PORTLAND HARBOR RI/FS

FIELD SAMPLING PLAN:

ROUND 3 SAMPLING FOR PRE-BREEDING WHITE STURGEON (*ACIPENSER TRANSMONTANUS*) TISSUE

February 9, 2007

DRAFT DOCUMENT: DO NOT QUOTE OR CITE
This document is currently under review by US EPA and its federal, state, and tribal partners, and is subject to change in whole or in part.

WE-06-0004

Prepared for:

The Lower Willamette Group

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LIST OF ACRONYMS

Axys	Axys Analytical Services, Ltd.
BERA	baseline ecological risk assessment
CAS	Columbia Analytical Services, Inc.
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
EPA	US Environmental Protection Agency
ERA	ecological risk assessment
FSP	field sampling plan
GI	gastrointestinal
GPS	global positioning system
HASP	health and safety plan
Integral	Integral Consulting, Inc.
LWG	Lower Willamette Group
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzo-*p*-dioxin
PCDF	polychlorinated dibenzofuran
PTFE	polytetrafluoroethylene
QA	quality assurance
QAPP	quality assurance project plan
QA/QC	quality assurance/quality control
QC	quality control
RI/FS	remedial investigation and feasibility study
RM	river mile
Study Area	Portland Harbor Superfund Study Area
SVOC	semivolatile organic compound
Windward	Windward Environmental LLC
WMG	wide-mouth glass

1.0 INTRODUCTION

This field sampling plan (FSP) describes the objectives, methods, and procedures for collecting tissue from pre-breeding white sturgeon (*Acipenser transmontanus*) within the Portland Harbor Superfund Study Area (Study Area), and for laboratory chemistry analyses of the samples. Determining selected contaminant concentrations in pre-breeding white sturgeon tissue will be helpful to complete the baseline ecological risk assessment (BERA), as outlined in the *Portland Harbor Remedial Investigation/ Feasibility Study Programmatic Work Plan* (Integral et al. 2004b). The residence time of sturgeon in the Portland Harbor and Lower Willamette River is not precisely known. Since younger sturgeon are assumed to travel less distance than their adult counterparts, it is assumed that pre-breeding sturgeon caught in the Study Area are likely to have greater exposure to the Study Area. This FSP presents a sampling and analysis design for collecting tissue samples and analysis of tissue samples for chemicals of interest (COIs).

1.1 OBJECTIVES OF PRE-BREEDING WHITE STURGEON TISSUE COLLECTION

The specific objectives of the Portland Harbor pre-breeding white sturgeon tissue sampling effort are to:

- Obtain site-specific pre-breeding white sturgeon whole-body tissue samples
- Determine whether COI concentrations in field collected white sturgeon tissues from the Portland Harbor site potentially pose unacceptable ecological risks to the sturgeon themselves.

1.2 DOCUMENT ORGANIZATION

The remaining sections of this document describe the sampling approach (Section 2.0), sample collection and processing methods (Section 3.0), and laboratory analytical methods (Section 4.0) that will be used in the Round 3 tissue study for pre-breeding white sturgeon. Reporting procedures are provided in Section 5.0, and references are listed in Section 6.0.

2.0 SAMPLING APPROACH

The sampling approach in this FSP will provide information on tissue concentrations to characterize concentrations of COIs in white sturgeon captured across a range of conditions throughout the Study Area.

Sample collection will require the use of set lines with multiple baited hooks to collect pre-breeding white sturgeon samples for tissue residue analyses.

Collection of pre-breeding white sturgeon will be attempted in five reaches of the Study Area from River Mile (RM) 2.0 to about RM 11 (Figure 2-1) for a maximum of three weeks. If the collection permits allow, three pre-breeding white sturgeon per reach will be collected using set lines with multiple baited hooks. The locations of the set lines within each reach will be determined in the field based on habitat preferences (i.e., deepwater pools). Incidental information on the distribution of pre-breeding white sturgeon in the deepwater pools will be collected using a catch per unit effort approach.

Figure 2-1. Proposed Round 3 sampling locations for white sturgeon (*A. transmontanus*) tissue

2.1 TEAM ORGANIZATION AND RESPONSIBILITIES

Windward Environmental LLC (Windward) will coordinate the overall field effort, collect the tissue samples, process the tissue samples in the field, provide on-water navigation support (including assisting in the field), and be responsible for sample handling and transport to the field laboratory. Integral Consulting, Inc. (Integral), will be responsible for on-water navigation, sample handling and storage at the field laboratory, and transport to the analytical laboratory. Specific staff assignments are detailed in Table 2-1.

Table 2-1. Team roles and responsibilities

Staff	Company	Role	Responsibility
Ian Stupakoff	Integral	field coordinator	• oversee planning and coordination • oversee staging area activities • oversee field lab – sample transport and storage
Thai Do	Windward	field coordinator/ field QA manager	• provide field collection QA • oversee tissue collection and processing QA
Keith Pine	Integral	CERCLA project coordinator	• coordinate overall RI/FS efforts
Lisa Saban	Windward	ERA project manager	• coordinate overall ERA activities
Gene Revelas	Integral	sampling and analysis coordinator	• manage RI tasks and coordinate field activities
Maja Tritt	Integral	lead chemist/ chemistry QA manager/ laboratory oversight manager	• coordinate laboratory analysis • oversee laboratory • coordinate data validation
Tom Schulz	Integral	lead database manager	• maintain accurate and updated project database • develop EPA database deliverables

CERCLA – Comprehensive Environmental Response, Compensation, and Liability Act
EPA – US Environmental Protection Agency
ERA – ecological risk assessment
QA – quality assurance
RI/FS – remedial investigation/feasibility study

The communication flow for the tissue sampling effort will generally be the same as that described in the *Portland Harbor Remedial Investigation/Feasibility Study Round 2 Field Sampling Plan: Sediment Sampling and Benthic Toxicity Testing* (Integral et al. 2004a). During field operations, the field staff will report to the field coordinators (Ian Stupakoff and Thai Do). Staff responsible for processing the tissue samples will report to the field quality assurance (QA) manager (Thai Do). The chemical laboratories will report to the chemistry QA manager (Maja Tritt). The field coordinators (Ian Stupakoff and Thai Do), laboratory oversight manager (Maja Tritt), and lead database manager (Tom Schulz) will report to the sampling and analysis

coordinator (Gene Revelas). Issues requiring the attention of the US Environmental Protection Agency (EPA) will be discussed with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) project coordinator (Keith Pine) and ecological risk assessment (ERA) project manager (Lisa Saban). To the extent possible, official communications between EPA and LWG will occur through their respective project managers.

A protocol modification form (Appendix A) will be completed for any significant change to the FSP or quality assurance project plan (QAPP); EPA approval will be required for all such changes. Any field staff or manager may request changes. The protocol modification form should be submitted to the ERA project manager, field QA manager, or sampling and analysis coordinator. If the changes are approved, the CERCLA project coordinator and LWG will be notified and will submit the forms to the EPA project manager for approval. If circumstances require immediate action, verbal authorization can be obtained and the change implemented, but a protocol modification form must still be completed and submitted as soon as possible to document the change and ensure that all managers are informed.

The field coordinator or sampling and analysis coordinator will notify the EPA project manager at least 1 week prior to beginning field activities so that EPA can schedule any necessary oversight tasks. EPA's project manager will contact the field coordinator and sampling and analysis coordinator to coordinate these activities and determine appropriate logistics. The sampling and analysis coordinator will notify EPA, in writing, when field activities are completed.

2.2 NAVIGATION AND STATION COORDINATES

A computer-integrated navigation system, using a Trimble ProXRS global positioning system (GPS) or equivalent unit, will be used to record the position, time, and date of each sample collected. As a back-up, station locations will also be recorded in a water-resistant field logbook. Handwritten field logbooks will be collected periodically from the field crew to accompany the GPS data. Beginning and ending coordinates for each set line used for sturgeon sampling will also be recorded and mapped. These data will be reviewed immediately after daily downloads to communicate and correct any data entry errors with the field crew.

2.3 SAMPLE EQUIPMENT DECONTAMINATION

All equipment used in the sampling effort will be decontaminated according to the standard operating procedure for tissue sample handling and processing, which was provided in Appendix A of the *Portland Harbor Remedial Investigation/Feasibility Study Field Sampling Plan: Subyearling Chinook Tissue Collection* (Integral et al. 2005a).

2.4 FIELD DATA MANAGEMENT AND SAMPLE IDENTIFICATION

The following sections describe in detail how information acquired in the field will be recorded and how samples will be identified.

2.4.1 Field Logs

All field activities and observations will be noted in a waterproof field logbook. Information will include personnel, date, time, station designation, sampler, samples collected, and general observations. Any changes that occur at the site (e.g., personnel, responsibilities, deviations from the FSP) and the reasons for these changes will be documented in the field logbook.

Logbook entries will be clearly written with enough detail so that participants can reconstruct events later, if necessary. Requirements for logbook entries will include the following:

- Logbooks will be bound, with consecutively numbered pages.
- Removal of any pages, even if illegible, will be prohibited.
- Entries will be made legibly with black (or dark) waterproof ink.
- Unbiased, accurate language will be used.
- Entries will be made while activities are in progress or as soon afterward as possible (the date and time that the notation is made should be noted, as well as the time of the observation itself).
- Each consecutive day's first entry will be made on a new, blank page.
- The date and time, based on a 24-hour clock (e.g., 0900 for 9 a.m. and 2100 for 9 p.m.), will appear on each page.
- When field activity is complete, the logbook will be entered into the Portland Harbor project file.

In addition to the preceding requirements, the person recording the information must initial and date each page of the field logbook. If more than one individual makes entries on the same page, each recorder must initial and date each entry. The bottom of the page must be signed and dated by the individual who makes the last entry. The field team or task leader, after reading the day’s entries, must also sign and date the last page of each daily entry in the field logbook.

Logbook corrections will be made by drawing a single line through the original entry, allowing the original entry to be legible. The corrected entry will be written alongside the original. Corrections will be initialed and dated and may require a footnote for explanation.

The type of information that may be included in the field logbook and/or field data forms includes the following:

- Names of all field staff
- Sampling vessel
- A record of site health and safety meetings, updates, and related monitoring
- Station name and location
- Date and collection time of each sample
- Observations made during sample collection, including weather conditions, complications, and other details associated with the sampling effort
- General description of the retrieved samples
- Sturgeon health assessment measurements and notes
- Any deviation from the FSP

As soon after collection as possible, field notes and data sheets will be scanned to create an electronic record for use in creating the field sampling report. Field data will be hand-entered into the database. One hundred percent of the transferred data will be verified based on hard-copy records. Electronic QA checks to identify anomalous values will also be conducted following entry.

2.4.2 Station and Sample Identification

A unique code will be assigned to each station and sample as part of the data record. The first component, LW3, will identify the data as belonging to the Lower Willamette River Remedial Investigation/Feasibility Study (RI/FS), Round 3. The second component will contain a two-letter abbreviation for the tissue sample type followed by the station (reach) number (001 through 005). The overall sample type is either "sturgeon tissue," abbreviated as ST, or "sturgeon gut content," abbreviated as SG. The third component will be a two-digit number (01-15) to designate the individual caught at the specific station. An example of a station and individual sturgeon tissue sample identification number is LW3-ST001-01. The gut content sample identification number for the same sturgeon would be LW3-SG001-01.

2.5 SCHEDULE

Pre-breeding white sturgeon tissue sampling will be conducted for up to 3 weeks beginning February 19, 2007. Sturgeon are known to be attracted to fishing bait (e.g., squid) throughout the year, but they feed less in the winter when the water is colder because their metabolic rate is relatively low. It may be difficult to collect the necessary fish from each of the five segments, so the first 2 to 3 days of the sampling effort will be used to identify areas within the Study Area in which to focus

subsequent sampling.[1] In the event that the proposed number of fish cannot be captured in a given segment, the field crew may take advantage of fishing opportunities elsewhere in the Study Area to ensure that a minimum of 15 sturgeon are captured upon consultation with EPA, assuming that the data quality objectives are still met. If an insufficient number of sturgeon are collected within the 3-week time period, EPA will be consulted to discuss whether additional collection is warranted and, if so, whether it should be continued as part of the current collection effort, or alternatively, conducted at a different time of year (e.g., during the summer).

A field sampling report will be submitted to EPA within 60 days of completing the field sample collection effort described in this FSP. LWG-validated analytical laboratory tissue data will be provided to EPA in an electronic format and SedQual format within 150 days of the completion of the sampling event.

2.6 HEALTH AND SAFETY

In preparation for the Round 1 sampling program, a health and safety plan (HASP) was submitted to EPA on June 14, 2002 (SEA 2002). This HASP was revised for Round 2 activities to ensure the health and safety of field and laboratory personnel and submitted to EPA on June 22, 2004 (Integral 2004). An addendum to the Round 2 HASP was provided in Appendix B of the *Portland Harbor Remedial Investigation/Feasibility Study Field Sampling Plan: Subyearling Chinook Tissue Collection* (Integral et al. 2005b). Additional specific health and safety information regarding this sampling event is also provided in the *Portland Harbor Remedial Investigation/Feasibility Study Health and Safety Plan*: *Round 3 Sampling for Pre-Breeding White Sturgeon* (Acipenser transmontanus) *Tissue* (Windward 2007).

[1] A commercial sturgeon fishing guide will be contracted for the initial 2 to 3 days of sampling to help determine the most suitable sampling locations.

3.0 SAMPLING COLLECTION AND PROCESSING

This section describes the sampling effort for pre-breeding white sturgeon tissue in the Study Area. This section also briefly describes how the tissue samples will be processed and stored in the field laboratory before being shipped to the chemistry laboratories. All deviations from the sample collection and processing activities will be recorded in the logbook and on a protocol modification form (Appendix A).

3.1 FIELD SAMPLING

Set lines will be used to collect white sturgeon for tissue analysis.[2] Each set line will be 80 meters in length and consist of a 0.64-cm nylon main line with 16 circle halibut hooks that will be baited with pickled squid and attached at 4.6-m intervals. Hook lines will consist of a 0.64-cm swivel snap and a 0.7-m-long gangion line tied between the swivel and the hook. The hooks used will be size 7 (12/0) circle halibut hooks. The use of large hooks will reduce the likelihood of other fish species being captured. The barbs on all hooks will be removed to facilitate release of fish not in the appropriate size range. Set lines will be deployed from a boat and generally set perpendicular to the shore. A minimum of three or four set lines will be placed daily, but the actual number will be determined in the field, depending on how many can be retrieved and processed each day. An anchor and float line will be attached to each end of the main line, and the set lines will be set overnight.

Wydoski and Whitney (2003) report that in some locations, male sturgeon mature at 9 years of age and that females mature at 13 to 16 years. The median age at sexual maturity for white sturgeon in the lower Columbia River was 24 years when they were 63.0 inches fork length, and 95% became mature when they were between 16 and 35 years of age at 48.8 and 77.2 inches fork length, respectively (DeVore et al. 1995). To ensure that only pre-breeding white sturgeon are analyzed, only those measuring within the legal size range (42 to 60 inches) will be retained. However, in the event that insufficient numbers of legal-sized fish are available, it may be necessary to retain smaller fish. EPA will be consulted for specific guidance on the acceptable sturgeon collection size and modifications to the sampling approach necessary to collect the 15 individual sturgeon.

Upon retrieval, all sturgeon will be brought on board and measured. Sturgeon within the accepted (i.e., target) size range will be placed in a clean cooler with aerated site water for live transport to the field laboratory for processing. Sturgeon that are not within the target size range will be released immediately with as little handling as possible. A total of 3 sturgeon from each study reach (or a total of 15 from the Study Area) will be collected for tissue analysis. Each retained sturgeon will be recorded on the field collection form provided in Appendix B.

[2] If use of set lines proves to be unproductive, the sampling method may be changed to rod and reel fishing using a commercial sturgeon fishing guide, upon consultation with EPA.

3.2 FIELD LABORATORY PROCEDURES

White sturgeon samples will be processed and stored at 4°C in the field laboratory and delivered to Columbia Analytical Services, Inc. (CAS), located in Kelso, Washington, within 24 hours. If shipping takes longer than 24 hours, the tissue samples will be frozen at -20°C at the field laboratory.

3.2.1 Fish Processing

In the field laboratory, each sturgeon will be processed using standard clean procedures (EPA 2000). Care will be taken during the processing to avoid contaminating the tissue samples; powder-free nitrile gloves will be worn whenever handling the sturgeon. Blood plasma samples will be collected in the field laboratory before sturgeon are euthanized.[3] Each sturgeon will be euthanized by pithing the fish through the notochord at the base of the head with a decontaminated steel blade or chisel with the aid of a 2-lb metal mallet, and then placed flat on a measuring board covered with clean aluminum foil to be measured for total length and fork length. The total length will be measured from the front of the jaw, which is most anterior, to the end of the longest caudal ray when the rays are squeezed together, but excluding the caudal filaments to the end of the tail. Fork length will be measured from the tip of the snout to the posterior end of the middle caudal rays (FishBase 2005). Both measurements will be recorded in the logbook and on the sample processing form (Appendix C). After length measurements are recorded, the fish will be weighed, and the weight will be recorded.

The age of the sturgeon may possibly be determined by counting annuli in transverse sections cut from the pectoral fin ray. Assessing the age of sturgeon has been problematic because recaptured PIT-tagged fish that have been at-large for several years have shown that individual growth is extremely variable and annuli are not laid down each year (Parsley 2007). Other factors that may increase variability in age readings include the presence of split annuli, false annuli, spawning bands, imbedded rays, and deteriorating sections (Whiteman et al. 2004). However, in terms of ease of collection, processing, legibility, and precision of interpretation, counting annuli is currently the best available technique to determine sturgeon age (Brennan and Cailliet 1989). A 1-cm segment near the joint from the leading spine of the pectoral fin ray will be collected from the left side of each fish, unless damaged, and placed in separate labeled envelopes. After all sturgeon are processed, the pectoral fin ray samples will be delivered to Oregon Department of Fish and Wildlife for age analysis.

In addition to the length and weight measurements and age determinations, an external fish health assessment will be conducted by or under the supervision and

[3]A representative for the Natural Resource Trustrees will collect and take custody of blood plasma samples to provide information about possible endocrine disruption in fish; US Fish and Wildlife Service personnel will provide a set of standard operating procedures to facilitate the effort.

guidance of US Fish and Wildlife Service personnel or other experienced fisheries biologist.[4] The primary purpose of the external health assessment is to record deformities or abnormalities (e.g., missing, clubbed, or curled fins; head or spinal deformities) that may be associated with site contaminants. A photograph will be taken of each external abnormality. At this time, any external abnormality may be excised and preserved in formalin for histological examination upon EPA's request.

After the external health assessment is conducted, each sturgeon will be dissected with a decontaminated ceramic knife to collect a sub-sample of liver tissue and to retrieve the gastrointestinal (GI) tract and remove the stomach contents. Before collecting a liver tissue sub-sample, the liver will be examined for anomalies, and observations will be recorded on the sample processing form.[5] The stomach contents will be placed onto a decontaminated stainless steel tray or polytetrafluoroethylene (PTFE) cutting board, and the GI tract will be placed back into the body cavity. General observations of the stomach contents will be recorded, including visual identification of prey items (i.e., prey species and percent sediment); and any bait (i.e., pickled squid) remains will be removed. Prey species will be identified to the lowest taxonomy possible at the field laboratory, and the mass of each prey species will be estimated. After identification, the stomach contents will be stored in a chemically clean 8-oz jar, and submitted for metals, PAH, and percent moisture analysis. At any time during processing, the gonads may be examined to identify the sex of the sturgeon.

Upon completion of the processing of each fish, each whole-body fish sample will be wrapped in aluminum foil, enclosed in a 2-mil US Food and Drug Administration (FDA)-approved food-grade polyethylene plastic bag with an identification label, and stored at -20°C until delivered to CAS for homogenization and analysis. The jars containing the stomach contents will also be stored at -20°C at the field laboratory until delivery to CAS for homogenization and analysis.

3.2.2 Equipment Decontamination

Equipment used to process sturgeon will be decontaminated before use. Tables and trays will be covered with clean, heavy-duty aluminum foil. Between stations, utensils will be decontaminated, new weigh pans will be used, and the aluminum foil on tables and trays will be replaced. The decontamination process of the utensils will be done in the following manner:

1. Rinse with potable water.
2. Scrub with brush and Alconox™ (or other phosphate-free detergent) solution.

[4] US Fish and Wildlife Service personnel will provide a set of standard operating procedures to facilitate the health assessment effort.
[5] Custody of the liver tissue sub-samples will be relinquished to a representative for the Natural Resource Trustees.

3. Rinse twice with deionized water.
4. Rinse with 0.1 N nitric acid.
5. Rinse with methanol.
6. Rinse with deionized water.

3.2.3 Waste Disposal

All disposable materials used in sample processing, such as paper towels and gloves, will be placed in heavy-weight (3-mil thickness or greater) garbage bags or other appropriate containers. Disposable supplies will be removed from the site by sampling personnel and placed in a normal refuse container for disposal at a solid waste landfill. Phosphate-free, detergent-bearing liquid wastes from the decontamination of the sampling equipment will be washed overboard or disposed of into the sanitary sewer system. Discard contaminated sharps (e.g., needles and blades) immediately after use, or as soon as feasible, into appropriate containers. Appropriate containers must be:

- Closable, puncture-resistant, and leak-proof on sides and bottom
- Accessible, maintained upright, and not capable of being overfilled
- Colored red or labeled with the biohazard symbol
- Labeled in fluorescent orange or orange-red, with lettering and symbols in a contrasting color.

3.2.4 Chain-of-Custody Procedures

Samples are in custody if they are in the custodian's view, stored in a secure place with restricted access, or placed in a container secured with custody seals. Each person who has custody of the samples will sign a chain-of-custody record, which will accompany the samples at all times. Chain-of-custody forms will be completed in triplicate; the person responsible for sample collection will retain a copy, and the other two copies will accompany the shipment to the laboratory. Copies of the chain-of-custody forms will be included in laboratory reports. At minimum, the chain-of-custody form will include the following information:

- Site name
- Field task leader's name and team members responsible for collection of the listed samples
- Collection date and time
- Sample type (e.g., sturgeon tissue)
- Sampling station location

- Number of sample containers shipped
- Requested analyses
- Sample preservation information
- Name of the field coordinator or his designee relinquishing the samples to the transporter, noting date and time of transfer and the designated sample custodian at the receiving facility
- Signature, date, time of receipt by custodian at receiving facility

The field coordinator or a designated field sample custodian will be responsible for all sample tracking and chain-of-custody procedures for samples in the field. The sample custodian will be responsible for final sample inventory and will maintain sample custody documentation. The custodian will complete chain-of-custody forms prior to removing samples from the sampling vessel. Upon transferring samples to the laboratory sample custodian, the field sample custodian will sign, date, and note the time of transfer on the chain-of-custody form.

The original chain-of-custody form will be transported with the samples to the laboratory. The laboratory sample custodian will be responsible for receiving samples and logging them into the laboratory's tracking system. The custodian will ensure that the chain-of-custody and sample tracking forms are properly completed, signed, and initialed upon transfer of the samples.

The custodian will enter the sample number into a laboratory tracking system by project code and sample designation. The custodian will assign a unique laboratory number to each sample and will be responsible for distributing the samples to the appropriate analyst or for storing the samples in an appropriate, secure area. Specific laboratory chain-of-custody procedures are described in the laboratory QA plan (Integral and Windward 2004a).

3.2.5 Sample Shipping and Delivery

White sturgeon samples will be stored at 4°C at the field laboratory prior to delivery to the laboratory within 24 hours. If samples are held at the field lab for more than 24 hours, the tissue samples will be frozen. Upon completion of final inventory by the field sample custodian, individual samples will be wrapped in foil and placed into a sealed polyethylene plastic bag. Samples will then be packed in a cooler lined with a large plastic bag. Ice in sealed plastic bags or "blue ice" will then be placed in the cooler to maintain a temperature of approximately 4°C during sample transport. The laboratory will immediately notify the chemistry QA manager upon receipt of any non-conforming samples (cooler receipt temperatures > 6°C). Upon notification, the chemistry QA manager will notify the field QA manager and the sampling and analysis coordinator, and packing procedures will be adjusted as needed.

When all samples have been packed, the chain-of-custody form will be placed into a zip-lock bag and taped on the inside lid of the cooler. A temperature blank will be added to each cooler. Each ice chest will be sealed with three chain-of-custody seals.

The coolers will be clearly labeled with sufficient information (i.e., name of project, time and date container was sealed, person sealing the cooler, and company name and address) to enable positive identification. Placards that state the following will be taped to the cooler.

- In case of delay or emergency, please contact Maja Tritt at 206-957-0353, ext. 21, or Ian Stupakoff at 360-705-3534.
- Fragile, Handle with Care

Coolers will be transported to CAS by laboratory courier. These packaging and shipping procedures are in accordance with US Department of Transportation regulations as specified in 49 CFR 173.6 and 49 CFR 173.24.

3.3 QUALITY CONTROL PROCEDURES

3.3.1 Field Quality Control Samples

Field quality control (QC) samples are used to assess the within-station variability (e.g., replicates), evaluate the effectiveness of sample homogenization and within-sample variability (e.g., splits), or confirm proper storage conditions (e.g., temperature blanks). The types of QC samples that will be collected in this study are described in this section.

Collection of additional field replicates is not feasible. One field duplicate will be generated at the laboratory when the fish are homogenized, by dividing the homogenate from one of the fish into two sample containers. The field duplicate will be analyzed as a separate sample. Sufficient sample mass will be available to complete laboratory QC (i.e., matrix spikes, matrix spike duplicates, laboratory duplicates) at the required frequency for all of the analyses.

The pickled squid that will be used for bait will be purchased in one large batch. A sub-sample will be collected from the bait and shipped to the chemistry laboratories for analysis of the chemicals listed in Section 4.2. The remainder of the bait will be stored frozen, if necessary, until used in the field sampling effort.

Temperature blanks will be included in each cooler to confirm proper cooler temperature upon receipt at the laboratory.

3.3.2 Corrective Actions

If corrective actions require a departure from the FSP, these changes will be documented on a protocol modification form (see Appendix A). In any other

circumstances where sampling conditions are unexpected, the appropriate sampling actions consistent with project objectives will be conducted after the field QA manager informs the sampling and analysis coordinator. This change will be noted in the field logbook, and a protocol modification form will be completed for the project files.

4.0 ANALYTICAL LABORATORY METHODS

This section summarizes options for chemical analyses planned for the tissue samples. White sturgeon tissue samples will be analyzed by CAS and by Axys Analytical Services, Ltd. (Axys), located in Sidney, BC, Canada. Alternative labs may be selected if schedule demands cannot be met by either of these labs.

4.1 ANALYTICAL LABORATORY SAMPLE PROCESSING

CAS will homogenize the tissue samples and perform the analyses for PAHs, SVOCs, phthalates, metals, butyltins, and moisture. Axys will perform the analyses for PCB congeners, dioxins and furans, pesticides, and lipids. Sturgeon tissue and stomach content samples will be stored at 4°C in the field laboratory and delivered to CAS within 24 hours. If delivery takes longer than 24 hours, the samples will be frozen in the field lab. The samples will be frozen (-20°C) upon receipt at CAS and will be stored frozen except during sample processing. CAS will prepare the tissue sample from each sturgeon by homogenizing the whole body; because homogenizing sturgeon tissue may be challenging because of the size of the fish, care will be taken to ensure that a representative homogenized whole-body sample is obtained. CAS will also prepare subsamples for analysis at Axys. All sample processing will occur under clean room conditions. An equipment blank will be prepared by rinsing the sample homogenization equipment with deionized water and collecting the rinse water in sample jars.

Once the homogenized samples have been prepared, the subsamples for chemical analysis will be placed in glass jars and stored frozen (below -20°C) until chemical analysis (EPA 2001). Some of the excess homogenized tissue will be archived for potential future analysis, and the remainder will be discarded. All samples will be maintained according to the appropriate holding times and temperatures for each analysis, as summarized in Table 4-1.

Table 4-1. Tissue sample container, preservation, holding time, and volume requirements

Analysis	Container Type	Container Size	Tissue Weight[a]	Preservation	Holding Time
Lipids and pesticides	WMG[b]	8 oz (one)	10g	deep frozen (-20 ±4ºC)	1 year
Butyltin compounds			10g		1 year
PCB congeners; PCDDs/PCDFs			10g		1 year
PAHs	WMG[b]	8 oz (one)	10g		1 year
Phthalate esters and SVOCs			10g		1 year
Mercury			5g		6 months[c]
Metals and percent moisture			5g		1 year
Archive	WMG[b]	16 oz (two)	500 g		1 year

[a] Matrix spike samples and duplicates will be prepared by the laboratory.
[b] Wide-mouth glass jar with Teflon®-lined lid.

[c] The holding time for mercury in frozen (i.e., archived) samples is 180 days, as approved by EPA (Humphrey 2002).

PAH – polycyclic aromatic hydrocarbon
PCB – polychlorinated biphenyl
PCDD – polychlorinated dibenzo-*p*-dioxin
PCDF – polychlorinated dibenzofuran
SVOC – semivolatile organic compound
WMG – wide-mouth glass

4.2 CHEMICAL ANALYSIS

White sturgeon tissue samples will be analyzed for the following constituents/ conventionals:

- Polychlorinated biphenyl (PCB) congeners (209)
- Dioxins and furans
- Organochlorine pesticides
- Polycyclic aromatic hydrocarbons (PAHs)
- Butyltin compounds
- Phthalates and semivolatile organic compounds (SVOCs)
- Metals
- Lipid content
- Percent moisture

Stomach contents will be analyzed for the following constituents:

- PAHs
- Metals
- Percent moisture

Axys will complete analysis of PCB congeners, dioxins and furans, pesticides, and lipids and CAS will complete the remaining analyses. Laboratory procedures will be completed as described in Integral and Windward (2004b; 2005). Analyte lists, detection limits, and laboratory QC specifications are provided in Integral and Windward (2005). All tissue data will be reported on a wet-weight basis.

Laboratory methods to be used for Round 3 are consistent with requirements provided in EPA methods and other widely accepted protocols. Modifications will be made to these methods, as necessary and technically feasible, to improve analytical sensitivity. Modifications are described in the Round 2 QAPP and QAPP Addendum 6 (Integral and Windward 2004b; 2005).

Axys completed analyses for pesticides, PCB congeners, and PCDDs/PCDFs using a single sample extract for the benthic tissue study (Integral and Windward 2005) and will use this approach for the lamprey ammocoete study (Windward 2006) as well. The extract was subsequently fractionated to separate the various analyte groups. This

approach was selected to utilize the limited tissue mass that was available for those samples to the fullest possible extent. However, for the sturgeon study described in this document, Axys may use the standard approach of performing separate extractions for pesticides, PCBs, and PCDDs/PCDFs because sufficient tissue mass will be available.

5.0 REPORTING

A white sturgeon tissue field sampling report will be prepared by Windward and submitted to EPA within 60 days of completing the field sample collection effort described in this FSP. The field sampling report will summarize field sampling activities, sampling locations, and field laboratory data, including the number of sturgeon collected at each location, weight measurements, and any deviations from the FSP.

LWG-validated analytical laboratory tissue data will be provided to EPA in an electronic format and in SedQual format within 150 days of completion of the sampling and analysis. Tissue chemistry results will be reported in tabular format in the data summary report. These data will also be incorporated into the RI report and baseline risk assessment, which will be prepared after all sampling and analysis rounds for the project are completed.

A tissue data summary report for sturgeon will be developed within 90 days after tissue chemistry sampling and analyses. This report will be prepared by Windward and will include tissue results and maps summarizing the chemistry results for selected analytes.

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APPENDIX A: PROTOCOL MODIFICATION FORM

Protocol Modification Form

Project Name and Number: _____
Material to be Sampled: _____
Measurement Parameter: _____

Standard Procedure for Field Collection & Laboratory Analysis (cite reference): _____

Reason for Change in Field Procedure or Analysis Variation: _____

Variation from Field or Analytical Procedure: _____

Special Equipment, Materials or Personnel Required: _____

Initiator's Name: _____ Date: _____
Project Manager: _____ Date: _____
QA Manager: _____ Date: _____

APPENDIX B: FIELD COLLECTION FORM

Field Collection Form

Page _____of ______

Project Name: ________________________________ **Project No.** ____________________________

Date: ________________________________ **Crew:** ____________________________

Sample Location	Sample ID	Total Length (cm)	Fork Length (cm)	Comments

APPENDIX C: SAMPLE PROCESSING FORM

Sample Processing Form

Sample ID:
Species:
Collection Date:
Processing Date:
Processing Time:
Processed by:

Whole-body weight (g/lb):
Total length (cm/in):
Fork length (cm/in):
Pectoral fin ray collected? Y / N L / R
Blood sample ID:
Blood sample volume (ml):

EXTERNAL EXAMINATION

Eyes: left		**right**	
☐ normal	☐ other (*specify*):	☐ normal	☐ other (*specify*):
☐ exopthalmic		☐ exopthalmic	
☐ hemorrhagic		☐ hemorrhagic	
☐ opaque		☐ opaque	
☐ missing		☐ missing	
☐ emboli		☐ emboli	
No. in fixative _____	No. of photos _____	No. in fixative _____	No. of photos _____

Head:	**Body surface:**	**Opercles**:
☐ normal	☐ normal	☐ normal
☐ tumors	☐ tumors	☐ slight shortening
☐ lesions	☐ lesions	☐ severe shortening
☐ parasite	☐ parasite	☐ other (*specify and include location*):
☐ other	☐ other	No. in fixative _____ No. of photos _____

Gills: left		**right**	
☐ normal	☐ other (*specify*):	☐ normal	☐ other (*specify*):
☐ frayed		☐ frayed	
☐ clubbed		☐ clubbed	
☐ marginate		☐ marginate	
☐ pale		☐ pale	
No. in fixative _____	No. of photos _____	No. in fixative _____	No. of photos _____

Fins:	
☐ normal	☐ hemorrhagic
☐ mild erosion	☐ emboli
☐ severe erosion	☐ other (*specify*):
☐ frayed	
No. in fixative _____	No. of photos _____

INTERNAL EXAMINATION

Liver observations:	
Liver sub-sample ID:	**Liver sub-sample weight (g):**
Color ☐ dark- to light-red ☐ tan (coffee with cream) ☐ general discoloration (*specify*): ☐ focal discoloration (*specify*): ☐ nodules ☐ other (*specify*): No. in fixative _____ No. of photos _____	

Stomach contents:	
Stomach contents sample ID:	**Stomach content weight (g):**
Taxonomy: **Percent sediment & weight (g)**:	

ADDITIONAL NOTES
