STATEMENT OF WORK

A. EPA Region/Client: Region 10

B. Authorized By: _____

C. Prepared By: ROBERT MELTON

D. Date: _____

E. Site Name: _____

1. General description of analytical service requested:

Note: This SOW is written for the measurement of marine tissue samples. The SOW is customized to meet the Data Quality Objectives (DQOs) of the applicable Quality Assurance Project Plan (QAPP) for the Assessment of Chemical Contaminants In Fish Consumed By Four American Indian Tribes In The Columbia River Basin, Revision 5.0., 06/03/96 Therefore, the SOW requires special procedures in the extraction, clean-up, analysis, and reporting of data in order to meet the DQOs of the project. The use of the letters, xxxx, in the following text requires input from the user of the SOW in order to comply with the specifications of the QAPP.

This SOW requires the high resolution capillary column gas chromatography/high-resolution mass spectrometry (HRGC/HRMS) analyses for Polychlorinated Dibenzo-p-Dioxins (PCDDs) and Polychlorinated Dibenzo-p-Furans (PCDFs) such as 2,3,7,8-tetrachlorodibenzo-p-dioxin, 2,3,7,8-tetrachlorodibenzo-p-furan, tetra through octa polychlorinated dibenzodioxin homologues, and tetra through octa polychlorinated dibenzofuran homologues listed in EPA Method 1613B in xxxx fish tissue samples plus one Performance Evaluation (PE) fish tissue sample using EPA Method 1613B: Tetra- through Octa- Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. All the performance specifications of Method 1613B shall be used and achieved by the laboratory.

Percent lipid determination for all marine tissue samples as per Method 1613B is required. Confirmation of 2,3,7,8-

Tetrachloro-p-dibenzofuran is required using a dissimilar capillary GC column such as DB-225, SP-2330, or equivalent. Measurement results for 2,3,7,8-TCDF must be made from the confirmation column. This requires full calibration (both initial and continuing calibration) of the confirmation column in order to report 2,3,7,8-TCDF results. The laboratory must provide written documentation that all quality control (QC) requirements of Method 1613B have been met.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or marine tissue; and whether low, medium or high concentration):

xxxx low-level fish tissue samples plus one Performance Evaluation (PE) sample will be submitted for PCDD/PCDF measurements. In addition, the laboratory will purchase and analyze two samples each (total of six PE samples) of fish matrix reference material PE samples EDF-2524, EDF-2525, and EDF-2526 in the same manner and at the same time that project samples are measured. Accuracy requirements of acceptable recovery ranges for these PE samples have been documented by Cambridge Isotope Laboratories. These acceptable accuracy recovery ranges will be required by the laboratory which measures PCDDs/PCDFs. The estimated cost of procuring two alliquotes each of PE samples EDF-2524, EDF-2525, and EDF-2526 is approximately $2100. This cost should be added to the laboratory's bid price for completion of this SOW.

The subcontract laboratory which is responsible for measuring PCDDs/PCDFs will measure one sample each of PE samples EDF-2524, EDF-2525, and EDF-2526 when the first Sample Delivery Group is measured using Methods 1613B. The Contractor will designate a second SDG during the latter phase of the project for the subcontract laboratory to measure one sample each of PE samples EDF-2524, EDF-2525, and EDF-2526.

3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):

xxxx -- The samples will be collected by EPA to assess chemical contaminant exposure from consumption of Columbia

River fish by four Native American Tribes. The first phase of this study was completed in October of 1994 by the Columbia River Inter-Tribal Fish Commission (CRITFC).

4. Estimated date(s) of collection:

The samples will be collected between xxxx and xxxx.

5. Estimated date(s) and method of shipment:

The samples will be shipped via Federal Express during the week(s) listed in item 4. Tissue samples are to be kept at minus (-) 20 degrees C until analysis. Marine tissue samples are to be maintained at -20 °C for six months by the lab. All tissue sample amounts that remain after extraction is completed and extract solutions shall be retained by the lab at -20 °C for a period of one year from date of sample arrival. EPA has the right to request these remaining sample amounts and extracts for a period of one year from the time of sample arrival. Two samples each of PE samples EDF-2524, EDF-2525, and EDF-2526 will be procured directly by the laboratory for this project.

6. Number of days analysis and data required after laboratory receipt of samples:

The complete data package is required within 35 days of Validated Time of Sample Receipt of the last sample in each Sample Delivery Group (SDG). Tissue samples shall be extracted within 30 days of sample collection. All sample extracts shall be injected within 40 days from date of extraction.

7. Analytical instrumentation and protocols required:

Project samples and PE samples are to be prepared, analyzed, confirmed, documented and reported as specified in EPA Method 1613B: Tetra- through Octa- Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, except as is specified in this SOW for laboratory services. The use of other analytical methods is not be acceptable for this work. Only labs with experience using this method shall perform this

work. Labs bidding on this SOW are required to submit a Lab Quality Assurance Plan (Lab QAP) and Standard Operating Procedures (SOPs) for the measurement of project samples and the PE sample using Method 1613B and this SOW. Labs bidding on this SOW are required to own and use two high resolution GC/MS instruments for this work because second GC column TCDF confirmation and calibration is required by this SOW.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

The compounds to be reported are listed in Table 1 of Method 1613B.

The laboratory which measures project samples will use the following procedure prior to removing a ground sample from a sample bottle for analysis of target compounds:

- Place sample container containing ground fish tissue/eggs in a 34°C to 40°C refrigerator 24 hours prior to removing sample.
- Remove sample bottle from the refrigerator and place on the lab bench at room temperature until all ice crystals in the sample bottle have melted.
- Hand stir the thawed tissue vigorously with a 1/4 inch solid glass rod for 3 minutes.
- Immediately remove sample containing tissue and liquid from sample bottle for weighing and laboratory analysis.
- Fill out a Corrective Action Form (see Attachment 18 to the QAPP) if any sample bottles contain contain either chunks of fish tissue or pieces of fish skin. A copy of this Corrective Action Form must be sent to the Contractor Project Manager.

* The Laboratory shall achieve a Minimum (Quantitation) Limit (ML) of 0.2 ng/Kg (wet weight) for isomers 2,3,7,8-TCDD and 2,3,7,8-TCDF. This lower ML shall be achieved by the use of a low initial

calibration point of 0.1 ng/ml and an ultra-low sensitivity HRMS system.

* Final volume of sample extracts is 20 uL or lower.

* This Initial Calibration of the instrument system for both the primary and secondary confirmation GC column must be determined within 30 days of the time that the first sample in each SDG is measured on the GC/MS system. All labeled and native standards used to measure initial calibration standards, method blanks, verification standards, calibration verification standards, and sample extracts must be from the same lot number and preparation date.

* Fortify project samples and the PE sample with isotopically labeled $^{13}C_{12}$-PCDD and $^{13}C_{12}$-PCDF internal calibration standards as is specified in Method 1613B.

* Measure specified PCDDs and PCDFs in sample extracts using the cleanup procedure and isotopically labeled recovery standards specified in Method 1613B. The lowest level of the initial calibration standards shall be at or below the required Minimum Quantitation Limits (MQLs) specified in this SOW.

* All instructions in Method 1613B shall be followed for all aspects of sample analyses, including but not limited to:

  1) Preparation, storage and analysis of all standards.

  2) Preparation and storage of all project samples and the PE sample.

  3) Cleanup, storage and analysis of all sample extracts.

  4) Instrument calibration.

  5) Quality Assurance/Quality Control.

  6) The option in the method for reporting the analytical results using a 2,3,7,8-TCDD Toxicity Equivalency Factor (TEF) must be used.

7) Second GC column confirmation and measurement of 2,3,7,8-TCDF on the secondary GC column which is calibrated for TCDF measurements is required when 2,3,7,8-TCDF is detected on the primary column. This requires full calibration (both initial and continuing calibration) of the confirmation column in order to report 2,3,7,8-TCDF results.

8) If polychlorinated diphenyl ether (PCDPE) interferences to the measurement of PCDF isomers are present after initial cleanup and analysis procedures are used, then the laboratory must remove these PCDPE interferences prior to final analysis of the extracts using the PCDPE cleanup procedure described in Method 1613B. If the PCDPE interferences are still present after additional PCDPE cleanup steps, then the laboratory must contact the Contractor for instructions.

9) The laboratory shall trace and report the accuracy of the initial calibration curve and of calibration verification standards by measuring a Quality Control (QC) Check Sample which originates from a source which is different from the source of standards used for the initial calibration curve and calibration verification standards.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.

The data package shall include all original documentation generated in support of this Statement of Work and Method 1613B. This includes, but is not limited to: sample tags, custody records, shipping information, standards and sample preparation records, instrument printouts such as chromatograms, extracted ion current profiles (EICPs), quality control requirements, precision and accuracy requirements, etc. When information and documentation required by this SOW or Method 1613B is recorded in permanently bound notebooks and in computer files, copies of the appropriate information shall be submitted to EPA as part of data deliverables

The following additional deliverables are required. Note

that the following requirements are specified in order to emphasize general documentation requirements and are not intended to supersede or change requirements of Method 1613B:

* The lab must submit a copy of the analytical contract, the original sample packing list, chain-of-custody records, sample log-in records, and a Case Narrative describing the analyses and discussing any and all problems experienced during the analyses. The Case Narrative shall include a discussion of the presence of any interferences, the steps used to remove PCDPE interferences from extracts, the criteria used to qualitatively identify target isomers, and the failure of the lab to meet any of the requirements of the SOW or Method 1613B. The lab shall also submit Sample tags, custody seals, chain-of-custody records, and laboratory log in records with the data package. In addition, the data package shall contain the following records and data:

* Analyst bench records describing dilutions, weighing of project samples and the PE sample, sample size, final extract volumes, amount injected, and example calculations such that an independent data reviewer may recreate the calculations from the raw data which is submitted with the data package.

* Detailed explanation of the quantitation and identification procedure used for each of the homologous series and for isomer specific analysis.

* Example calculations of response ratios (RRFs), sample results and detection limits.

* Tabulated recoveries of spiked labeled PCDDs and PCDFs, Internal Standards, Cleanup Standards, and Surrogates used to measure each sample.

* Standard curve RFs, RRFs and %RSDs for initial and calibration verification.

* Simultaneous offset display of single ion chromatograms (EICPs) for each GC column for analyte peaks and for polychlorinated diphenyl ether (PCDPE) peaks in order to check for PCDPE interferences which may co-elute with native target compounds. The hard copy of the EICP of PCDPE

interference peak to 2,3,7,8-TCDF must be expanded on both GC columns to between 50 to 100% full scale in order to visually inspect for PCDPE interference to the measurement of 2,3,7,8-TCDF.

* Tabulated sample detection limits for analytes which are not measured in each sample.

* Deliverables to the Region shall be in the form of a purge file - i.e. **paginated original documents**, not copies of original documents. If an original document cannot be provided in each SDG, then the exact location of the original shall be stamped or recorded in ink on the copy.

After delivery of analytical results and data to EPA, the laboratory shall respond within seven days to written requests from EPA for additional information or explanations that result from the Government's inspection activities. Submissions of re-calculated data, missing deliverables, etc. shall be paginated for easy inclusion into the purge file. For example, if a Form I was left out of the purge file and should have followed page 5555, the submission of the missing page should have page 5556 or page 5555a recorded depending upon whether page 5556 has already been assigned to another page in the purge file.

* All Sample Tracking Reports (i.e. the signed chain-of-custody forms and the signed packing lists).

* DC-1 (Sample Log-In Form)

* DC-2 (Inventory Sheet) - This provides a Table of Contents for data sections in the Case File Purge.

* All of the Sample tags.

* The custody seals.

* A **copy** (not the original) of the SOW.

* Any telephone logs referring to the project samples and the PE sample.

* A Case Narrative signed by the laboratory manager or his/her designee certifying the accuracy and validity of all data reported and describing any changes to requirements in Method 1613B and in the SOW and problems encountered during

the analyses along with documenting their resolution(s). **In addition:** any pre-award conditions/specifications accepted by a regional representative shall be documented in or attached to each case narrative. In the Case Narrative, the laboratory manager shall acknowledge that these measurement results are submitted in support of US EPA regulations and/or programs.

* Tabulated sample results, with units, percent solids, and sample weights or volumes clearly specified.

* Blank data with tabulated results. Specify which samples go with which blank.

* Submit all GPC cleanup calibration and extract run information.

* Submit all additional sample cleanup records and data.

* Sample data including:

  - Tabulated results.
  - All data system printouts.
  - Manual worksheets.

* Raw QC data including:

  - Blank data in chronological order:
  I) Tabulated results.
  ii) All blank data system printouts.

  - Initial Precision and Accuracy data as required by Method 1613B.

  - Calibration Verification data.

  - Ongoing Precision and Accuracy data.

  - Results from the measurement of the QC Check Sample and six PE samples (two each of EDF-2524, EDF-2525, and EDF-2526.

* Detailed explanation of the quantitation and identification procedure used for each of the homologous series and for isomer specific analysis.

* List of exact ion masses, response factors and retention

times used for each isomer/class.

* Tabulated recoveries of Labeled Internal Standards and Clean-up Standards compared to the concentration used.

* Calibration curve(s) labeled with date and time of preparation.

* Standard curve RFs, RRFs and %RSDs for initial and calibration verification standards.

* EICPs of performance check mixtures showing first and last eluting compounds of each homologous series as well as the percent valley resolution for labeled TCDD and TCDF isomers as is required by Method 1613B.

* Complete documentation of initial and calibration verification and samples to include tabulated results of ion ratios and offset simultaneous displays of the single ion chromatograms of the two most abundant ions in the molecular ion region.

* Bench sheets for sample preparation indicating dates, times, methods of sample digestion/preparation and analysis, and volumes/amounts/concentrations of standard and reagents added, instrument run time/date, dilutions made, etc. Submit preparation/weight logs for percent moisture and percent lipid determinations. All bench sheets and logs will be labeled with the date and shall bear the analyst's signature.

* A formula (including definitions) showing how measurement results were calculated, with examples of actual calculations of response ratios (RRFs), sample results and detection limits.

Ship all regional deliverables to:

Laura Castrilli
USEPA Region 10 9th Floor
1200 Sixth Avenue MS/ES-095
Seattle, WA 98101

10. Other (use additional sheets or attach supplementary information as needed):

All hardcopy data reports and raw data shall be clear and legible. If discrepancies are found, the laboratory shall be required to resubmit non-compliant data or reports at no additional cost to the Government within 10 days after request by EPA.

11. Name of sampling/shipping contact:
Phone:

xxxx

12. Data Requirements

Minimum (Quantitation) Limits (MLs)

The following are required MLs:

Media, Units	Tetra CDDs/CDFs	Penta-Hepta CDDs/CDFs	Octa CCDs/CDFs
tissue ng/kg	0.2	5.0	10.0

13. Additional QC Requirements of the SOW

[1] The sum of the area counts for the two quantitation masses listed in Table 8 of Method 1613B for each of the two instrument recovery internal standards for samples, blanks, and standards (such as OPR standards and VER standards) must not vary by more than a factor of two (-50% to +100%) from the associated average areas of the five initial calibration standards.

14. Action Required if Limits are Exceeded

Laboratory:
If any QC limits specified in Method 1613B or in this SOW are exceeded, the laboratory must contact EPA for resolution.