reported value.
N	There is presumptive evidence that the analyte is present; the analyte is reported as a tentative identification.
NJ	There is presumptive evidence that the analyte is present; the analyte is reported as a tentative identification. The reported value is an estimate.
U	The analyte was not detected at or above the reported value.
UJ	The analyte was not detected at or above the reported value. The reported value is an estimate.

Qualifier/ Remark Code	Definition (Codes With No Reported Values)
A	Absent – The target parameter was analyzed for but was not present or was undetected. <u>No value is reported with this qualification.</u>
NA	Not Applicable, the parameter was not analyzed for, or there is no analytical result for this parameter. <u>No value is reported with this qualification.</u>
P	Present at an undetermined level – The target parameter is present but not quantifiable or no quantifiable result was determined. <u>No value is reported with this qualification.</u>

Qualifier/ Remark Code	Definition (Codes With No Reported Values)
R	The presence or absence of the analyte can not be determined from the data due to severe quality control problems. The data are rejected and considered unusable. <u>No value is reported with this qualification.</u>
T	A trace of the subject parameter was present. For asbestos analysis the subject parameter was identified but at a low level that a quantifiable percentage of content is unreliable. <u>No value is reported with this qualification.</u>
TNTC	Too Numerous To Count – Any membrane where the total number of bacterial colonies exceeds 200 per membrane, or if the colonies are not distinct enough for accurate counting (i.e. confluent growth).

Qualifier/ Remark Code	Definition (Codes Assigned To Values Generated via Field or Screening Methods)
F	The associated datum was generated using field methods and/or screening methods. The identification of the analyte is acceptable and the reported value has been found to be acceptable for use.
JF	The associated datum was generated using field methods and/or screening methods. The identification of the analyte is acceptable and the reported value is an estimate.
JKF	The associated datum was generated using field methods and/or screening methods. The identification of the analyte is acceptable; the reported value is an estimate and may be <u>biased high</u> . The actual value is expected to be less than the reported value.
JLF	The associated datum was generated using field methods and/or screening methods. The identification of the analyte is acceptable; the reported value is an estimate and may be <u>biased low</u> . The actual value is expected to be greater than the reported value.
UF	The associated datum was generated using field methods and/or screening methods. The analyte was not detected at or above the reported value.
UJF	The associated datum was generated using field methods and/or screening methods. The analyte was not detected at or above the reported value. The reported value is an estimate.

Qualifier/ Remark Code	Cross Reference to Older Codes
A	UND, ND – Undetected, Not detected
NA	NAR, NAF – No analytical result, Not analyzed for
P	PNQ – Present but not quantified
R	REJ - Rejected
T	TRACE

NOTE: For any qualifier code see the QA memo or case narrative for a more detailed description of its use.