

JOHNSON LAKE FISH TISSUE MONITORING STUDY

SAMPLING AND ANALYSIS PLAN

PREPARED FOR:

OWENS-BROCKWAY GLASS CONTAINER INC.
5850 NE 92ND DRIVE
PORTLAND, OR, 97222

PREPARED BY:

GRETTE ASSOCIATES^{LLC}
2102 NORTH 30TH STREET, SUITE A
TACOMA, WASHINGTON 98403
(253) 573-9300

FEBRUARY 4, 2022



TABLE OF CONTENTS

1	INTRODUCTION.....	1
1.1	Project Site Description and Background	1
1.2	Remediation Activities.....	1
1.3	Sampling and Analysis Plan (SAP)	2
1.3.1	Purpose	2
1.3.2	Scope	2
1.3.3	Organization.....	2
2	FISH SAMPLING.....	4
2.1	Fish Collection.....	4
2.2	Tissue Sample Collection, Decontamination, and Packaging.....	5
2.3	Sample Transport and Chain of Custody Procedures	6
3	LABORATORY ANALYSIS	7
3.1	Testing Facilities.....	7
3.2	Laboratory Chain of Custody	7
3.3	Chemicals of Concern (Chemicals of Interest) Screening	7
3.4	Testing Methods	7
4	QUALITY ASSURANCE.....	8
5	REFERENCES.....	9

LIST OF TABLES

Table 1. Sample Holding Time and Storage Criteria.....	7
--	---

LIST OF FIGURES

Figure 1: Site Map of Johnson Lake	10
--	----

LIST OF ATTACHMENTS

- Attachment A. ODFW Scientific Taking Permit
- Attachment B. ODEQ May 25, 2017 SAP approval and comments
- Attachment C. ALS Standard Operating Procedure MET-TISP (5/29/16) – Fish Tissue Sample Preparation
- Attachment D. ODEQ January 29, 2018 Letter and Comments

1 INTRODUCTION

This Sampling and Analysis Plan (SAP) has been prepared to describe the Johnson Lake fish tissue monitoring study, consistent with the *Remediation Operations & Maintenance Plan* (O&M Plan), *Sediment Remedial Action* (DOF 2012b), hereafter “O&M Plan.” In support of this SAP, the approved Scientific Take Permit from the Oregon Department of Fish and Wildlife is included as Attachment A. Approval of the 2017 SAP by comment letter from Oregon Department of Environmental Quality (DEQ), comments which are incorporated herein, are included as Attachment B. DEQ comments from the 2017 collection and sampling effort are included as Attachment C. The analytical laboratories (ALS Global) standard operating procedures for fish tissue sample preparation are attached as Attachment D.

The 2022 Johnson Lake fish tissue monitoring study is a requirement of the O&M Plan in response to the results of the 2017 monitoring effort showing contaminant levels in fish samples exceeding the levels specified in the 2007 Record of Decision for the site cleanup.

1.1 PROJECT SITE DESCRIPTION AND BACKGROUND

Owens-Brockway Glass Containers Inc. (Owens) owns and operates a glass manufacturing plant located at 5850 NE 92nd Drive, Portland, Oregon (the “Property”). Owens’ Property is approximately 43 acres in size and is located on the south shore of Johnson Lake. Johnson Lake extends over 18 acres and is directly connected to the Whitaker Slough, which in turn flows to the Columbia Slough. Johnson Lake is bounded to the south by the Owens Property and to the west and east by other industrial facilities and highways. The Owens Property and Johnson Lake are collectively referred to as the “Site.”

Sediments in Johnson Lake were found to contain polychlorinated biphenyls (PCBs), as described in the *Johnson Lake Investigation Work Plan* (ARCADIS 2004a), *Site Investigation Report* (ARCADIS 2004b), and *Johnson Lake Risk Assessment* (Environ and ARCADIS 2004). Based on the previous investigations and a Feasibility study prepared in 2006, DEQ selected a final remedy for the site in the Record of Decision (October 2007) and Record of Decision Amendment (July 2009). The 2007 Record of Decision and the 2009 Amendment are referred to collectively in this document as the ROD.

1.2 REMEDIATION ACTIVITIES

As described in the ROD, the final remedy for cleanup of the PCBs and other contaminants at the site consisted of the following tasks.

1. Surface soils from the low-lying area were excavated.
2. Excavated soils were disposed of at an off-site landfill.
3. The excavated area was covered with clean soil and revegetated.
4. A stormwater bio-swale (swale) was constructed.
5. A thin layer cap was placed over Johnson Lake sediments.

All of the above-listed tasks have been completed and are addressed in detail in the two Site completion reports:

- *Project Completion Report, Soil Remediation Action*, March 22, 2011 (Dalton Olmsted Fuglevand (“DOF”) and Environ 2011) (Tasks 1 through 4)
- *Project Completion Report, Sediment Remedial Action*, September 24, 2012 (DOF 2012a) (Task 5).

1.3 SAMPLING AND ANALYSIS PLAN (SAP)

1.3.1 Purpose

The purpose of this SAP is to describe the Johnson Lake fish tissue monitoring study and build upon previous study efforts. The objective of this study is to evaluate the level of PCBs in fish tissue sampled from Johnson Lake, consistent with the O&M Plan (DOF 2012b). The ROD included the following objective with respect to PCBs in fish tissue, “Prevent human consumption of fish tissue with tissue concentrations of greater than 0.003 ug/kg PCB congener 126”. Fish tissue results will be compared with this concentration.

In 2017, a collection and sampling field effort was completed, and all field and lab data were included in a monitoring report submitted to DEQ in January of 2018. As PCBs were still present in fish tissue at levels exceeding the ROD standard, further efforts to collect fish tissue in 2022 will be conducted and will include information provided in the 2018 report as well as a summary of results from the 2017 and 2004 events for comparison (DEQ 2018). This SAP addresses comments from DEQ regarding the 2017 sampling efforts.

1.3.2 Scope

The SAP describes the procedures for the following:

- Fish collection (including environmental data collection)
- Tissue sampling (for fish categories with fillet samples), decontamination, and packaging
- Laboratory Analysis (EPA Method 1668C; NOAA or Bligh and Dyer (1959) for lipids)
- Quality Assurance

1.3.3 Organization

The sampling and analysis for the Johnson Lake fish tissue monitoring study is based on the monitoring report requirements as described in the O&M Plan (DOF 2012b), with additional input as provided during discussions between Rob Webb of DOF (“Project Engineer”), Susan Sholl of Owens, Scott Maharry of Grette Associates, and DEQ personnel in early 2017. Further monitoring report requirements will be incorporated based on comments from DEQ in the 2018 comment letter. Specifically, the minimum requirements for the monitoring report are as follows:

- The date of the fish tissue collection
- Location of fish collection
- Fish tissue PCB concentrations
 - per DEQ, these should be reported both as total PCB and by congener
 - per DEQ, these should be reported both on the basis of mass and lipid content
 - per DEQ, these should be reported as total PCB, PCB TEQ and, lipid normalized total PCB concentrations in addition to PCB congener 126 concentrations.
- A description of the area as a whole and details of any observed deficiencies or required maintenance
- Results of cap inspection with photos (2017, to be provided by the Project Engineer)

2 FISH SAMPLING

2.1 FISH COLLECTION

The following fish collection program is consistent with the O&M Plan (DOF 2012b) as well as phone and email communication between the Project Engineer and DEQ personnel in early 2017 and the 2018 DEQ comment letter.

The fish collection will be conducted on foot or from a small vessel(s) using a waveform electro-shocker, traditional hook and line methods, and nets or seining. Fish will be collected from a variety of locations around and within the lake, as practicable, and where fish can be collected to represent general fish tissue concentrations lake-wide. Composite samples of each fish collected shall adequately represent the sampling category and be of the same type (species) and size (age) class. The collection will be consistent with EPA Guidelines (Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1 Fish Sampling and Analysis) (EPA, 2015), in a manner such that the differences between lengths of individuals in a composite sample and the average of the lengths shall not exceed 10 percent, and at least one replicate shall also be analyzed. (DEQ 2018)

The lake has been remediated with a thin-layer cap, as described in Section 1.2. Fish collection methods must not damage the cap. The Project Engineer advises that the cap is resilient enough for sampling methods which could require biologists to walk on it, including seining (R. Webb, personal communication).

Fish collection will target the following categories of fish. The goal for each category is to collect enough fish for the lab to composite five samples of the same fish type (fillets) and size range (whole fish) into a single composite sample for each category.

- Game fish fillets: One composite sample will target fish typically consumed by humans (e.g., largemouth bass).
- Panfish fillets: One composite sample will target panfish (e.g., bluegills); panfish tend to be smaller than game fish and may be consumed by humans.
- Rough fish fillets: One composite sample will target rough fish that are not typically targeted by humans but may be consumed by humans (e.g., suckers, carp).
- Large whole-fish: One composite sample will target fish between 125 and 300 mm in length that may be consumed by large wildlife species.
- Small whole-fish: One composite sample will target fish <125 mm in length that may be consumed by smaller wildlife species.

During the 2017 collection effort, a single white sturgeon (Acipenser transmontanus) approximately six feet in length was observed in Johnson Lake. The sturgeon was not harmed during the field collection and may still be present in the lake. If this is the case, interactions with the sturgeon will be avoided to the extent practicable. The sturgeon will be included in the list of

potential bycatch species in the application for a Scientific Taking Permit obtained from the Oregon Department of Fish and Wildlife for the project. Permit is included as Attachment A.

Individual fish comprising a composite sample should be of similar size (the smallest individual in a composite should be no less than 75% of the total length of the largest individual). Where possible, fish should be of a harvestable size and weight. Samples collected will be representative of fish likely to be consumed by humans and large enough that this exposure would be reasonable.

Due to habitat limitations and low oxygen conditions previously documented in Johnson Lake, it may not be practicable to collect and analyze the numbers and types of fish samples described above as there may not be a significant fish population in the lake. A good faith effort (four days moving about the lake on foot or by boat, attempting to electroshock or otherwise collect fish that are present) will be made to characterize fish PCB concentrations that are representative of those existing in Johnson Lake. In the event that adequate fish are not collected from each category, a second 4-day collection and sampling effort will be made over the same period at a later date during 2022. This approach is consistent with the O&M Plan (DOF 2012b). The previous sampling indicates that small fish do not inhabit the center of the lake (ARCADIS 2004b).

Sampling methods will include the use of a backpack electro shocker with dip nets, passive nets (e.g., gill net, Fyke net), beach seine, and hook and line angling.

Fish collected at each sample location will be enumerated by species, categorized (game fish, panfish, rough fish), and measured. Fish collected during sampling from a specific location within the lake will be temporarily held in a decontaminated cooler filled with water from Johnson Lake. Once sampling activities from that area are complete, fish targeted for tissue analysis will be euthanized via CO₂ immersion (dry ice added to holding water), wrapped in aluminum foil, and stored on ice in collection bags for transport to ALS Global laboratories in Kelso, Washington for analysis. In an effort to implement humane practices, larger fish will be dispatched by stunning the fish with an appropriately sized blunt tool just above the eyes on the back of the skull. Any fish not required for tissue analysis will be returned to the location in the lake from which they were collected prior to CO₂ immersion.

Based on the previous sampling in Johnson Lake (ARCADIS 2004b, Grette Associates 2017), it is unlikely that more than five fish of any fillet category or >125 mm will be collected during the course of sampling. If fish in any category continue to be collected after the goal of five individuals has been met, they may be used to replace previously collected fish to get a more spatially-representative sample of fish tissue in each category. This will be noted on field forms.

Each sampling location will be recorded using a dGPS. This will include locations where any fish collection was not successful. Water temperature and dissolved oxygen levels will be collected at each sample location in the middle water column, along with weather conditions.

2.2 TISSUE SAMPLE COLLECTION, DECONTAMINATION, AND PACKAGING

Filleting of fish and compositing of samples will be done in the analytical laboratory, not in the field. When preparing euthanized fish for transport to the lab, care will be taken to avoid cross-contamination. Nitrile gloves and aluminum foil work surfaces will be changed regularly to avoid contamination.

Individual fish will be rinsed with analyte-free-ionized water, wrapped in aluminum foil, bagged, labeled, and placed in an ice cooler for shipment to the analytical laboratory.

The analytical laboratory will composite the fillet and whole-fish samples into the single composite samples for each category (Section 3). Filleting of fish will be done according to the analytical laboratory's Tissue Sample Preparation Standard Operating Procedure (MET-TISP, dated 5/29/16). Additional filleting procedures and requirements can be found in Attachment B.

2.3 SAMPLE TRANSPORT AND CHAIN OF CUSTODY PROCEDURES

After collected and bagged/labeled, fish will be placed in the ice coolers and packed for shipping. The field representative will log each sample sent for analysis on a chain of custody (COC) form, noting sample identification, date and time of collection, requested testing, and comments as appropriate.

Chain-of-custody procedures will commence in the field and will track the delivery of the samples to the laboratory. Upon transfer of possession to the testing laboratory, the chain-of-custody forms will be signed by the persons transferring custody of the samples.

3 LABORATORY ANALYSIS

3.1 TESTING FACILITIES

The collected fish will be delivered to and processed at ALS Global in Kelso, WA. The processed samples will then be shipped to ALS's laboratory in Houston, Texas, for analysis.

3.2 LABORATORY CHAIN OF CUSTODY

A laboratory chain-of-custody record for each set of samples will be maintained throughout all sampling, processing, and analytical activities. Information tracked by the laboratory chain-of-custody records include a sample identification number, date and time of sample receipt, analytical parameters required, location and conditions of storage, date and time of removal from and return to storage, the signature of the person removing and returning the sample, the reason for removing from storage, and final disposition of the sample.

3.3 CHEMICALS OF CONCERN (CHEMICALS OF INTEREST) SCREENING

The evaluation of results shall consider total PCB, PCB TEQ, and lipid normalized total PCB concentrations in addition to PCB congener 126 concentrations to support evaluation of data trends over time (DEQ 2018). They will also be reported relative to tissue mass and tissue lipid content.

3.4 TESTING METHODS

PCB congeners will be analyzed using EPA method 1668C. The storage temperature for this analysis is 4°C, holding time is 14 days.

Table 1. Sample holding time and storage criteria

Sample Type	EPA Method	Holding Time	Temperature	Sample condition
Fish tissue	1668C	14 days until extraction	4° C	fillets (with belly flap attached); whole fish
Fish tissue (lipids)	NOAA or Bligh and Dyer (1959)	14 days until extraction	4° C	fillets (with belly flap attached); whole fish

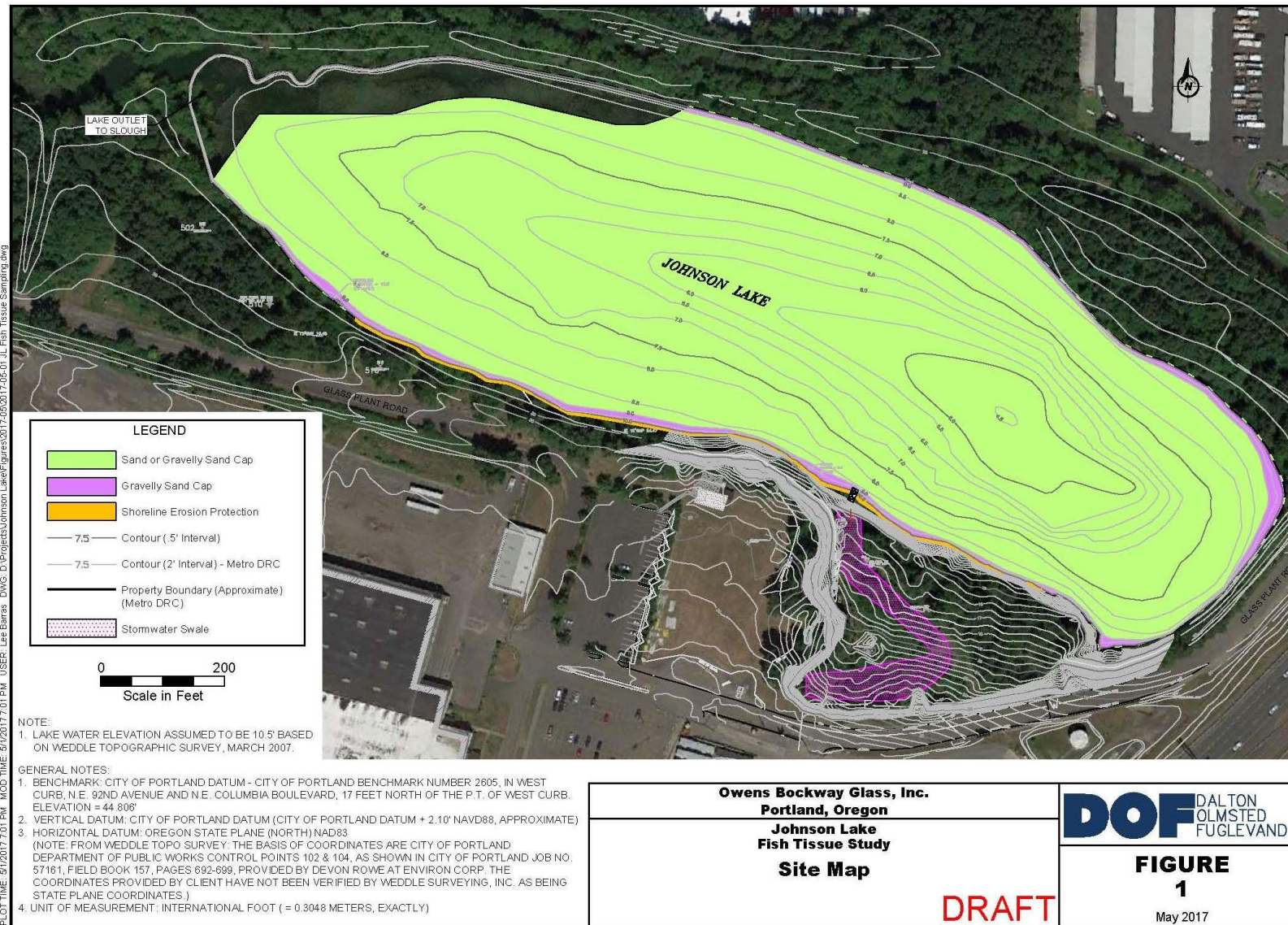
4 QUALITY ASSURANCE

All sampling and analysis will be done in accordance with the existing DEQ-approved Quality Assurance Project Plan (QAPP) for the Johnson Lake Remediation Project, as well as comments provided in the DEQ 2018 response letter to DOF.

5 REFERENCES

- ARCADIS. 2004a. Johnson Lake Investigation Work Plan, Revision 2. Prepared for Owens-Brockway Glass Container, Inc. Portland Oregon, by ARCADIS G&M, Inc., Cleveland, Ohio. January 29, 2004.
- ARCADIS. 2004b. Johnson Lake Site Investigation Report. Prepared for Owens-Brockway Glass Container, Inc. Portland Oregon, by ARCADIS G&M, Inc., Cleveland, Ohio. July 15, 2004.
- Dalton, Olmsted & Fuglevand, Inc. (DOF). 2012a. Project Completion Report – Sediment Remedial Action. Johnson Lake, Portland, Oregon. Prepared for Owens-Brockway Glass Container Inc. September 24, 2012.
- Dalton, Olmsted & Fuglevand, Inc. (DOF). 2012b. Remediation Operations & Maintenance Plan (O & M Plan): Sediment Remedial Action. Johnson Lake, Portland, Oregon. Prepared for Owens-Brockway Glass Container Inc. December 20, 2012.
- Grette Assoc. 2018. Revised Johnson Lake Fish Tissue Monitoring Study – Monitoring Report
- Oregon Department of Environmental Quality. (DEQ). 2018. Johnson Lake Fish Monitoring Study Comment Response Letter. Prepared for Dalton Olmsted Fuglevand
- United States Environmental Protection Agency. (EPA) .2015. EPA. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Fish Sampling and Analysis. Vol. 1 3rd edition. Office of Water. EPA 823-B-00-007
- Webb, R. personal communication. Phone call with M. Shelton, April 26, 2017.

Figure 1: Site Map of Johnson Lake



JOHNSON LAKE FISH TISSUE MONITORING STUDY



SAMPLING AND ANALYSIS PLAN

ATTACHMENT A: ODFW SCIENTIFIC TAKING PERMIT

JOHNSON LAKE FISH TISSUE MONITORING STUDY

SAMPLING AND ANALYSIS PLAN

ATTACHMENT B: ODEQ MAY 25, 2017, SAP APPROVAL AND COMMENTS



Oregon

Kate Brown, Governor

Department of Environmental Quality

Northwest Region Portland Office

700 NE Multnomah St., Suite 600

Portland, OR 97232

(503) 229-5263

FAX (503) 229-5471

TTY 711

May 25, 2017

Mr. Rob Webb, Principal Engineer
Dalton Olmsted Fuglevand
1236 Finn Hill Road
Poulsbo, WA 98370

RE: 2016 Johnson Lake Fish Tissue Monitoring Study
ECSI # 2086

Dear Rob:

Thank you for providing the *Sampling and Analysis Plan* (dated May 9, 2016) for fish tissue monitoring in Johnson Lake, located west of I205 adjacent to the Owens Brockway Glass Container, Inc. facility (5850 NE 92nd Drive in Portland, Oregon). This plan was submitted as part of the long-term monitoring of the remedial action implemented by Owens Brockway Glass Container, Inc as described in the *Remediation Operations and Maintenance Plan-Sediment Remedial Action* (December 20, 2012). DEQ approves the plan assuming the following:

1. A Scientific Taking Permit for fish is obtained from Oregon Department of Fish and Wildlife and included as an appendix to the fish tissue sampling results report.
2. All methods indicated in Section 2.1 will be utilized throughout the lake including off-shore areas of deeper water unless sufficient samples are obtained with a subset of the methods. This includes boat electro-shocking, deeper water angling, and off-shore netting with gill and Fyke nets. We expect these methods will be necessary to obtain representative fish, particularly those fish large enough to support determinations on fish advisories.
3. Fish sampling, processing and analysis procedures will follow EPA guidance provided in *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories Volume 1 Fish Sampling and Analysis, 2000* (<https://www.epa.gov/fish-tech/epa-guidance-developing-fish-advisories>).
4. A minimum of three fish will be included in each composite consistent with EPA guidance (five is preferred). Individual fish comprising a composite should be of similar size (smallest individual in a composite should be no less than 75% of the total length of the largest individual). For evaluation of impacts to human health and larger ecological receptors, fish should be of harvestable size or weight. Please coordinate with DEQ

during sampling so that we can participate in reviewing collected fish and determining an appropriate compositing plan.

5. Processing of fillets will be conducted in the laboratory rather than in the field. Laboratory clean room conditions are required to properly contain and control fish fluids during resection. Laboratory standard operating procedures for fish processing, resection, and homogenate preparation should be provided to DEQ in advance of fish collection. Fillets should not be rinsed as a standard practice as suggested in the work plan. Rinsing is only a contingency if there is rupture of organs during filleting which should be avoided consistent with standard laboratory practices. If rupture occurs, please attempt to consult with DEQ prior to selecting fish for analysis.

Please call me if you have any questions at (503) 229-6148.

Sincerely,

A handwritten signature in blue ink, appearing to read "Jennifer Sutter".

Jennifer Sutter, Project Manager
NWR Cleanup and Tanks

Cc: Sue Sholl, Owens Illinois
Susan Smith, Owens Illinois
Rebecca Hillwig, Oregon Health Authority
Tim Dean, City of Portland
Jennifer Peterson, DEQ NWR Cleanup and Tanks
ECSI File #2086

JOHNSON LAKE FISH TISSUE MONITORING STUDY



SAMPLING AND ANALYSIS PLAN

ATTACHMENT C: ALS STANDARD OPERATING PROCEDURE MET-TISP (5/29/16) – TISSUE SAMPLE PREPARATION

ALS Standard Operating Procedure

DOCUMENT TITLE:
REFERENCED METHOD:
SOP ID:
REVISION NUMBER:
EFFECTIVE DATE:

TISSUE SAMPLE PREPARATION
N/A
MET-TISP
10
5/29/2016





STANDARD OPERATING PROCEDURE

SOP No.: MET-TISP
Revision: 10
Effective: 07/01/2014
Page 1 of 22

TISSUE SAMPLE PREPARATION

ALS-KELSO

SOP ID:	MET-TISP	Rev. Number:	10	Effective Date:	5/29/16
---------	----------	--------------	----	-----------------	---------

Approved By:

Date: 5/11/16

Department Manager/Technical Director - Jeff Coronado

Approved By:

Date: 5/11/16

QA Manager - Carl Degner

Approved By:

Date: 5/12/16

Laboratory Director - Jeff Grindstaff

Issue Date: _____

Doc Control ID#: _____

Issued To: _____

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature

Title

Date

Signature

Title

Date

Signature

Title

Date

Signature

Title

Date



TABLE OF CONTENTS

1.SCOPE AND APPLICATION	3
2.METHOD SUMMARY	3
3.DEFINITIONS	3
4.INTERFERENCES	4
5.SAFETY	4
6.SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE	4
7.STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS	4
8.APPARATUS AND EQUIPMENT	4
9.PREVENTIVE MAINTENANCE	5
10.RESPONSIBILITIES	5
11.PROCEDURE	5
12.QA/QC REQUIREMENTS	13
13.DATA REDUCTION AND REPORTING	13
14.CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA	13
15.METHOD PERFORMANCE	14
16.POLLUTION PREVENTION AND WASTE MANAGEMENT	14
17.TRAINING	14
18.METHOD MODIFICATIONS	15
19.REFERENCES	15
20.CHANGES SINCE THE LAST REVISION	15



TISSUE SAMPLE PREPARATION

1. SCOPE AND APPLICATION

- 1.1. This standard operating procedure describes procedures for the initial preparation of tissue samples prior to sample analysis. Customer-specific contracts or statement of works (SOWs) with alternate procedures will take precedence over this SOP.
- 1.2. This SOP is intended to provide guidance for the preliminary preparation of tissue samples prior to the sample aliquoting and analytical preparation described in individual analytical SOPs. The procedures described in this SOP also apply to compositing and subsampling of tissue samples for analyses to be subcontracted.

2. METHOD SUMMARY

- 2.1. Tissue samples are inherently heterogeneous requiring special considerations in order to obtain a truly representative sample aliquot for analysis. This SOP provides guidance for handling tissue samples prior to the sample preparation steps described in analytical SOPs. This SOP applies to samples delivered to the lab in whole body form or in the form of pre-dissected tissues.
- 2.2. The sample handling strategy must consider:
 - what analyses are to be performed (metals, organics, or both, and VOC or non-VOC),
 - how much sample is available
 - are the analyses to be performed on individual samples or composite homogenates,
 - are the analyses to be performed on whole body, edible portions or specific organs, and
 - are any of the analyses going to be subcontracted which may require subsampling.
- 2.3. Proper preparation and handling of tissue samples is required to obtain a representative sample, avoid contamination, and to ensure loss of sample and target constituents is minimized.

3. DEFINITIONS

- 3.1. Sample: The material presented to the laboratory for analysis or testing.
- 3.2. Sample Aliquot: A representative part or portion of a sample for analysis which is a fraction of the whole sample. See subsampling also.
- 3.3. Compositing: The process by which sample aliquots from two or more samples are united to form a combined sample which is subsequently analyzed.
- 3.4. Subsampling: The process by which a representative portion is obtained from a whole sample.



STANDARD OPERATING PROCEDURE

SOP No.: MET-TISP
Revision: 10
Effective: 07/01/2014
Page 4 of 22

-
- 3.5. Service Request: The service request (SR) is a document prepared at the time of sample receipt and summarizes sample analysis and reporting instructions about a customer's sample(s).
 - 3.6. QAPP: Quality Assurance Project Plan document provided by the client specifically written for their project.
 - 3.7. VOC Analyses: Volatile organic compounds (VOC) analyses, including halogenated and aromatic volatile organic compounds and gasoline range organics (GRO) analyses.
 - 3.8. Non-VOC Analyses: Any analysis other than a VOC analysis.

4. INTERFERENCES

- 4.1. If precautions are not taken, cross-contamination can occur when handling tissue samples in large quantities. Equipment must be thoroughly cleaned as described in this SOP and related SOPs. Also, the SOP describes the use of homogenization and rinsate blanks to monitor any possible contamination.
- 4.2. For organics samples, polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials.

5. SAFETY

- 5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.
- 5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

- 6.1. Refer to the determinative method.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

- 7.1. Not applicable to this procedure.

8. APPARATUS AND EQUIPMENT

Note: Refer to the Procedure section for specific equipment used based on the determinative analysis to be performed. The use of implements and surfaces may vary depending on the analyses to be performed.

- 8.1. Hobart Food Chopper, or comparable device.
- 8.2. Tisumizer.



- 8.3. Waring blender, or similar device.
- 8.4. Freeze-drier, Labonco or equivalent.
- 8.5. Glass or PTFE cutting boards.
- 8.6. Knives and cutting implements – refer to Procedure section.
- 8.7. Standard laboratory glassware (beakers, scintillation vials, etc.)
- 8.8. VOA vial – pre-cleaned, 40ml with Teflon-lined cap.
- 8.9. Pre-cleaned glass jars with PTFE lined lids, various sizes.
- 8.10. Gloves: Should be talc free and of non-contaminating materials.
- 8.11. Heavy duty aluminum foil.

9. PREVENTIVE MAINTENANCE

- 9.1. No specific maintenance steps are needed other than normal cleaning and inspection of apparatus.
- 9.2. For organics samples, polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials.

10. RESPONSIBILITIES

- 10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.
- 10.2. Sample custodians, together with project chemists and department supervisors, are responsible for documenting any required sample preparation (including the percent solids or percent lipids determination if required) on the service request. All personnel preparing tissue samples should be familiar with the contents of this document prior to commencing work.
- 10.3. Tissue sample preparation is to be performed only by lab analysts instructed in the proper handling techniques outlined in this SOP. It is the responsibility of the analyst to perform this procedure to complete all documentation required for data review.

11. PROCEDURE

- 11.1. Sample Login



Any special sample handling must be noted on the service request and on a label attached to the sample itself. During sample receiving, a sample custodian must follow the procedures described in Section 11.1.1.

11.1.1. Tissue Samples with Limited Quantity

An assessment of the required sample quantity should be made by the project chemist when taking delivery of the sample. This assessment must take into consideration the tests, the required detection limits and the necessary quality assurance samples. If the quantity of sample given to the laboratory is insufficient for the analyses requested, the sample custodian will, along with the project chemist estimate the total amount of sample available. A "LIMITED SAMPLE VOLUME" tag is attached to the sample on which is recorded the estimated sample quantity.

In some cases it may be beneficial to perform sample preparations as described in this SOP prior to estimating the sample amount. In this case, the analyst preparing the sample will provide the project chemist with an estimate of the amount available.

The project chemist must determine if limited sample quantity exists and set the priorities for the analyses and, if possible, estimate the quantity of sample to be *used for each test*. This information is to be documented and placed in the project file and on the service request to communicate to the laboratory staff. For example, 8081 use 10 g; metals use 1g.

NOTE. Samples that are quantity limited and require multiple analyses must be identified as soon as possible. Optimally, this should happen during sample login; however, discovery at any time should trigger appropriate actions as described in Section 11.1.1.

11.2. Sample Homogenization

This section outlines the steps for preparing homogenous samples of whole fish, edible fish (fillets), edible shellfish, worm composite homogenates, eggs, and plant tissues.

11.2.1. Samples for Organics Analyses:

Equipment used for the processing of tissue samples for organics analyses should be of stainless steel, anodized aluminum, glass or polytetrafluorethylene (PTFE). Polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials. Filleting should be done on glass or PTFE cutting boards that are cleaned properly between samples or on cutting boards that are covered with heavy duty aluminum foil (hexane rinsed) that is changed between samples. Tissue should be handled with precleaned, high quality, corrosion-resistant stainless steel instruments. Fillets or homogenate should be stored in cleaned glass jars of suitable dimensions with PTFE lined lids. If the sample is to be analyzed for VOCs, the homogenization steps should be performed on sample tissue that is partially frozen or chilled. An aliquot of the homogenate should be placed in a clean 40ml vial and labeled "FOR VOA ANALYSIS ONLY".



STANDARD OPERATING PROCEDURE

SOP No.: MET-TISP
Revision: 10
Effective: 07/01/2014
Page 7 of 22

Prior to handling each sample, utensils, cutting boards and containers should be washed in a detergent hot water solution and rinsed with tap water, hexane, and DI water. Pre-cleaned, certified sample containers may be used without further cleaning. If the sample is to be analyzed for VOCs, methanol is substituted for the rinsing of implements with acetone and the hexane rinsing of the aluminum foil. Exposure to solvent vapors must be minimized.

11.2.2. Samples for Metals Analyses:

Equipment used in the processing of samples for metals analyses should be of PTFE, ceramic, polypropylene or polyethylene. Filleting should be performed on PTFE cutting boards which are cleaned after each sample. Knives with titanium or high quality stainless steel blades may be used for tissue resections. Tissue should be stored in glass jars with PTFE lined lids.

Prior to sample handling, utensils, cutting boards and containers should be washed in a detergent hot water solution, rinsed with tap water, 25% HCL (except metal utensils), and DI water. Pre-cleaned, certified sample containers may be used without further cleaning.

11.2.3. Samples for both Metals and Organics Analyses:

If the sample is to be prepared for both organics and metals, care must be taken to use equipment and cleaning procedures that are non-contaminating for both. Quartz, ceramic, glass and PTFE are recommended materials for sample processing equipment. Knives with titanium or high quality stainless steel blades may be used for tissue resections. Glass or PTFE cutting boards should be used. If the sample is to be analyzed for VOC's, the homogenization steps should be performed on sample tissue that is partially frozen or chilled. An aliquot of the homogenate should be placed in a clean 40ml vial and labeled "FOR VOA ANALYSIS ONLY".

Prior to handling each sample, utensils, cutting boards and containers should be washed in a detergent hot water solution and rinsed with tap water, acetone, methanol, or hexane (as appropriate), and DI water. Precleaned, certified sample containers may be used without further cleaning. Non-metallic surfaces and utensils should also be rinsed with 25% HCL followed by DI water. If the sample is to be analyzed for VOCs, methanol is substituted for the rinsing of implements with acetone. Exposure to solvent vapors must be minimized.

11.2.4. Sample Preparation

Each tissue sample may be homogenized in the original glass bottle container if there is sufficient space to allow thorough mixing. If homogenization is not achievable in the original container, place the entire sample contents into a clean glass jar. Generally, liquids contained in the container are to be considered part of the sample. If the sample requires size reduction prior to homogenization, chop the sample into the 1-2" chunks using a titanium or stainless steel bladed knife. Large samples may require the use of industrial food processors such as a Hobart Food Chopper, or comparable device. Size-reduced chunks of tissue are thoroughly homogenized to a paste-like consistency using a Tisumizer, Waring blender, or similar device until it reaches a paste-like consistency. Transfer the sample paste to a glass jar for storage and freeze until ready for sample extraction. The new sample



container is labeled with the sample I.D., the word “homogenized”, initialed, and dated.

11.2.4.1.Whole Fish Tissue

Samples may be frozen in the field or in the laboratory. While still partially frozen, rinse the fish with DI water to remove extraneous materials and liquids. Cut the fish into appropriate size chunks and mechanically macerate the sample using cutting tools appropriate for the size of the sample and the analysis type. If necessary, process fish tissue chunks through the Hobart Food Chopper. To ensure thorough mixing, divide the ground sample into quarters, mix opposite quarters and then mix halves. Homogenize sample using a Tisumizer or Waring blender until it reaches a paste-like consistency. Transfer the sample paste to a glass jar for storage and freeze until ready for sample extraction. The new sample container is labeled with the sample I.D., the word “homogenized”, initialed, and dated.

11.2.4.2.Edible Fish Tissue

If the client or QAPP indicates that only edible tissue be analyzed, the fish must be filleted. If the sample arrives pre-filleted, the sample tissue may be frozen before processing. If the sample is not yet filleted, the sample should remain chilled until the filleting is completed. Freezing can result in the contamination of edible tissues from the bursting of internal organs. Fish having ruptured internal organs should be noted on the prep benchsheet and the Project Chemist consulted. Rinse the fish with DI water to remove extraneous materials and liquids. Remove scales from scaled fish or skin from non-scaled fish. Rinse the fish again prior to filleting. A separate or clean cutting board should be used for filleting. Gloves should be changed between samples. Carefully remove the fillets from the carcass by following the steps outlined in Appendix A. Care should be taken to avoid contaminating fillet with inadvertent puncture of internal organs. Cut the fillet tissue into appropriate size chunks and mechanically macerate the sample using cutting and grinding tools appropriate for the size of the sample and the analysis type. Proper selection of maceration equipment must consider the potential contaminants, sample size/volume and amount of tissue likely to be lost in using the equipment.

Divide the ground sample into quarters, mix opposite quarters and then mix halves. Again homogenize the sample using an appropriate blending mixer. Continue repeating this process until the sample is truly homogenous and no chunks of tissue remain. Freeze sample until ready for extraction.

11.2.4.3.Shellfish Tissue

Shellfish should be frozen as soon as possible after receipt by the laboratory unless samples can be prepared within 48 hours of sampling. Edible portions of various shellfish are described below and resection described in Appendix B. Thawing of frozen shellfish samples should be kept to a minimum during tissue removal to avoid loss of liquids. Shellfish should be rinsed with DI water prior to tissue removal to dislodge external debris. When multiple



organisms constitute a single sample, the edible tissues are collected, composited and homogenized.

11.2.4.3.1. Bivalve mollusks (oysters, clams, mussels, and scallops).

Bivalves are typically prepared by severing the adductor muscle, prying open the shell, and removing all of the soft tissue. The soft tissue includes viscera, meat, and body fluids.

11.2.4.3.2. Crabs

Edible tissue includes all leg and claw meat, back shell meat and body cavity meat. Internal organs generally are removed. If the crab is soft shelled, the entire crab is used in the sample.

11.2.4.3.3. Shrimp and Crayfish - Edible tissue includes the tail meat.

11.2.4.3.4. Lobster - Edible tissue includes the tail and claw meat.

11.2.4.4. Worms

Samples are typically supplied to the lab in sample jars containing multiple organisms. Liquid and specimens constitute the entire sample and are blended together typically in the sample container. When a worm sample containing dirt particles or significant amounts of water is encountered, the technician should contact the project chemist to seek guidance from the client.

11.2.4.5. Eggs

Avian eggs are typically removed from the shell and blended. Aquatic eggs are blended including the soft shell.

11.2.4.6. Internal Organs Extraction

Organs such as livers or kidneys must be identified and removed by an experienced sample technician following clear written resection procedures or other guidance provided by the client.

11.2.4.7. Plant Tissue

Plant tissue should be handled using the size reduction, homogenization and implement cleaning steps outlined in Sections 11.2.1, 11.2.2, 11.2.3, and 11.2.4. Where these procedures are inappropriate, specific written procedures or guidance from the client is recommended.

If drying is requested by the client or is project-specified, a subsample for mercury analysis is taken from the wet sample, and then the plant tissue is dried at 60°C prior to homogenization.

11.2.4.8. Small Mammals and Rodents



STANDARD OPERATING PROCEDURE

SOP No.: MET-TISP
Revision: 10
Effective: 07/01/2014
Page 10 of 22

-
- 11.2.4.8.1. There are two primary concerns in working with small mammals and rodents: safety and sample homogenization.
- 11.2.4.8.2. Small mammals are potential carriers of lethal viruses, such as hantavirus and rabies, and bacteria that can be contracted through inhalation or direct contact. Typically, these organisms are excreted in the feces and distributed on the air as the fecal matter dries. During the sample preparation process, tissue is typically freeze-dried in order to calculate a percent solids value and to analyze for metals. As such, it is possible to increase the potential for dispersion of the bacteria or viruses after the sample is homogenized and processed. Prior to processing, all samples should be stored frozen.
- 11.2.4.8.3. Prior to sample homogenization, instructions should be received from the client regarding the processing of the hide. For organics, it is recommended that the hides be left on the carcass and the entire sample be homogenized. For metals, there is a potential for accumulation in the hair. As a non-digestible portion of the rodent, inclusion of the hair may result in a high bias if the data is to be used in estimating bioaccumulation up the food chain. Skinning may be a preferred alternative when metals are the primary chemicals of concern.
- 11.2.4.8.4. Homogenization should be done while the carcass is still partially frozen.
- 11.2.4.8.5. If the hide is to be included in the homogenization, snip the feet from the animal using stainless steel scissors.
- The tail should be removed if it will prevent complete homogenization of the sample (e.g., the tail of a mouse or rat may result in incomplete homogenization and should not be included with the sample). Remove seeds, grasses, and mud from the hide.
- 11.2.4.8.6. If the hide is to be removed from the carcass, make an incision through the skin on the back of the neck (do not cut into the muscle). In most cases, the hide can be removed by pulling the incision horizontally along the back in one direction, and over the ears, head and snout in the opposite direction. The eyes are usually lost during this procedure. Continue to skin the animal by peeling the hide over the hind legs, off the underside of the animal, and around the front legs. The hide is removed at the hind legs and the snout. Care should be taken not to tear the connective tissue under the hide. Fat should be scraped from the hide when possible and included with the sample. Rinse the skinned carcass with DI water to remove any hair or dirt that has accumulated during the skinning procedure.
- 11.2.4.8.7. Homogenize the sample using a stainless steel Waring blender. Select a blender cup that is sized in accordance with the amount of sample to be homogenized. That is, small samples should be homogenized using small blender cups. This will improve the overall homogenization and recovery of the sample. Continue to mix the



sample into a paste like consistency. Make sure no chunks of muscle, hide, or bone are distributed in the sample. Transfer the sample paste to a glass jar for storage and freeze. The new sample container is labeled "homogenized", initialed, and dated.

11.2.5. General Provision for Handling Large Sample Mass

In some cases, large specimens will be received by the laboratory for homogenization prior to chemical analysis. For the purpose of this SOP, 'large' is defined as requiring preliminary size reduction to allow sequential processing of the sample. Sub-samples of the whole specimen should be cut to a size appropriate for the blender, mixer, or grinder that will be used. After each individual fraction is processed, the homogenized material is added to a reservoir large enough to hold all fractions as they accumulate. The reservoir will be constructed of a material suitable for the analytical application as defined under Section 10.2.3. For very large specimens (i.e. >20 pounds), high grade stainless steel containers are used (large bowls or small drums).

Blending of the combined fractions to achieve a whole homogenous material is achieved via manual mixing. In general, this is accomplished using a high grade stainless steel paddle or spoon of appropriate size (i.e. relative to the whole homogenate). Very large specimens (i.e. >20 pounds) generally require secondary processing through the grinder, particularly when large amounts of skin, bone, and/or cartilage is present. In these cases, the Hobart grinder is generally used.

11.3. Compositing

Each sample is to be logged in and receive a lab code. Additionally, the sample composite also is assigned a lab code. The compositing process is to be performed by trained staff. It is to be performed in an area free of contamination. It is imperative that the samples are treated in a manner consistent with the requirements of the tests to be performed on the composited sample. Compositing of homogenates should be performed according to this SOP or specific instructions provided by the client.

11.3.1. Documentation (use applicable bench sheet)

The analyst preparing the composite will document

- that homogenization was done before removing an aliquot,
- the quantity of each (field or discrete) sample used for the composite,
- the date and time of compositing, and
- any other pertinent observations.

11.3.2. Tissue Samples with Limited Quantity

Samples and sample composites that are quantity limited will be handled by the same procedure as described in Section 10.1.1

11.3.3. Compositing Procedure

11.3.3.1. Each tissue sample is first homogenized as per instructions in Section 11.2.



11.3.3.2. An equal weight of sample aliquot from each of the homogenized samples is weighed into a clean glass sample bottle. The amount to be weighed of each sample will depend upon the number of analyses to be performed on the composite and if the quantity of any individual sample is limited.

11.3.3.3. The mixture of the individual sample aliquots is thoroughly homogenized in the glass container. The composite sample bottle is labeled with:

- the name of the composite,
- the lab code of the composite,
- the analyst's initials
- the date of composite preparation
- The composite sample and the remaining (individual, discrete) samples are stored frozen until analysis.

11.3.4. Tissue Samples Requiring VOC Analyses

A separate aliquot of the composite homogenate should be placed in 40ml vial container for later analysis by the VOA department. Each container should be labeled with the lab identifier, date, initials, and "FOR VOA ANALYSIS ONLY". To minimize losses of volatile constituents, the sample should be kept as cold as possible, the work should be completed as quickly as possible, and the VOA vial filled to the top to minimize head space.

11.4. Sub-sampling

The sample is first thoroughly homogenized as per Section 11.2. A sample aliquot is removed and placed into a clean glass container of appropriate size and labeled as follows:

- the name of the sample,
- the lab code of the sample,
- "homogenized" written on the label,
- the purpose of the sub-sample (e.g. "dioxin subsample")
- the analyst's initials
- the date.

11.5. Freeze-Drying

11.5.1. Depending on project specifications, samples may require freeze-drying. Freeze-drying may be performed on a separate portion of sample to determine % Freeze-Dried Solids, or may be done on the analytical subsample for certain tests. The analyst should obtain direction from the supervisor and/or Project Chemist.

11.5.2. Weigh 5-8 g of sample (wet weight) into a scintillation vial. Freeze the sample for at least 2 hours.

11.5.3. Remove the sample from the freezer and place in the freeze drier for at least 24 hours or longer if necessary for the particular sample matrix.

11.5.4. Record the measurements on the applicable bench sheet.



- 11.5.5. When freeze drying samples that require PFOA/PFOA, aluminum foil must be placed on the container before putting the lid on, after the freeze drying process and before freezing the sample.

12. QA/QC REQUIREMENTS

- 12.1. A rinsate blank should be prepared to accompany each batch of tissue samples. The blank is comprised of a collection of DI water rinses of cleaned equipment (knives, cutting boards and mixers/grinders) *prior* to the commencement of sample batch preparation. If contamination of the samples is suspected, the rinsate blank is extracted and analyzed for contaminants. The rinsate blank should be labeled with the extraction date and the associated SR numbers and stored at 4° C. In the event that contamination is suspected, the rinsate blank can be analyzed to confirm the presence of contaminants in the tissue preparation process.
- 12.2. A homogenization blank is prepared to determine if the homogenization equipment was effectively cleaned between samples. Unless a project plan specifies otherwise, the laboratory prepares two homogenization blanks with each shift of sample preparation. One is a 500 mL aliquot for Metals testing and the other is a 1000 mL aliquot for Organics testing. Any requirements other than the labs default procedure must be defined in the project plan and communicated to the laboratory.
- 12.2.1. Some project quality plans may require homogenization blanks between each sample. Following the blending of a tissue sample decontaminate the Hobart mixer (model HCM62) by following these steps:
- Wash the bowl, blade assembly, and lid with soap and hot water.
 - Rinse all parts with deionized water.
 - Move to fume hood and hexane rinse all parts.
 - Allow excess hexane to evaporate.
- 12.2.2. Reassemble the mixer and make ready for the next sample.
- 12.2.3. Fill the bowl with deionized water and turn the mixer on for the approximately average time used for the type of samples being processed.
- 12.2.4. Aliquot the deionized water to bottles appropriate for the testing being conducted and preserve accordingly. If insufficient sample volume is produced for the required testing, repeat the procedure after the next tissue sample is homogenized.

13. DATA REDUCTION AND REPORTING

- 13.1. Sample handling documentation must include information about sample homogenization (was it done or not), compositing, and sub-sampling. The established and applicable data bench sheets provide a means for recording this information. Completed bench sheets listing the sample handling information are filed in the project file with the raw data.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA



STANDARD OPERATING PROCEDURE

SOP No.: MET-TISP
Revision: 10
Effective: 07/01/2014
Page 14 of 22

-
- 14.1. Refer to the SOP for *Non Conformance and Corrective Action (CE-QA008)* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.
- 14.2. Handling out-of-control or unacceptable data
- 14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.
- 14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):
- Quality control results outside acceptance limits for accuracy and precision
 - Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
 - Sample holding time missed due to laboratory error or operations
 - Deviations from SOPs or project requirements
 - Laboratory analysis errors impacting sample or QC results
 - Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc)
 - Sample preservation or handling discrepancies due to laboratory or operations error

15. METHOD PERFORMANCE

- 15.1. Refer to determinative methods.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

- 16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.
- 16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.
- 16.3. This method uses non-halogenated solvents and any waste generated from this solvent must be collected. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

17. TRAINING

- 17.1. Training outline – Training Plan



STANDARD OPERATING PROCEDURE

SOP No.: MET-TISP
Revision: 10
Effective: 07/01/2014
Page 15 of 22

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst until the supervisor feels the new employee can work independently. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.2. Training is documented following ADM-TRAIN, ALS-Kelso Training Procedure.

When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

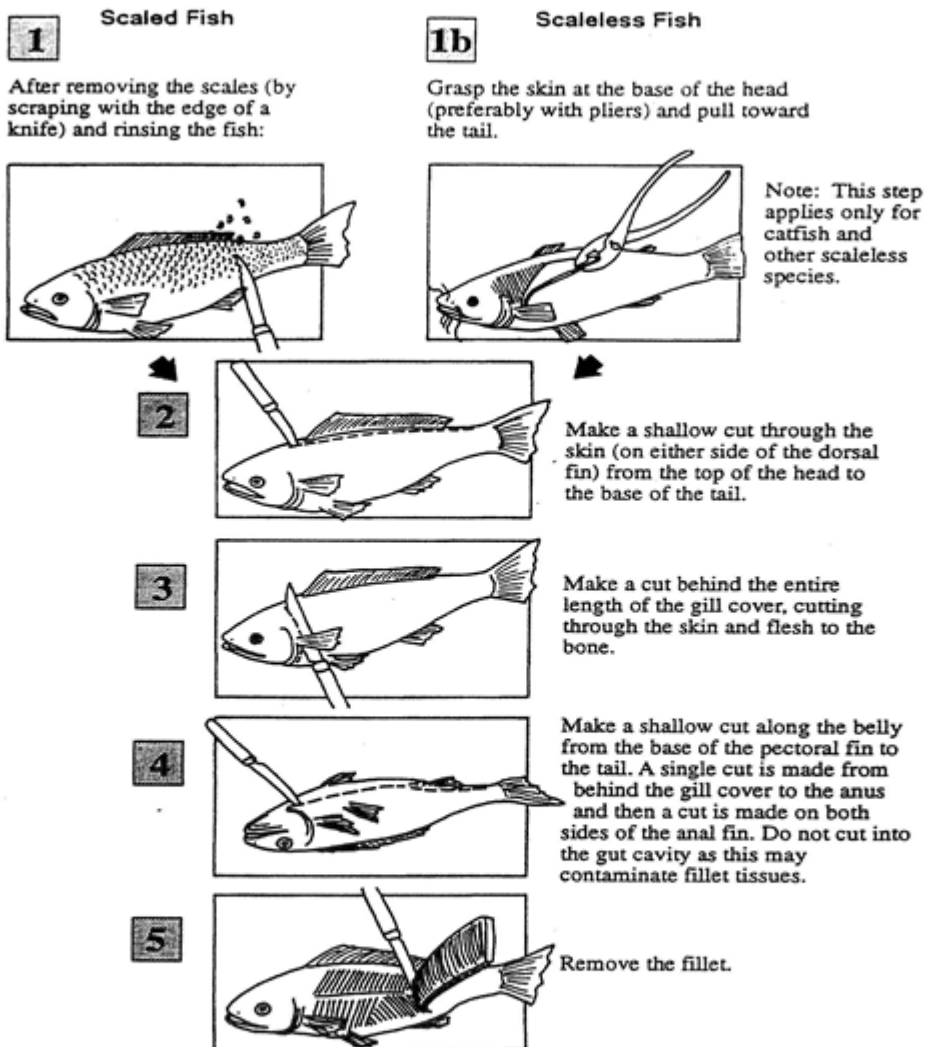
18.1. This section is not applicable because this procedure is a laboratory developed method.

19. REFERENCES

- 19.1. Kateman and L. Buydens, *Quality Control in Analytical Chemistry*, Second Edition, John Wiley & Sons, Inc., New York, NY, 1993: Chapter 2 on Sampling and especially sections 2.5 (Sample Quality) and 2.7 (Handling of Samples).
- 19.2. *Guidance For Assessing Chemical Contaminant Data For Use In Fish Advisories*; Volume 1; Fish Sampling and Analysis, 3rd Edition; USEPA Office of Water; EPA 823-B-00-007; Nov 2000.
- 19.3. *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*; Tetra Tech, Inc.; final report TC-3991-04 Recommended Guidelines for Measuring Organic Compounds in Puget Sound Sediment and Tissue Samples Revision April 1996.
- 19.4. *PCB's and Mirex In Fish Tissue and Clams* New York State Department of Health Wadsworth Center For Laboratories and Research; Albany, N.Y. 10/6/81
- 19.5. *Draft Method 1613-Tissue*; Determination of PCDDs and PCDFs in Fish and Other Tissue Using Method 1613; USEPA Office of Water June 1993.

20. CHANGES SINCE THE LAST REVISION

- 20.1. Signature page: Updated Quality Assurance manager.
- 20.2. Added the sentence in Section 11.5.5.

**APPENDIX A**
Fish Filleting Procedure**7. LABORATORY PROCEDURES I — SAMPLE HANDLING**

Source: U.S. EPA, 1991d.

Figure 7-3. Illustration of basic fish filleting procedure.

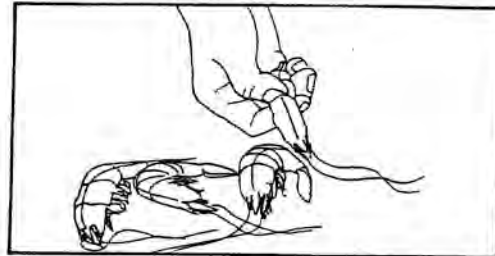


APPENDIX B
General Procedure for Removing Edible Tissues from Shellfish

Heading, peeling and deveining shrimp

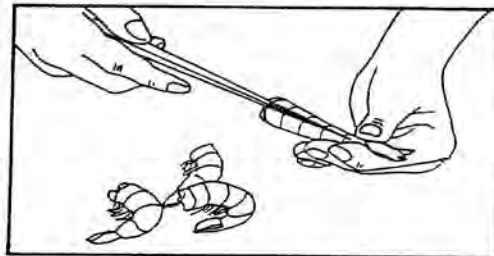
1

To head a shrimp, hold it in one hand. With your thumb behind shrimp head, push head off. Be sure to push just the head off so that you do not lose any meat.



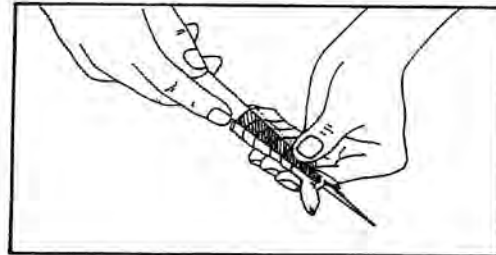
2

If using a deveiner, insert it at head end, just above the vein.



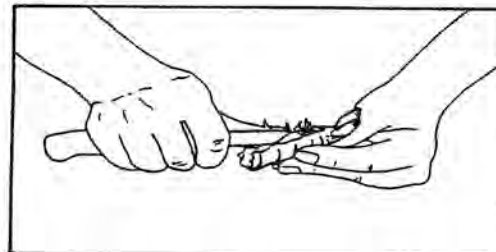
3

Push through shrimp to the tail and split and remove shell. This removes vein at the same time.



4

If you prefer to use a paring knife, shell shrimp with your fingers or knife. Then use knife to gently remove vein.



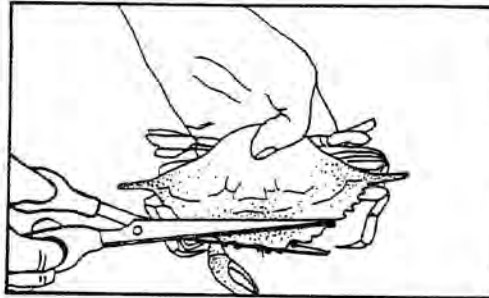
Source: UNC Sea Grant Publication UNC-SG-88-02



Cleaning soft-shell crabs

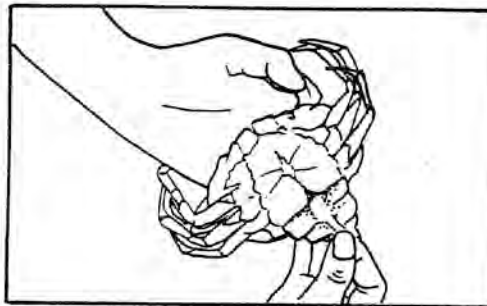
1

Hold crab in one hand and cut across body just behind eyes to remove eyes and mouth.



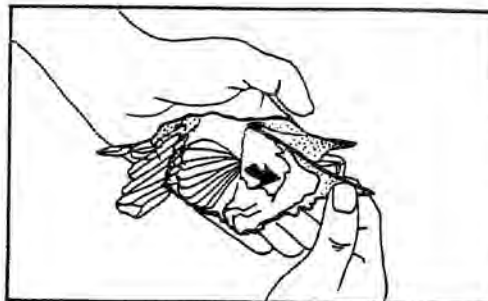
2

Turn crab on its back. Lift and remove apron and vein attached to it.



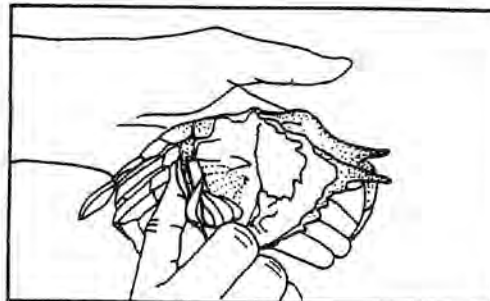
3

Turn crab over and lift one side of top shell.



4

With a small knife, scrape off grayish-feathery gills. Repeat procedure on other side.



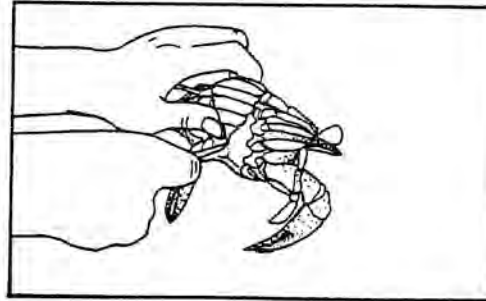
Source: UNC Sea Grant Publication UNC-SG-88-02



Cleaning hard-shell crabs

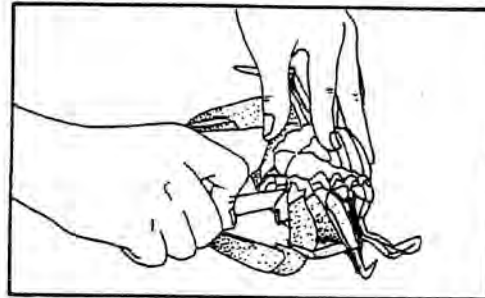
1

Hold crab in one hand. Turn crab over and stab straight down at point of apron with a knife.



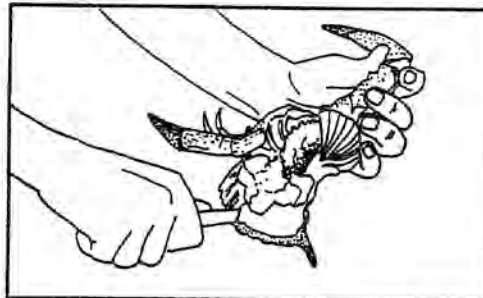
2

Make two cuts from this point to form a V-pattern that will remove mouth.



3

Do not remove knife after making second cut. Firmly press crab shell to cutting surface without breaking back shell. With other hand, grasp crab by legs and claws on the side where you are holding knife, and pull up. This should pull crab body free from back shell.



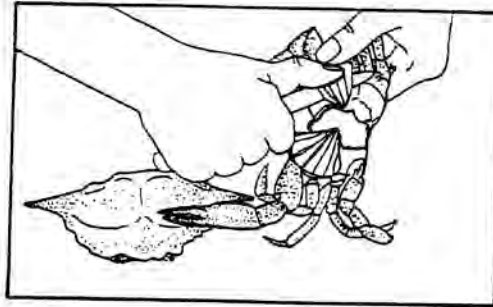


STANDARD OPERATING PROCEDURE

SOP No.: MET-TISP
Revision: 10
Effective: 07/01/2014
Page 20 of 22

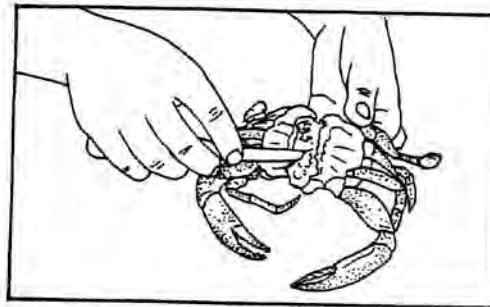
4

Remove gray, feathery gills, which are attached just above legs. Cut and scrape upward to remove gills.



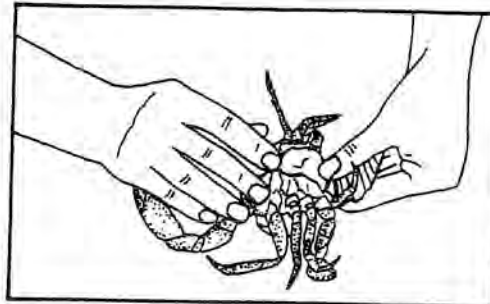
5

Remove all loose material—viscera and eggs—from body cavity.



6

If apron did not come loose with shell, remove it.



Source: UNC Sea Grant Publication UNC-SG-88-02



Shucking oysters

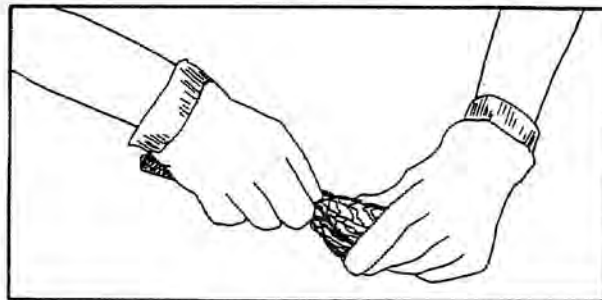
1

Oyster shells are especially sharp; be sure to wear gloves to protect your hands. Chip off a small piece of shell from the thin lip of the oyster until there is a small opening.



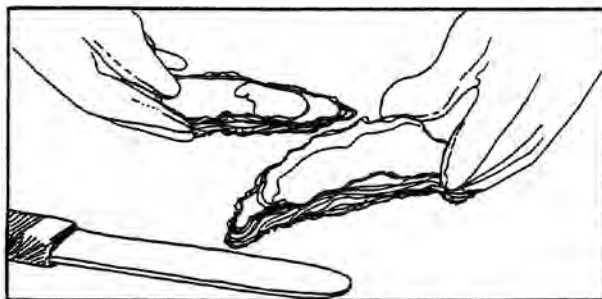
2

Insert knife blade into the opening and cut muscle free from top and bottom shells.



3

Remove oyster meat from the shell.



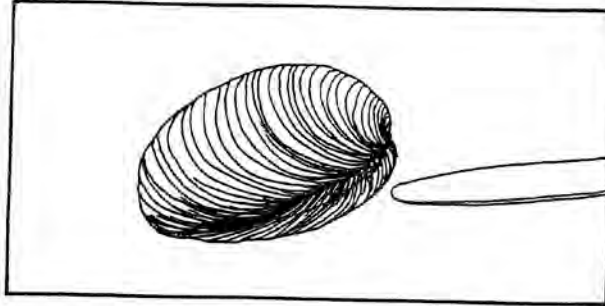
Source: UNC Sea Grant Publication UNC-SG-88-02



Shucking clams

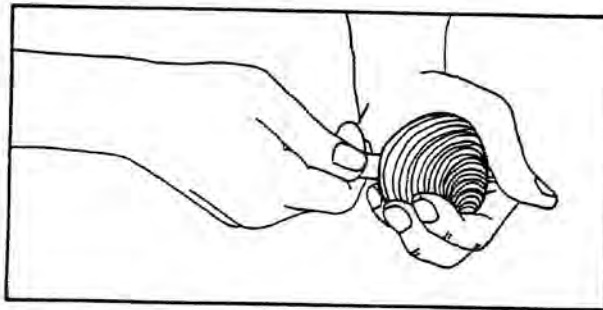
1

In the back of clam near the hinge is a black ligament. Toward the front where ligament ends is a weak spot. Insert your knife at this spot.



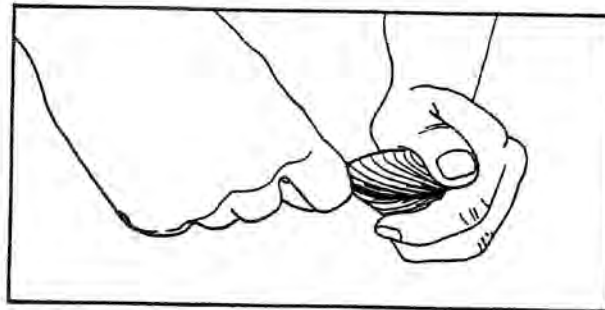
2

Inside are two muscles. Run the knife around the shell to sever both muscles.



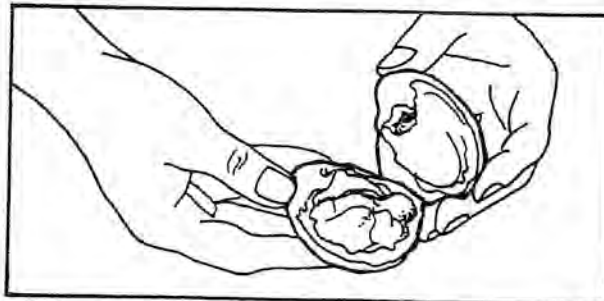
3

Now insert the knife blade into the front of the shell and separate the two shells.



4

Scrape the meat free from the top and bottom shell.



Source: UNC Sea Grant Publication UNC-SG-88-02

JOHNSON LAKE FISH TISSUE MONITORING STUDY



SAMPLING AND ANALYSIS PLAN

APPENDIX D: ODEQ January 29, 2018, Letter and Comments



Oregon

Kate Brown, Governor

Department of Environmental Quality

Northwest Region Portland Office

700 NE Multnomah St., Suite 600

Portland, OR 97232

(503) 229-5263

FAX (503) 229-5471

TTY 711

January 29, 2018

Mr. Rob Webb, Principal Engineer
Dalton Olmsted Fuglevand
1236 Finn Hill Road
Poulsbo, WA 98370

RE: 2016 Johnson Lake Fish Tissue Monitoring Study
ECSI # 2086

Dear Rob:

Thank you for providing the *Revised Johnson Lake Fish Tissue Monitoring Study – Monitoring Report* (dated January 19, 2018) documenting the results of fish tissue monitoring conducted in Johnson Lake, located west of I205 adjacent to the Owens Brockway Glass Container, Inc. facility (5850 NE 92nd Drive in Portland, Oregon). This report was submitted as part of the long-term monitoring of the remedial action implemented by Owens Brockway Glass Container, Inc. as described in the *Remediation Operations and Maintenance Plan-Sediment Remedial Action (OMP)* (December 20, 2012). The revised version addresses comments provided by DEQ (letter dated December 18, 2017) and DEQ approves the revised report.

As noted in our previous letter, because PCBs are still present in fish tissue in Johnson Lake at levels that exceed protective concentrations for human consumption, warning signs must be maintained and fish must be sampled again in 2022. Documentation of the 2022 fish tissue sampling event should include the information provided in the 2018 revised report as well as a summary of the results from the 2017 and 2004 events for comparison. In addition, the 2022 sampling effort and evaluation should incorporate the following:

1. The level of effort detailed in the 2018 report was not sufficient to meet data quality objectives specified in the OMP. The level of sample collection effort at the 2022 sampling event should be sufficient to ensure that the required number of fish specified in the OMP are collected and that fish are collected throughout the lake. Fish in each category must adequately represent that category; e.g., samples representing fish likely to be consumed by humans should be large enough that this exposure would be reasonable. This may require sampling with different gear types at multiple times during 2022 to collect fish when lake conditions support capture. The use of a full range of available gear types (e.g. beach seining and gill nets) is not precluded by the presence of algae and vegetation, but rather is used to ensure success in these conditions. Gear can be modified to exclude non-target species such as sturgeon and turtles.

2. The evaluation of results should consider total PCB, PCB TEQ, and lipid normalized total PCB concentrations in addition to PCB congener 126 concentrations to support evaluation of data trends over time.
3. The revised report did not clearly indicate that composite homogenates of fish tissue were created using equal mass from each individually homogenized fish sample. Lab protocols for sample preparation should specify this procedure will be followed.
4. Composite samples of each fish should be of the same type (species) and size (age) class. Consistent with EPA Guidelines (Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1 Fish Sampling and Analysis) the differences between lengths of individuals in a composite sample and the average of the lengths should not exceed 10 percent and at least one replicate should also be analyzed.

I am retiring at the end of this month. The new project manager for this site is Heidi Nelson. Please notify Heidi at (503) 229-6802 of any activities that have potential to expose underlying contaminated sediment in the lake and any necessary maintenance that is warranted. While it is a long way out, a draft sampling plan for the 2022 fish sampling event should be submitted to Heidi no later than 60 days in advance of the planned sampling.

Sincerely,



Jennifer Sutter, Project Manager
NWR Cleanup and Tanks

Cc: Sue Sholl, Owens Illinois
Susan Smith, Owens Illinois
Rebecca Hillwig, Oregon Health Authority
Tim Dean, City of Portland
Heidi Nelson, DEQ NWR Cleanup
Jennifer Peterson, DEQ NWR Cleanup
ECSI File #2086