Appendix D

FWP/DEQ Fish Tissue Quality Assurance Project Plan for Lake Koocanusa

Date: 4/30/2018
Number: WQSMQAP-02
Approvals:

Trevor Selch, Project Manager, Toxicologist, FWP — Date 5/1/18

Terri Mavencamp, Project Manager, WQPB, SeTSC Co-Chair DEQ — Date 4/30/18

Myla Kelly, Water Quality Standards Section Supervisor, WQPB, DEQ — Date 5/7/18

Eric Urban, WQPB Bureau Chief, MRWG SC, DEQ — Date 5/1/18

Michelle Hauer, QA/QC Officer, WQPB, DEQ — Date 5/2/18

Michael Sokal, BCMOE SeTSC Co-Chair — Date 4/30/2018

Michael Pipp, Information Management and Technical Services, IMTS, DEQ — Date 5/1/2018
IMTS or designee

Contents

REVISION TABLE

Revision No.	Date	Modified By	Sections Modified	Description of Changes

1.0 PROJECT MANAGEMENT

This Quality Assurance Project Plan (QAPP) describes the planning, field collection, quality assurance and control and the analysis of fish tissue collected in Lake Koocanusa to measure the selenium tissue concentration of fish in Lake Koocanusa and to inform a site-specific selenium criterion/objective for Lake Koocanusa. Fish tissue samples prepared under this QAPP will be analyzed for selenium and percent moisture, and will be temporally (w/in 30 days) and spatially (within approximately 10 miles) matched to dissolved selenium water column concentrations, benthic macroinvertebrate and surface macroinvertebrate selenium concentrations and possibly to concentrations of selenium in the suspended sediment (funding pending) (DEQ, 2018a and DEQ 2018b).

This QAPP was prepared in accordance with the EPA QA/R-5, *EPA Requirements for Quality Assurance Project Plans* (USEPA 2001). This QAPP is a dynamic document that is subject to change as activities progress. When/if changes occur in the planning/collection/analysis components, the QAPP will be revised accordingly and the revision noted in the revision table and circulated for approval, and forwarded to all project participants listed in the distribution list (Section 1.2).

1.1 PROJECT ORGANIZATION

Montana Fish Wildlife and Parks (FWP) and Department of Environmental Quality (DEQ) are working together to collect and analyze fish tissue on Lake Koocanusa. The general project organization is outlined in Figure 1 and shows the project managers, Selch and Mavencamp who are responsible for developing and updating the QAPP with input from the Selenium Technical Subcommittee (SeTSC) and Monitoring and Research Committee (MRC). Jim Dunnigan and Trevor Selch are the field leads and will report and deviations from the QAPP to Mavencamp and the DEQ quality assurance/quality control (QA/QC) officer, Michelle Hauer. Michelle Hauer, has the role of approving the QAPP and ensuring that it is adhered to as well as determining if QAPP modifications are necessary. DEQ Data management has the responsibility of entering the data into DEQ's Montana EQuIS Water Quality Exchange database (MT-eWQX) and of performing QC on data received from the labs (please see attachment C for the QC checklist).

FWP has the responsibility of

- Planning field activities with input from the MRC/SeTSC (project manager, Selch and field lead Dunnigan)
- Overseeing field activities, including field QC and addressing any deviations from the QAPP (project manager, Selch and field lead Dunnigan)
- Overseeing tissue preparation and QC from the field to the lab (Selch)
- Coordinating with DEQ to by using the appropriate forms and aiding in the creation of QAPP so that the data can be entered as a DEQ project into DEQ's (MT-eWQX) (FWP all parties indicated in Figure 1 will follow the data processes set out in this QAPP)
- Communication of results to the SeTSC (Selch)

The FWP field project lead and field team are responsible for collecting fish, delivering them to the lab, following the procedures described in this QAPP, and documenting their activities using the correct site visit forms (Attachment A).

DEQ has the responsibility of

- Coordinating and communicating the needs of the SeTSC with the field planning (Mavencamp
- Coordinating with FWP to ensure data are collected in a way that can be entered into MT-eWQX (site visit forms, site visit stickers, etc.)
- QAPP preparation in collaboration with FWP and MRC/SeTSC
- Coordinating with the laboratory (and setting up lab contracts) to ensure proper methods, method detection limits and processes are being followed.
- DEQ QA/QC Officer has the responsibility of reviewing and approving this QAPP.
- DEQ Data Management, Information Management and Technical Services (IMTS),Michael Pipp/Jolene McQuillan, is responsible for data validation of received data from the lab and entering the data into DEQ's MT-eWQX and subsequently sending the data to EPA's National WQX Warehouse where the data will be available for use by the public and other agencies through EPA's Water Quality Portal.

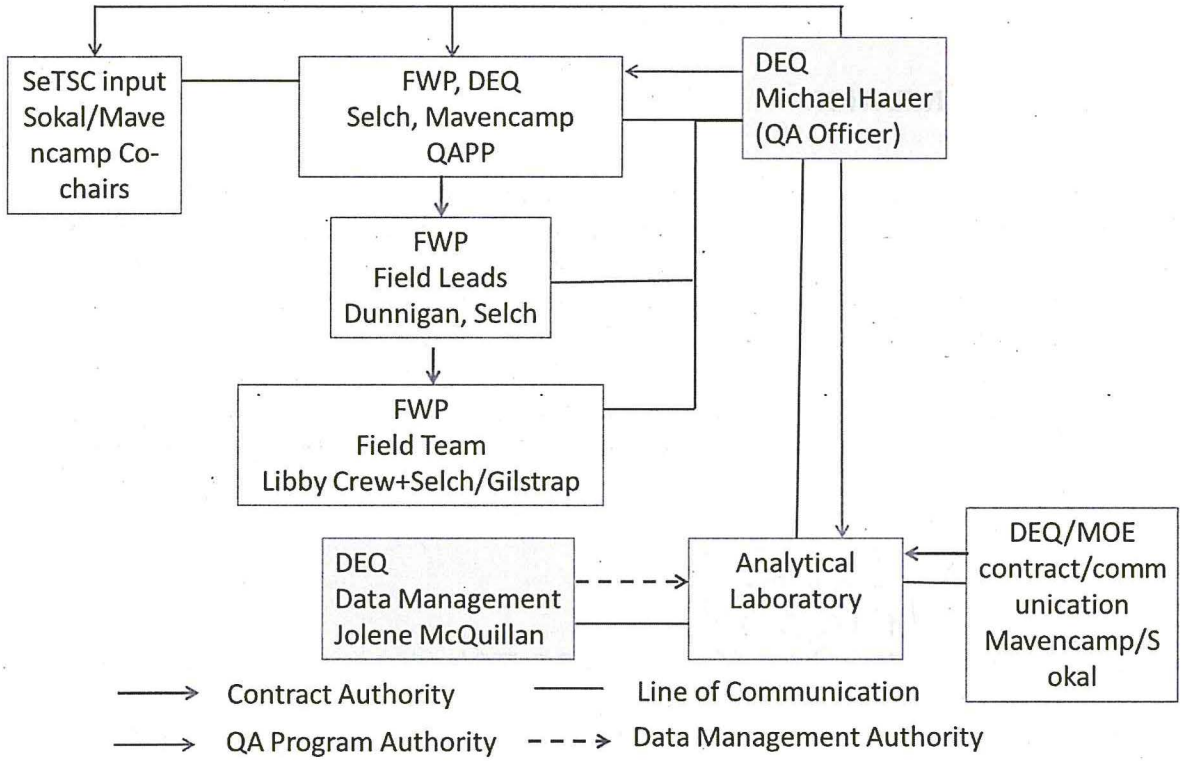

Figure 1. Project Organization Chart. Blue boxes indicate data quality/management and green indicates an independent analytical laboratory. Lines of communication, green, flow both ways.

1.2 Project Team and Responsibilities

Field Personnel:
James Dunnigan: Field Manager, FWP
Monty Benner, Conservation Technician, FWP
Thomas Ostrowski, Conservation Technician, FWP

Jared Lampton, Conservation Technician, FWP
Neil Benson, Conservation Technician, FWP

FWP fish processing in Helena/ FWP Project Manager: Trevor Selch

DEQ Project Manager, QAPP preparation, logistics and laboratory contracts: Terri Mavencamp

Fish processing assistance: Lindsay Gilstrap/Terri Mavencamp (FWP/DEQ)

QA/QC Michelle Hauer/Jolene McQuillan (DEQ)

Data handling and processing for WQX and STORET: Jolene McQuillan (DEQ)

1.3 Distribution List

All persons on the distribution list will receive electronic copies of the final QAPP and any subsequent revisions.

Trevor Selch, FWP, Helena, MT
Jim Dunnigan, FWP, Libby, MT
Terri Mavencamp, DEQ, Helena, MT
Myla Kelly, DEQ, Helena, MT
Darrin Kron, DEQ, Helena, MT,
Michelle Hauer, DEQ, Helena, MT
Michael Pipp, DEQ, Helena, MT
Jolene McQuillan, DEQ, Helena, MT
Eric Urban and Robyn Roome, DEQ and ENV, Steering Committee, Monitoring and Research Working Group (MRWG)
Selenium Technical Subcommittee
Monitoring and Research Committee

1.4 Problem Definition

Selenium is indirectly discharged into the international waterbody, Lake Koocanusa, from steelmaking coal mines owned by Teck Coal Limited (Teck Coal) in the Elk Valley, British Columbia (B.C.). Selenium concentrations in Lake Koocanusa range from approximately .6 µg/L to 1.5 µg/L, or higher in certain areas of B.C.

Teck Coal submitted the Elk Valley Water Quality Plan (approved in July, 2014) to address increasing selenium and nitrate water concentrations in B.C.- while maintaining concentrations within Lake Koocanusa at or below Provincial water quality guidelines, the Plan does not address U.S. waters of Lake Koocanusa. As a result, an unmatched dataset exists for parameters in Lake Koocanusa on the Canadian and U.S. sides.

The U.S. has pursued routine water column monitoring via multiple programs, including U.S. Geological Survey (USGS) water quality monitoring and more recently the U.S. Army Corps of Engineers (USACE). Since 2008, in a collaborative effort between FWP and DEQ, fish tissues have been collected and analyzed for a suite of metals, including selenium. FWP has fish monitoring stations at three locations throughout the reservoir, the Canadian portion near the Elk, Rexford, near the Tobacco River and Tenmile. In 2008, tissue analysis was performed on fish from the Canadian and Rexford stations; and in 2013, the effort included the analysis of fish from Tenmile (McGillivray) and Rexford in the spring and Rexford and Canada in the Fall. The study will be repeated in 2018, including the Tenmile site in the spring, Rexford in the spring and fall and the Canadian site in the fall.

1.5 Project/Task Description

DEQ/FWP are collaborating to collect and analyze fish tissue in Lake Koocanusa. Fish will be collected by the FWP fisheries division, and sent to Energy Laboratory in Helena, MT for analysis of selenium and percent moisture.
The following are key design components of fish collection in Lake Koocanusa:

3 Fish identified as being critical to the development of a site-specific selenium criterion/objective will be selected from the gill netting efforts conducted by FWP to assess population trends in Lake Koocanusa and analyzed for selenium in multiple tissues (filet, whole body, and/or egg/ovaries) as available.

4 Fish will be caught and analyzed for selenium from the Canadian site in the fall, Rexford in the spring and fall and Tenmile (McGillivray) in the spring.

1.6 Quality Objectives and Criteria

The overall quality objective for the analysis of fish tissue for selenium is to continue baseline monitoring of fish tissue selenium in Lake Koocanusa, evaluate concentration trends (compare to 2008 and 2013 as well as other data collected in Canada) and help inform a site-specific selenium criterion/objective for Lake Koocanusa. Fish tissue values can be used for Se model validation, derivation of conversion factors and trophic transfer factors and/or in bioaccumulation factor (BAF) model derivation. Please see Section 2.0 for technical details of the project design.

To have usable data to meet the above objectives, the following measurement quality objectives have been identified: the laboratory reporting limits (RLs) must be low enough to yield usable results. Based on past fish tissue concentrations we do not anticipate that RLs will be a concern, and are set at 0.5 µg/g (dry weight) for selenium. Accuracy and precision are important and must lie within the guidelines outlined in section 6 of this QAPP and in the analytical methods. Completeness is defined as the percentage of samples collected in the study for which usable analytical results are produced. The goal for completeness is 90% and is calculated at the sample-analyte level. For baseline monitoring and trends analysis, we would like to collect a minimum of five fish of each identified species and sampling event (location and time) (identified in Section 4).

To use these results to develop site-specific criterial we need temporal coordination (collected within the same year, for this project we are aiming to collect matched data within a 30-day time frame) and

spatial coordination (the reservoir has been functionally split into three sections, the lower, mid and upper sections or Forebay/McGillivary; Rexford/International Border; and Canada), spatially, we would like matched samples collected within a maximum of 10 miles of one another.

We propose to use the performance based measurement system (PBMS) concept, meaning that as long as the data quality objectives (performance or acceptance criteria) are defined, the data quality indicators (DQIs defined in section 6) are identified and the appropriate measurement quality objectives (MQOs) are met, the data should be appropriate to use for the project objectives.

1.7 Special Training/Certification

All FWP employees have been trained or mentored in the field sampling methods described in the QAPP. All FWP employees who use electrofishing, are trained and attend an electrofishing course. FWP fishing methods/trainings are covered under different documents, i.e., FWP processes, Bonneville Power Administration(BPA)Mitigation document (Dunnigan et al. 2014).

Staff relevant to this project are trained and experienced in proper sampling, field analysis and boat safety. Training for field procedures under this QAPP will be performed by FWP project leaders. Sampling personnel are experienced fisheries technicians knowledgeable in local fish species, proficient in field sampling techniques and familiar with this QAPP and Fish Tissue Standard Operating Procedure SOP (DEQ, 2015).

Laboratories analyzing samples under this QAPP are responsible for providing personnel qualified for the methods requested and adhering to their laboratory Quality Assurance Plan LQAP. Laboratories MT DEQ uses for analyzing samples are either certified through the State of Montana, accredited under national programs, or their quality system is known and meets DEQ's requirements (DEQ QMP, 2015).

2.0 Introduction and Background

Lake Koocanusa is a managed reservoir. Water levels in Lake Koocanusa are generally lowest in late winter/early spring (i.e., February through April) and highest in summer/early fall (Minnow Environ. Inc., 2014). Several biological sampling programs have occurred in Lake Koocanusa (Dalbey et al., 1998; Dunnigan et al., 2016; Montana FWP, 2013; Minnow, 2017).

This QAPP describes the fish sample collection by FWP in 2018/2019 in Lake Koocanusa, fish handling and lab analysis. The sampling is being done to support baseline monitoring and fish tissue concentration trends, specifically selenium, and support the development of a site-specific selenium criterion/objective for Lake Koocanusa.

This QAPP is a collaborator vie effort with FWP population studies on Lake Koocanusa that falls under the Bonneville Power Administration mitigation grant program that funds FWP population studies, including the fish catch efforts referred to in this QAPP https://www.cbfish.org/Project.mvc/ProjectDocuments/1995-004-00 .

In 2008 and 2013, Bull Trout, Longnose Sucker, Northern Pikeminnow, Kokanee, Peamouth, Rainbow Trout, and Westslope Cutthroat Trout, were targeted for tissue and opportunistic egg/ovary sampling. Egg/ovary samples were taken if ovaries were with eggs, but the stage of development was not noted. Sites sampled were the Canadian portion of the reservoir around the mouth of the Elk River, Rexford around the confluence of the Tobacco River and Tenmile or the lower 1/3 of the reservoir (also referred to as McGillivray).

Figure 2 shows results of 2013 tissue collection across the aforementioned sampling sites: Canada, Rexford and McGillivray (Tenmile).

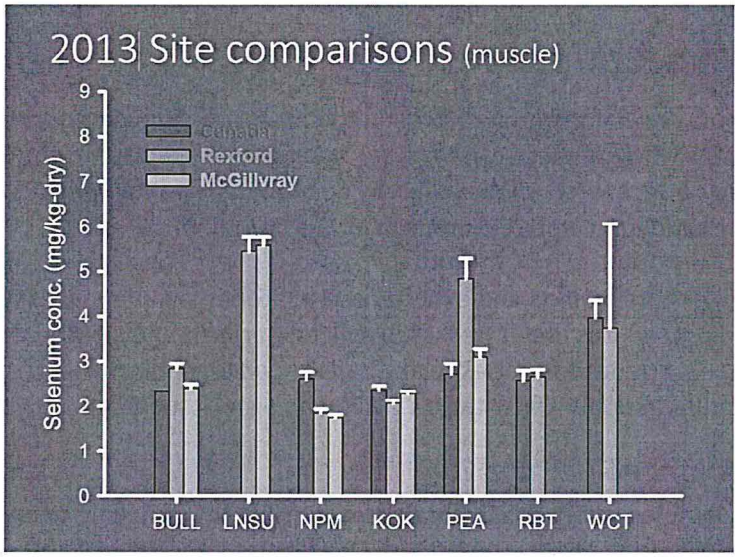

Figure: 2 Muscle selenium concentrations from the 2013 FWP/DEQ fish tissue monitoring effort. Figure from Selch presentation, Kalispell, 2017.

Selenium muscle tissue, whole body and egg values will be compared to current BC Ministry of Environment (MOE) and the EPA recommended selenium fish tissue criterion (EPA 2016 Se Criteria).

Aquatic Life Selenium Guidelines / Criterion			
Agency	Fish Tissue (Whole Body)	Fish Tissue (Egg-ovary)	Fish Tissue (muscle)
EPA suggested [1]	8.5 mg/kg dw	15.1 mg/kg dw	11.3 mg/kg dw
MT current	None	None	none
MOE (current guidelines)[2]	4 mg/kg dw	11 mg/kg dw	4 mg/kg dw

Table 1: Aquatic Life Selenium Guideline/Criterion

[1] EPA Fact Sheet, Aquatic Life Criterion https://www.epa.gov/sites/production/files/2016-06/documents/se_2016_fact_sheet_final.pdf

[2] BC MOE Companion Document to Ambient Water Quality Guidelines for Selenium Update, Water Protection and Sustainability Branch, Environmental Sustainability and Strategic Policy Division, 2014.

Lake Koocanusa is in the Middle Kootenai 4th level HUC 17010101 and has been assigned a B-1 beneficial use classification (ARM 17.30.609). A B-1 beneficial use classification is applied to waters that are suitable for domestic use after conventional treatment, growth and propagation of salmonid fishes, associated aquatic life and wildlife, and agricultural and industrial uses. Lake Koocanusa is considered threatened by MT DEQ because of rising selenium levels from sources outside state jurisdiction or borders and is impaired for aquatic life by other flow regime alterations due to impacts from Libby Dam (Montana 2016 list of impaired waters http://deq.mt.gov/Portals/112/Water/wqpb/CWAIC/Reports/IRs/2016/App_A.pdf).

3.0 Objectives and Sampling Design

3.1 Objective

The goal of this project is to continue the baseline monitoring of fish tissue selenium in Lake Koocanusa, evaluate concentration trends and use the data to inform the development of a site-specific selenium criterion/objective.

The objective of this sampling project is to collect fish at three locations in the reservoir to examine the species, spatial and temporal variations in selenium fish tissue concentrations. The objectives will be met by coordinating with FWP's fish population monitoring effort of Lake Koocanusa outlined in (BPA) project reports for the 'Mitigation For the Construction and Operation of Libby Dam' found here https://www.cbfish.org/Project.mvc/ProjectDocuments/1995-004-00 .

3.2 Selection of Sites

FWP has split the reservoir into three study areas. For 2018/2019 field fish sampling, there will be three main sites where FWP will set gillnets. The sites around Tenmile are considered the lower 1/3 of the

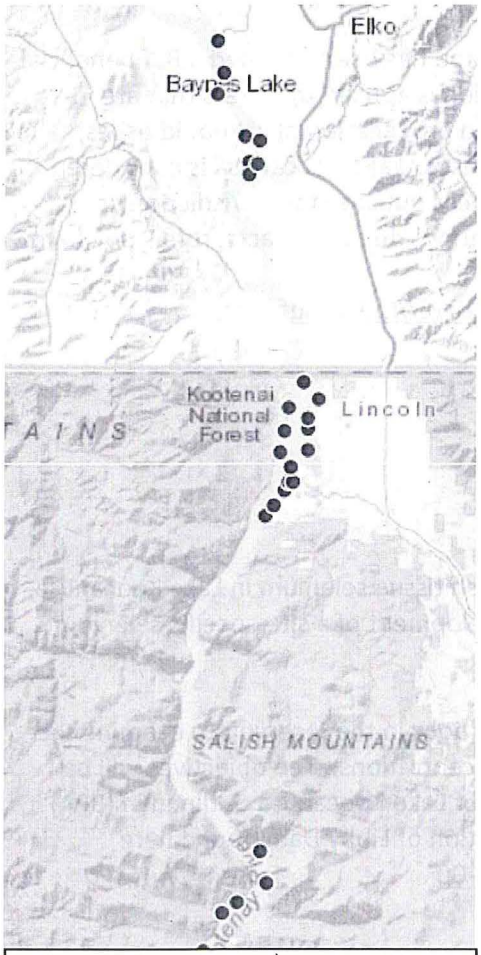

Figure 3: Map of FWP Gillnet Sites for Long term species/trend information. The exact location of each net will be recorded on the SVF.

reservoir, the middle 1/3 is considered the sites around Rexford and the upper 1/3 of the reservoir is considered "Canada" where nets are set near the Elk River confluence. The areas are affected differently by dam operation and reservoir morphology (Chisholm, 1987). These geographic areas were chosen based on reservoir morphometry, effects of reservoir drawdown and political boundaries (Chisholm, 1987).

Sites for 2018/2019 are the same as previous years for the Canada and Rexford sites. For the Tenmile site, net locations have been spread out from the Tenmile site to the Forebay to give a more complete spatial representation of this area. Figure 3 shows the Gillnet locations for 2018. Gillnets are often set near the mouth of incoming tributaries and the locations of each net set (lat, long) will be recorded. The Tenmile site will be fished in the Spring and the Canada site in the Fall. Rexford will be fished in the Spring and Fall. At Rexford, 14 nets are set in the spring and 7 in the fall. Please see attachment B for a list of the lat/longs.

3.3 Sampling Timeframe

Sampling will run from approximately May 2018 through the end of September, 2018. The sampling time may be extended, if necessary. Reasons that the sampling may be extended are if trout or another target species become available and may supplement our catch numbers. If this occurs, the QAPP will be addended or modified.

4.0 Field Sampling Methods

4.1 Fish Sample Collection

Fish identified by the SeTSC for the focus of fish tissue collection are as follows:
Cutthroat Trout; Rainbow Trout, Peamouth Chub, Red Side Shiner, Northern Pikeminnow and Longnose/Largescale Sucker.

Burbot, Kokanee and Bull Trout (for trends and baseline analyses will also be collected for selenium analysis).

Species	Tenmile -spring Catch rate	Rexford Spring catch rate	Rexford Fall catch rate	Canada Fall Catch rate	Target catch rate / season/species	Actual Catch prediction	Stomach analysis	multiple tissue analysis	Spawning time
Bull Trout (Salvelinus confluentus)	11	50	1	1	5	5/5/1/1			
Kokanee (Oncorhynchus nerka)	3	24	74	124	5	3/5/5/5			Nov-Dec
Largescale sucker (Catostomus macrocheilus)	70			20	10	10/0/0/5	Y	Spring eggs / filet/whole body	April-May
Longnose sucker (Catostomus catostomus)	25	15	0	.4	10	10/10/0/1	Y	Spring eggs / filet/whole body	April-early July
Mountain White Fish (Prosopium williamsoni)	11			8					
Northern Pikeminnow (Ptychocheilus oregonensis)	73	290	160	143	10	10/10/10/5		Spring eggs / filet/whole body	May-Early July
Peamouth (mylocheilus caurinus)	192	886	160	180	10	10/10/10/5		Spring eggs / filet/whole body	May-June
Yellow perch (Perca flavescens)	1	14	1	11					April-May
RB Trout (Oncorhynchus mykiss)		11	4	4	10	0/10/5/5		Spring eggs / filet/whole body	Late April-early July "spring"
Westslope Cutthroat Trout (Oncorhynchus clarki lewisi)		2	1	5	10	0/5/5/5		Spring eggs / filet/whole body	Late April-early July "spring"
Burbot** (Lota lota)				.1	5	5			

Redside Shiner* (Richardsonius balteatus)	5	5	5		5/5/5	5/5/5	Yes focus	Spring eggs / filet/whole body	May-early August
Predicted Total analyses						186		250 analyses	

*Redside Shiners will be fished via electrofishing.

' fish per sampling event. An event is setting the gillnets overnight at each location outlined in the QAPP (attachment B).

**Burbot are occasionally caught in gillnets, in the case that they are found, they will be analyzed for selenium.

Table 3. Previous catch rates (catch rates from Jim Dunnigan, email communication, 2018). Fish for SeTSC focus are shaded in gray. The target catch rate/season/species column is the maximum number of fish we will pull from the nets/event for 2018 sampling. Based on past fishing efforts, the next column "Actual Catch prediction" estimates how many of each species/event we will catch for planning and cost estimates. The fish where stomach analysis will be performed on are indicated as are spawning times when eggs may be present.

All fish, excluding the Redside Shiner will be collected from annual gill-netting efforts conducted by FWP in 2018 during fall and spring on Lake Koocanusa (see attachment B for net locations).

All fish will be measured (total length (mm), weight (g)) and sampled in the field as described in "Mitigation for the Construction and Operation of Libby Dam, Annual Report 2012" (Dunnigan et al., 2014)- excerpt below. Fish will be sampled by lethal means (fish in nets are largely expired). If eggs are available in key species (see Table 3) they will be collected as described in the Fish Tissue Sampling Standard Operating Procedure, DEQ, 2015 (attachment D).

Gillnets: Sinking gillnets will be deployed in May at 14 locations (for past locations, see Table D1). Floating gillnets will be deployed at seven locations in the fall (Attachment B) in both the Montana and British Columbia portions of Lake Koocanusa. FWP retrieves the nets after soaking overnight by pulling them back into the boat and storing them in individual tubs marked by location. Tubs are then transported to shore, where a team of biologists and technicians remove fish from the nets, weigh, and measure each fish by net site. Dunnigan et al. (2014) provides additional methodology details related to gill net sampling on Lake Koocanusa. Once the nets are pulled from the reservoir and brought to shore, all fish in the nets will be measured, weighed and recorded by the FWP Libby crew for their population studies and reported on in their annual mitigation reports. Targeted fish for selenium analysis will be sorted out and processed as described below.

4.2 Fish Sample Preparation

Sources of field contamination to tissue should be identified and eliminated or minimized before the sampling event begins.

When fish are processed in the field, care should be taken to avoid contamination (away from exhaust in a clean area). Personnel must wear new, nitrile, disposable gloves prior to any contact with the

specimen. The sampling site, date, species, length (mm-TL) and weight (g) are measured and recorded in the field. If a clean area has been set up in the field, the filet can be extracted (or this can be done in a clean lab) from the right-hand side of the fish using a sterile (standard) fillet knife and cutting board and this should be noted on the site visit form (SVF) that the extraction was done in the field. For Se analysis, the filet is prepared skin off. Upon extraction, the muscle fillet sample is either placed in labeled tinfoil and placed in zip-lock style bag, or placed directly into a sterile amber glass jar with Teflon lid and placed in cooler on ice for submission to the laboratory for analysis. Between samples, the fillet knife is cleaned with phosphate free lab soap and water. The cutting surface is kept clean by frequent rinses, or by placing tinfoil on the surface of the board between samples. The sampling volumes, containers, preservation and holding time requirements for all samples collected are summarized in Table 6. The samples will be frozen at -20°C until the time of analysis.

Where eggs are found, they will be removed in the field or lab. The eggs will be collected as described in the DEQ 2015 Fish Tissue SOP (DEQ, 2015), as follows:

"Collection of fish eggs for sampling purposes may be desired for projects investigating concentrations of certain parameters. Field collection activities must coincide with the spawning times of the project's target species. Spawning times for species typical to Montana can be found in "Spawning Times of Montana Fishes," (Skaar, 2001). It may be difficult to achieve sufficient sample mass from small species or immature populations; therefore, SAPs should contain contingencies for these events.

If fish will be sacrificed, eggs can be harvested during dissection for collection of other tissues through the procedures for filleting and organ harvesting described above. Eggs are located along the gonads and typically form in rows. Manual stripping or spawning of eggs from ripe females should provide sufficient mass of eggs for sampling purposes and allow return of fish to the environment. Spawning activities should be performed in a manner that minimizes handling of fish and anesthetics should be used to reduce stress (Piper et al., 1982). Eggs themselves are delicate and can be damaged if fish are handled and/or spawned too roughly (Shrable et al., 1999).

Manually strip eggs as follows:

- Anesthetize fish if appropriate (see **Section 2.4.2**).
- Ready clean sample glassware. One large container to collect eggs in and distribute them to smaller containers may be appropriate.
- Rinse fish in ambient water, particularly in the area of the vent (see **Figure 3-1)** to remove any foreign matter and potential contaminants.
- Hold the fish about the head with one hand while positioning the vent over the receiving container and tilted slightly downward. Two individuals may be necessary to sample very large specimens–one person holding the fish and one person performing the stripping.
- Apply gentle pressure with the other hand to the ventral side beginning well behind the pectoral fin and moving slowly toward the vent. Direct the resulting stream of eggs into the sampling container.
- Room must be left in final sampling glassware for sample expansion upon freezing (Murphy, 2012).
- Label the samples in accordance with ***Section 7.2***, complete **Attachment A** – Fish Tissue SVF, freeze the samples as quickly as possible, and store at ≤ -20°C.

If the eggs are not easily stripped with gentle pressure, the fish is likely not ripe. Do not attempt to force stripping with excess pressure as harm to the fish and damage to the eggs may occur. Under this circumstance, the fish should be returned to the water without sampling or it will need to be sacrificed to obtain eggs."

For whole body analysis, the remaining carcass or whole fish will be wrapped in tinfoil, put in a large zip lock bag and placed on ice. Processing of whole body fish will be done in the FWP lab in Helena or Energy Labs in Helena, for details, please see below in section 5.

Lab Measurement	Container	Holding Time	preservation
Moisture			
Digestion for metals analysis			
Selenium (& Cd, Cu, Pb, As)	Whole fish or filets in aluminum foil and in zip top bag on ice Eggs in screw top jar (glass/HDPE) or whirl pac	6 months*	-20°C

* U.S. EPA, 2000

Table 4. Fish tissue containers/packaging, preservation and holding times

4.2.1 Lethal Method

If necessary, fish will be euthanized by a sharp, forceful blow to the head using a blunt, clean instrument. The force of the blow should be similar (slightly less) to that needed to drive a nail into wood (Erway et al., 2004). Fish from gillnets are typically already expired.

4.3 Deviations from QAPP

Any deviations from the QAPP will be reported to the field lead as soon as the deviation is identified. The field lead will inform the project managers, Selch, Mavencamp and the QA/QC officer, Hauer. The deviations will be recorded in writing and appended to the QAPP file (hard copy and on-line). If the deviations will affect laboratory processes and data, both the lab, the DEQ data manger, Jolene McQuillan, and the DEQ QA/QC officer, Michelle Hauer, will be notified. A plan to address the deviation will be sent to the appropriate individuals.

If changes to the QAPP are needed the QA/QC officer will determine if they significantly impact the technical and quality objectives of the project. If they do, Terri Mavencamp will modify the QAPP to document the change and submit the revision for approval. Following approval, the changes may be implemented.

5.0 Laboratory Analytical Measurements

Lab Measurement	Method	Reporting Limit (mg/kg)
Moisture	D2974A (modified dry 60-65C)	0.20 %
Digestion for metals analysis	EPA 3050	n/a

Selenium (Cd, Cu, Pb, As also using same method)	SW6020	0.50

Table 5. Laboratory methods and required reporting limits.

Fish Tissue moisture determination: The following excerpt is from the EPA Technical Support for Fish Tissue Monitoring for Implementation of EPA's 2016 Se Criterion # EPA820-F-16-007, Sep, 2016.

> *Egg and tissue samples should be thawed, and wet weight recorded for each individual sample. To prevent cross contamination between samples, a plastic foil (e.g., parafilm®) should be placed on the scale and replaced after each weighing. Samples are oven dried at 60°C until constant weight is recorded. It is required to record the moisture content for each individual sample in order to express analytical data on a dry weight basis. Trace element (e.g., selenium) analysis is routinely performed using hydride generation atomic absorption spectrophotometry (HG-AAS) or inductively coupled plasma-mass spectrometry (ICP-MS) and reported on a dry-weight basis (Janz and Muscatello, 2008).*

Whole Body Fish Analysis: Whole body fish will be homogenized in a high-power blender before digestion and analysis either in the FWP lab or at the analytical laboratory. Fish may be cut into smaller 2.5 cm cubes with high-quality stainless steel or titanium knives prior to homogenization. Fish should be ground until homogenous in appearance. Percent moisture will be determined for the whole-body fish as performed for filets (see above). If the filet/eggs were extracted for separate analysis, the final whole-body percent moisture and selenium concentrations will be determined from the mass percent and concentrations the filet will be added back to the whole fish during homogenization and included in the analysis.

6.0 Quality Assurance and Quality Control Requirements

Data quality indicators (DQIs) need to be defined to ensure the quality of the data for decision-making. DQIs, which include precision, accuracy, representativeness, completeness, comparability, and sensitivity, are quantitative and qualitative criteria established for the data acquired within this design to assure it is of sufficient quality for its intended use. The DQIs for this project are defined below.

6.1 Precision

Precision is the degree of agreement between or among independent measurements of a similar property (often reported as relative percent difference [RPD]). This indicator relates to the analysis of duplicate laboratory or field samples. Duplicates document the effect of sample homogeneity and matrix limitations on method performance. This project will rely on analytical and field duplicates to assess precision based on their RPD.

$$\text{RPD (as \%)} = ((\text{Sample Result} - \text{Duplicate Result})/((\text{Sample Result}+\text{Duplicate Result})/2)) \times 100$$

For samples that come back at the RL or 5 times the MDL, the pairs should agree within 30%. For samples that come back at less than five times the RL the pairs should agree within +/-50%. If duplicates

fail the above criteria, all associated sample results will be qualified with a "J", which means estimated (for qualifiers, please see appendix C).

In addition to these duplicate composite splits, all labs are to perform and report on the standard suit of QC measures.

Performance criteria are based on overall data quality and not achieving the DQI for a single precision measure does not imply the data are unacceptable for use in the study.

Analytical Precision (Laboratory Duplicates)

Precision quality control (QC) for all laboratory methods will follow the frequency specified in the analytical method or as described in a laboratory quality assurance plan (LQAP). Precision for laboratory duplicates will be assessed by ensuring that the RPD is ≤30%.

Field Precision (Field Duplicates)

Field duplicates shall be collected for 10% of all samples collected. Precision for field duplicates will be assessed by ensuring that the RPD is ≤30%.

Fish duplicates will be taken either by two biopsies in one fish or by splitting the whole (sacrificed) fish into two samples.

If duplicates fail the above criteria, all associated sample results will be qualified with a "J" flag.

6.2 Accuracy

Accuracy is the degree of agreement of a measurement with a known or true value. To determine accuracy, a laboratory or field value is compared to a known or true concentration. Measures of accuracy include calibrations (accuracy over a range of values), laboratory control samples (LCS) and sample specific controls such as matrix spikes (MS).

Laboratories are responsible for method accuracy in initial and continuing calibrations in accordance with the analytical method requirements. LCS and MS are common measures of accuracy in analytical laboratories. LCSs are prepared by spiking reagent water with a known concentration of an analyte. The results are compared to the known value to determine a percent % Recovery.

$$\% \text{ Recovery (LCS)} = (\text{Analytical Result}/\text{True Value}) \times 100\%$$

Matrix Spikes are prepared by spiking a sample with a known concentration of an analyte. The results are compared to the known value to determine a percent % Recovery.

$$\% \text{ Recovery (MS)} = ((\text{Spiked Sample Result} - \text{Sample Result})/\text{Amount Spiked}) \times 100\%$$

Matrix spikes, calibration and continuing calibration and certified reference material measures should fall within the acceptance criteria specified for each method. If they do not, they should be rerun until compliance is achieved or results may be rejected from the lab and affect lab completeness and payment.

6.3 Representativeness

Representativeness is the expression of the degree to which data accurately and precisely represent an environmental condition in time and space. The selection of the sampling design (e.g., sample location, number of samples, and collection period) affects the monitoring project's representativeness. For this project, representativeness will be achieved by ensuring that spatial and temporal components are properly selected to adequately characterize the environmental condition and that this QAPP and field collection standard operating procedures (SOPs) are followed.

6.4 Completeness

Any loss of data due to site access issues, QC failures, or laboratory mistakes may result in no trend analyses, calculations of CFs, TTFs, or use in Se model development. To calculate completeness, compare the number of valid measurements completed (samples collected or samples analyzed) with those you originally planned to take. Our goal is to get 5 fish of each target species at each location and time collected. Because catches are variable and certain species prefer various locations in the reservoir, this goal may not be attainable. The completeness goal for this monitoring project is at least 90% of this goal collected and passing QC evaluation.

6.5 Comparability

Comparability is the extent to which data from one study can be compared directly to data from another study. To achieve a comparable result, both the field collection method and the analytical method must be comparable. This is achieved through the use of standardized sampling and analytical methods and by adhering to this QAPP, project sampling plans and SOP (DEQ, 2015).

6.6 Sensitivity

For this study, the MDL and ML will be used to define the sensitivity of each measurement process. The MDL is the minimum concentration that can be measured with 99% certainty that the analyte concentration is greater than zero. The procedures are given in 40CFR 136, Appendix B. The ML is the lowest concentration on the calibration curve or approximately the MDL x 3.18. The measurement quality objective for this fish tissue study is that the MDL and MLs are sufficiently sensitive to meet the reporting limits in Table 5. If the results fall between the MDL and ML, the results will be "J" flagged as estimates.

Analytical Sensitivity QC (Method Blanks)

Sensitivity quality control (QC) for all laboratory methods will follow the frequency specified in the analytical method or as described in a laboratory quality assurance plan and include continuing calibration verification standards (CCV) run at intervals during an analytical run. These should be the same standards as the calibration standards to be able to detect sensitivity changes during analysis. All data obtained while an instrument is out of control is not reportable and all samples must be reanalyzed or flagged with the appropriate qualifier and reported along with a narrative describing why the instrument was out of control and when/how corrective action was taken.

7.0 Data Analysis, Recordkeeping, and Reporting

7.1 Sample Handling and Custody

Field crews are responsible for the integrity of samples from the time of collection until shipment or drop-off to a laboratory. This responsibility includes proper preservation, labeling, sample custody documentation, and storage.

Samples will be hand delivered from the FWP field office in Libby to Helena by FWP staff. Once in Helena, the chain of custody section of the SVF will be updated and the fish stored in the freezer in the FWP laboratory. Samples will then be processed and hand delivered to Energy labs with the SVFs.

7.2 Sample Handling Procedures

After samples are collected and labeled, samples are placed in a clean cooler on ice. This temperature will be maintained until received by Energy Labs. The laboratory will keep samples at -20°C (or frozen) until the time of analysis.

7.3 Sample Labeling

All samples must have DEQ site visit stickers, unique Sample IDs, and should be clearly linked to the information on the SVF (attachment A). When multiple samples are made from one fish (i.e., filet, eggs, whole body (remaining carcass), the sticker must have a unique suffix for each tissue type, Filet = F1;F2, Egg = EGG and Whole body = WB.

7.4 Sample Custody

Custody documentation (SVF) will accompany all samples from the field to the laboratory (see Attachment A for the SVF with chain of custody information at the bottom). Field personnel will initiate custody documentation before samples are stored in the cooler and maintain the custody forms until the samples are shipped. The project lead will sign the custody documentation and inspect the integrity of the samples and documentation before shipment. Any missing information or discrepancies will be communicated to the field crew (if applicable). Once the laboratory or other recipient receives the samples, the recipient will sign the custody documentation. The custody will be tracked until the sample reaches Energy Labs and is analyzed. Each handler of the samples will inspect the integrity of the samples and documentation during the sample receipt. Any issues or discrepancies identified by the laboratory will be communicated to the project leads and field tech/laboratory coordinator.

7.5 Data Analysis

Data will be analyzed to determine the variation in selenium concentrations (muscle, egg/ovary, whole body) in each species and will be compared to past years. Typical statistical analysis will be run on the data (regression analysis, goodness of fit (R^2 analysis)) to determine the relationship between selenium concentrations in different body tissues, i.e., muscle and ovary, ovary and whole body, etc.

Conversion factors for each species will be determined where there are enough data and where the data can be plotted on a linear plot. A statistical relationship between egg-ovary and whole body (or muscle) or between filet and whole body will be determined for each species using ordinary least squares (OLS) linear regression of the matched pairs of measurements as described in the EPA aquatic life selenium criteria document on page 77 (EPA, 2016). Conversion factors that may be calculated from the data may be seen in Table 6.

Type of Analysis	Determination	Conversion Factor (s)
Fillet	Percent moisture, Se	Muscle to WB
Whole Body	Percent moisture, Se	muscle to whole body Egg/ovary to whole body
Egg/ovary	Percent moisture, Se	Egg ovary to whole body Egg ovary to muscle

Table 6. Possible conversion factors that may be calculated from the data.

In addition, the fish may be compared to Fish Consumption Advisories. For more details on this, please see the FCA Draft QAPP, 2018.

Fish meals/month	Fish tissue concentrations (ppm wet weight)	Fish tissue concentrations (ppm dry weight) using a 75% moisture
Unrestricted > 16	• 1.5	0-6
16	>1.5-2.9	>6.0- 11.6
12	>2.9-3.9	>11.6-15.6
8	>3.9-5.9	>15.6-23.6
4	>5.9-12	>23.6-48
1	>23-47	>92-188
None (<0.5)	>94	>376

Table 7. Selenium fish tissue concentration guidelines (EPA, 2000b) and approximate conversions between fish tissue selenium wet and dry weight for quick reference.

7.6 Reports to Management

A brief report with the results of this study will be submitted to DEQ management. In the report, a brief project quality audit/assessment from the audit of data quality will be submitted; and if any of the DQOs were not achieved, a corrective action will be outlined describing how failure to meet the DQOs may be avoided in future study plans.

Results of this study will be presented to the SeTSC/MRC by FWP in the next face-to-face meeting planned for October 2018, currently scheduled to take place in British Columbia. The data will also be available on the wiki site either included in the selenium data pull or as a reference to where the data is available.

7.7 Data Management

DEQ-funded data are entered into MT-eWQX where it is subsequently entered into the USGS/EPA Water Quality Portal for public access https://www.waterqualitydata.us// This project is to follow the WQPB "internal process." This internal process consists of writing a sampling and analysis plan (SAP) or quality assurance project plan (QAPP) and submitting it to the DEQ QA/QC officer. Once the sampling plan has been approved and filed, and the sampling is being undertaken, appropriate Site Visit Forms (SVFs) and any additional field forms need to be filled out so that laboratory results can be processed by WQPB staff. One SVF will be filled out for each site/net collection, and multiple Site Visit Codes will be placed on each SVF. A unique Site Visit Code sticker will be used for each fish collected. If multiple samples are created from one fish, the same Site Visit Code sicker will be used and a unique suffix will distinguish between the multiple samples (such as C1200-F1 and C1200-F2). If the samples are to be brought back to the FWP lab for further processing, please fill out the SVFs at the time the samples are packaged for the labs.

When samples are dropped off at the Energy Labs, each SVF is signed and handed over to the lab personal who takes the temperature of the sample and signs the sample in. The lab will make copies of the SVFs for their records and will return the original SVFs to the FWP/DEQ staff that delivered the samples. The copies need to be filed with WQPB administration (currently Deanna Tartum), who will enter the forms into our electronic records system.
For site visit forms, tissue sample forms and fish ID summaries, please see attachments or find files here G:\WQP\6_DataMgmt\3_EQuIS\EQuIS_Resources\EQuIS_Field_Forms\Masters.

9.0 References

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10.0 ATTACHMENTS

10.1 ATTACHMENT A: SITE VISIT FORM

Fish Tissue – Multiple Samples
Site Visit Form

Project ID: _____

Date: _____ Time: _____ Personnel: _____

Waterbody: _____ Location: _____

Station ID: _____ HUC: _____ County: _____ AUID: _____

Latitude: . Longitude: . Elevation: _____ ft m

Sample Information (for all samples collected unless otherwise noted in comments)	
Lab Analysis:	
Preservation:	Ice/-20C Freezer Other: _____
Extraction:	FIELD LAB

Site Visit Comments

Chemistry Lab Information		
Lab Samples Submitted to:	Account #:	Term Contract Number:
Invoice Contact:		
Contact Name & Phone:		EDD ☒ Format: MT-eWQX Compatible
1) Relinquished By & Date/Time:	1) Shipped By: Hand ☐ FedEx/UPS ☐ USPS ☐	1) Received By & Date/Time:
2) Relinquished By & Date/Time:	2) Shipped By: Hand ☐ FedEx/UPS ☐ USPS ☐	2) Received By & Date/Time:

Lab Use Only - Delivery Temperature: Wet Ice _____ °C Dry Ice _____ °C _____

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Fish Tissue – Multiple Samples
Site Visit Form Continued

Samples Collected **Use one Site Visit Code sticker for each fish. Use the same Site Visit Code sticker for all the samples created from each fish.

Species Codes: BULL=Bull Trout; KOK=Kokanee; LING=Burbot; LN SU=Longnose sucker; LS SU=Largescale sucker; N PMN=Northern Pikeminnow; PEA=Peamouth; RB=Rainbow Trout; RS SH=Redside Shiner; WCT=Westslope Cutthroat Trout

Site Visit Code	Sample Collection Method	Species	Length (mm)	Weight (g)	Sex	Eggs Present	Developmental Stage
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	
	WHOLE FILLET EGGS	BULL KOK LING LN SU LS SU N PMN PEA RB RS SH WCT			M F	Y N	

10.2 Attachment B: Fish Sampling Locations from FWP for 2018

		Canada Gillnet Locations	**seasons**
Lat	Long	Site Description	Fall only
49.23524	-115.2718378	West of Ramp	
49.1991414	-115.2356747	North of Cabin (Campground)	
49.17813013	-115.2317702	Cabin	
49.16665998	-115.2311636	South Point Elk	
49.17628068	-115.2208864	North Point Elk	
49.19489758	-115.2164987	Across from Campground	
49.25347538	-115.2652918	South of Bridge	
49.27933877	-115.2725245	North & West of Kikomun (Sinker)	
		Tenmile Gillnet Locations	**Spring only**
		Site Description	
48.59060674	-115.2138805	South Point Ten Mile	
48.56416657	-115.2070315	Big Bend	
48.54680961	-115.2448403	North Point Bristow (Floater)	
48.53870172	-115.2649008	South Point Bristow	
48.505188	-115.288477	North Point Barron	
48.499751	-115.266774	South Point Warland	
48.468669	-115.310307	North Point Jackson	
48.422685	-115.300418	South Point Canyon	
		Rexford Gillnet Locations	**Fall and spring**
		Site Description	
48.87599549	-115.2103134	South Black Lake	Spring
48.88590903	-115.1993054	North Black Lake	Spring
48.89789834	-115.1853524	South of Far South Tobacco	Fall and Spring
48.90495353	-115.1797746	Far South Tobacco	Fall and Spring
48.9062048	-115.1741338	South Tobacco	Spring
48.91839349	-115.1757507	North Point Tobacco	Fall and Spring
48.93303993	-115.1554178	North of North Point Tobacco	Spring
48.95015213	-115.1538033	North Point Murray	Fall and Spring
48.960534	-115.1554967	South Sophie	Spring
48.97593677	-115.1398817	Middle Sophie	Spring

48.99008499	-115.1598875	North Sophie	Fall and Spring
48.96958238	-115.1785839	North Point Young Creek	Fall and Spring
48.94934256	-115.1855495	Sandhill	Fall and Spring
48.9308041	-115.1908913	North Point Dodge Creek	Spring

10.3 ATTACHMENT C: QUALITY CONTROL CHECKLIST

EDD - Activity

__ Lab EDD Activity IDs match the SVF/SUDS Activity IDs.

__ Lab EDD Activity Start Date and Time match the date/time the samples were collected.

__ Field duplicates and field blanks are clearly identified by an Activity Comment, Result Comment, and appropriate Activity Type.

Lab Report

__ Reporting detection limits meet the project-required detection limit defined in SAP.

__ Laboratory blanks/duplicates/matrix spikes/lab control samples were all within the required control limits defined within the SAP/QAPP. If any samples exceeded the control limits, the associated data is "J" flagged.

1.0 Lab Qualifier "S" (Spike recovery outside of advisory limits) = apply "J" flag with Result Comment "MS/MSD failed [high or low] (xx/xx%), expect [high or low] bias." Apply flag and comment to all associated results (in the same batch) that were detected above the LRL.

__ All method blanks are less than the project-required detection limit. If a method blank has a detect level at or above the reporting limit (LRL), then samples up to or equal 10x the detected value are "B" flagged with a Result Comment "Method blank contamination, results <[x.xx] mg/l are B flagged." The actual blank is not "B" flagged. Non-detects are not flagged.

EDD - Results

__ Holding times met. If any data exceeds the holding time, an "H" flag and Result Comment are added (such as "Sample exceeded EPA 7 day holding time.")

__ Ensure the Analytical Method ID matches the laboratory report and followed the method defined in the SAP.

__ Ensure approriate Result Value Units are used. Ensure that the Result Values and the Result Value Units correlate.

__ Ensure Characteristic ID, Characteristic Name, Method Speciation Name, and Sample Fraction are entered approriately and correctly. Refer to Appendix C in the data manager desk manual.

__ If the result value is between the MDL and the LRL, the result is "J" flagged and has a Result Comment "Result between MDL and LRL, J flagged as estimate."

__ All field blanks are less than the project-required detection limit. If a field blank has a detect level at or above the reporting limit (LRL), then samples up to or equal 10x the detected value are "B" flagged with a Result Comment "Field blank contamination, results <[x.xx] mg/l are B flagged." The actual blank is not "B" flagged. Non-detects are not flagged.

__ Field duplicates were all within the required control limits specified in the SAP/QAPP (usually if result value >5x LRL, then duplicates should be within 25% of each other). If any field duplicates exceeded the project-required control limits, all associated results are "J" flagged with a Result Comment "Field duplicate RPD >25% (xx%)." Or "Associated field duplicate RPD >25% (xx%)."

__ All samples requiring dilutions have a "D" result qualifier and the associated detection/reporting limits have the dilution factor applied.

__ For STREFPRO State Lab cations with a sample fraction of "Free Avail", add a Result Comment: "Analyzed directly from acid-preserved bottle without digestion." Only project allowed to have Free Avail sample fraction is STREFPRO.

__ All samples that have result qualifiers in the laboratory report have the appropriate qualifier, or equivilent qualifier, in the Result Qualifier field.

__ Ensure that Total Nitrogen results are greater than both Nitrate+Nitrite and Ammonia (within 10% is ok). If TN is less than the total, apply "J" flag and Result Comment describing why the Result Value is estimated.

__ Ensure that Total Phosphorus results are greater than SRP/Orthophosphate (within 10% is ok). If TP is less than SRP, apply "J" flag and Result Comment describing why the Result Value is estimated.

__ Ensure that total recoverable metal results for a particular analyte are greater than the dissolved fraction.

- When detected at normal levels, well above the reporting limit, always go with 10% since that is typical for an RPD limit if you were comparing duplicates. However, when it is a really low level, the 10% rule doesn't work. If they were different by more than the reporting limit for a low-level result, have the lab recheck the results.

__ Look for any unusual outlier data by analyte.

Data Qualifiers and Descriptions

Result Qualifier	Result Qualifier Description
B	Detection in blank.
D	Contract Required Quantitation Limit (CRQL) not met due to sample matrix interference, dilution required.
H	Holding time exceeded.
J	Estimated: The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample.
L	Lowest available reporting limit for the analytical method used.
R	Rejected: The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
**Any combination of these qualifiers can be associated with each result value.	

Quality Control Terminology and Descriptions

FIELD QC		
Term	**Description**	**Purpose/Usage**
Trip Blanks	Prepared at the lab prior to the sampling event and kept with the collected samples throughout the sampling trip.	To determine if cross contamination occurs between samples and identify contaminants that may be introduced into samples during transit to and from the lab.
Field Blank	Prepared in the field with lab water and kept with the collected samples throughout the sampling trip.	Monitors contamination resulting from field activities and or ambient levels of analytes present at time of sampling.
Field Duplicate	Two independent samples taken under the same conditions. Water samples would be two independent samples taken at the same location at the same time.	To determine the homogeneity of the samples collected.
LABORATORY BATCH QC		
Acronym	**Description**	**Definition**
LRB/Method Blank	Laboratory Reagent Blank	An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if method analytes or other interferences are present.

LFB/LCS	Laboratory Fortified Blank; Laboratory Control Sample	Reagent water spiked with a known amount of analyte. Ideally treated exactly like a MS/LFM. Control used to determine bias in sample spikes.
MS/LFM	Matrix Spike/Laboratory Fortified Matrix	An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations
MSD/LFMD	Matrix Spike Duplicate/Laboratory Fortified Matrix Duplicate	Determine method precision in sample concentrations are < 5X the RL.
DUP	Duplicate	Determine method precision in sample concentrations are > 5X the RL.
QCS	Quality Control Sample	A solution of method analytes of known concentrations which is used to fortify an aliquot of reagent water or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check either laboratory or instrument performance
SRM	Standard Reference Material	Primarily used as a QCS to verify instrument calibration.

LABORATORY ANALYSIS QC

Acronym	Description	Definition
ICB	Initial Calibration Blank	Monitors instrument drift at low end of calibration curve.
CCB	Continuing Calibration Blank	Monitors instrument drift at low end of calibration curve.
ICV	Initial Calibration Blank	Monitors instrument drift at a defined concentration near the mid range of calibration curve.
CCV	Continuing Calibration Blank	Monitors instrument drift at a defined concentration near the mid range of calibration curve.
IPC	Instrument Performance Check	Monitors instrument drift at a defined concentration near the mid range of calibration curve.
MS/LFM	Matrix Spike/Laboratory Fortified Matrix	An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations
MSD/LFMD	Matrix Spike Duplicate/Laboratory Fortified Matrix Duplicate	Determine method precision in sample concentrations are < 5X the RL.

DUP	Duplicate	Determine method precision in sample concentrations are > 5X the RL.
QCS	Quality Control Sample	A solution of method analytes of known concentrations which is used to fortify an aliquot of reagent water or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check either laboratory or instrument performance
SRM	Standard Reference Material	Primarily used as a QCS to verify instrument calibration.
IDL	Instrument detection limit	Signal just above baseline. 3-5x the STD DEV of 7 replicates of a blank. Not used for quantification.
MDL	Method detection limit	Statistical determination of the lowest concentration of an analyte with 95% certainty the analyte is present.
PQL	Practical Quantitation Limit	3-5x the MDL. Lowest level that quantification is determined
RL	Reporting Limit	Value a Laboratory reports results. Usually the PQL.

11.0 APPENDICES

11.1 APPENDIX A: LABORATORY QC SECTION, SELENIUM IN TISSUE:

A non-routine QC check was done for the following three laboratories that DEQ/FWP have used for fish tissue analysis: contract FWS lab Envirosystems Inc.; Energy Labs, Helena MT; and the Department of Public Health and Human Services lab (HHS), Helena, MT. The certified reference material, DORM-4 was purchased from the National Research Council of Canada and the specs are shown below https://www.nrc-cnrc.gc.ca/eng/solutions/advisory/crm/certificates/dorm_4.html .

The material will be thawed slightly (from -20°C) and subdivided into three portions weighing 5 g each and sent on ice overnight to the FWS lab and hand delivered to Energy and Human Health and Environmental labs. Each standard sample will be delivered with approximately 4 other fish tissue samples, and the analytes shown in table D-1 are requested: The estimated cost is 500-600$/lab.

Lab Measurement	Method	Reporting Limit (mg/kg)
Moisture	D2974	0.20
Digestion for metals analysis	EPA 3050	n/a
Selenium	SW6020	0.50 dry weight
Cadmium		.088 wet weight
copper		1 wet weight
lead		.02 wet weight
arsenic		.02 wet weight
Mercury	SW7471B	0.029 wet weight

Table A1: Requested Analytes for QC check of labs and requested reporting limits.

Element	(mg/kg)
Arsenic (b,d,f)	6.87 ± 0.44
Cadmium (a,d)	0.299 ± 0.018
Calcium (d,e)	2360 ± 140
Chromium (a,d,e)	1.87 ± 0.18
Copper (a,d,e)	15.7 ± 0.46
Iron (a,d)	343 ± 20
Lead (a,b,d)	0.404 ± 0.062
Magnesium (d,e)	910 ± 80
Manganese (b,d,e)	3.17 ± 0.26
Mercury (a,c,g)	0.412 ± 0.036
Nickel (a)	1.34 ± 0.14
Potassium (d,e)	15 500 ± 1000
Selenium (a,d,f)	3.45 ± 0.40
Silver (a,d)	0.0252 ± 0.0050
Strontium (d,e)	10.1 ± 0.8
Vanadium (d,e)	1.57 ± 0.14
Zinc (a,d)	51.6 ± 2.8

Table A-2 Certified reference material, DORM-4 specs purchased from the National Research Council of Canada.