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UPPER COLUMBIA RIVER

REMEDIAL INVESTIGATION AND FEASIBILITY STUDY

Quality Assurance Project Plan for the

Assessment of Sediment Toxicity to White Sturgeon (*Acipenser transmontanus*)

Prepared for

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Prepared by

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May 2010

SECTION A: PROJECT MANAGEMENT

A1 TITLE AND APPROVAL SHEET

QUALITY ASSURANCE PROJECT PLAN FOR THE "ASSESSMENT OF SEDIMENT TOXICITY TO WHITE STURGEON (*Acipenser transmontanus*)"

Quality Assurance Project Plan Approvals

EPA Project Coordinator:	Helen Bottcher	Helen H. Bottcher	Date: 05-07-10
EPA Quality Assurance (QA) Manager:	for Gina Grepo-Grove		Date: 5/14/10
Teck Project Coordinator:	Marko Adzic		Date: 05-07-10
Teck Technical Team Coordinator & Task Manager:	Markus Hecker		Date: 5/17/10
U of S Principal Investigator:	John Giesy		Date: 5/18/10
Task QA Coordinator:	Shaun Roark		Date: 5/18/10
Database Manager:	Randy O'Boyle		Date: 5/24/10
Analytical Chemistry Lab Coordinator:	Kris McCaig	Kris McCaig	Date: 5/7/10
Chemical Laboratory Project Manager:	Jeff Christian		Date: 5/27/10
Chemical Laboratory QA Manager:	Julie Gish	Julie Gish	Date: 5/24/10

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ACRONYMS AND ABBREVIATIONS

Agreement	June 2, 2006, Settlement Agreement
ASTM	American Society for Testing and Materials
AVS	acid volatile sulfides
BERA	baseline ecological risk assessment
BLM	biotic ligand model
$CaCO_3$	calcium carbonate
CAS	Columbia Analytical Services
COC	chain-of-custody
COPC	contaminant of potential concern
CUR	condition-upon-receipt
DBMS	database management system
DGT	diffusive gradient thin-film
DHSE	University of Saskatchewan Department of Health, Safety, and Environment
DO	dissolved oxygen
DOC	dissolved organic carbon
dph	days post hatch
DQO	data quality objective
Ecology	Washington State Department of Ecology
EDD	electronic data deliverable
ELS	early life stages
ENTRIX	ENTRIX, Inc.
EPA	U.S. Environmental Protection Agency
ERB	equipment rinsate blank
ESI	Environmental Services, Inc.
FSP	field sampling plan

ID	identification
ICP/MS	inductively coupled plasma/mass spectrometry
LC20	lethal concentration for 20 percent of the test population
LC50	lethal concentration for 50 percent of the test population
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LOAEC	lowest-observed-adverse-effect concentration
MDL	method detection limit
MRL	method reporting limit
MS/MSD	matrix spike/matrix spike duplicate
NOAEC	no-observed-adverse-effect concentration
PARCCS	precision, accuracy, representativeness, comparability, completeness, and sensitivity
PDA	personal digital assistant
QA	quality assurance
QA/QC	quality assurance and quality control
QAPP	quality assurance project plan
RI/FS	remedial investigation and feasibility study
RM	river mile
RPD	relative percent difference
SEM	simultaneously extracted metals
Site	Upper Columbia River site
SLERA	screening level ecological risk assessment
SM	Standard Methods for the Examination of Water and Wastewater
SOP	standard operating procedure
SRM	standard reference material
SRMD	standard reference material duplicate

TAL	target analyte list
TDS	total dissolved solids
Teck	Teck American Incorporated
TOC	total organic carbon
U of S	University of Saskatchewan
UCR	Upper Columbia River
USGS	U.S. Geological Survey

UNITS OF MEASURE

°C	degrees Celsius
L	liter(s)
mg	milligram(s)
mL	milliliter(s)
µg	microgram(s)

A3 DISTRIBUTION LIST

EPA Project Coordinator:	Helen Bottcher
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Database Manager:	Randy O'Boyle
Data Validator:	Rock Vitale
Analytical Chemistry Lab Coordinator:	Kris McCaig
Chemical Laboratory Project Manager:	Jeff Christian
Chemical Laboratory QA Manager:	Julie Gish

A4 INTRODUCTION AND TASK ORGANIZATION

A4.1 Introduction

This Quality Assurance Project Plan (QAPP) presents the approach and rationale for conducting a study to assess the toxicity of contaminants of potential concern (COPCs) associated with granulated slag from sediments in the Upper Columbia River (UCR) site (Site)[1] to early life stages (ELS), including the hiding stage, of white sturgeon (*Acipenser transmontanus*). Data obtained during this work will be used in the baseline ecological risk assessment (BERA) and overall Remedial Investigation/Feasibility Study (RI/FS). Primary objectives of the RI/FS are to investigate the nature and extent of unacceptable risk at the Site, to provide information to support baseline risk assessments for human health (to be completed by the U.S. Environmental Protection Agency [EPA]) and the environment (to be completed by Teck American Incorporated [Teck]), and to develop and evaluate potential remedial alternatives for the Site.

Sediment toxicity to white sturgeon ELS will be evaluated using field-collected sediments from areas previously hypothesized as suitable white sturgeon habitat, and containing a range of slag-related COPC concentrations. Based on work completed by others, suitable habitat for white sturgeon within the Site is limited to areas north of Kettle Falls and including Marcus Flats (Golder 2007; Howell and McLellan 2006).

In addition to the above-mentioned sediment toxicity tests, a limited number of water exposure acute toxicity tests using copper will be completed to supplement work being conducted by the U.S. Geological Survey (USGS).

A4.2 Task Organization

This section presents the organizational structure for activities associated with the work, including task management and oversight, fieldwork, sample analysis, and data management. ENTRIX, Inc. (ENTRIX), and the Environmental Toxicology Group at the

[1] The UCR site as defined within the June 2, 2006, Settlement Agreement is the areal extent of hazardous substances contamination within the United States in or adjacent to the Upper Columbia River, including the Franklin D. Roosevelt Lake, from the U.S.-Canadian border to the Grand Coulee Dam, and those areas in proximity to the contamination which are suitable and necessary for implementation of response actions (USEPA 2006a).

University of Saskatchewan (U of S) are conducting this work with oversight from EPA and with consultation from the Teck technical team.

A4.2.1 EPA Organization and Responsibilities

EPA will oversee Teck's activities associated with the work, and will coordinate U.S. Department of the Interior, Washington State Department of Ecology (Ecology), and tribal (i.e., the Confederated Tribes of the Colville Reservation and the Spokane Tribe of Indians) input regarding review of technical documents submitted by Teck. The project coordinator for EPA is Helen Bottcher. Ms. Bottcher will be responsible for ensuring that the work performed is consistent with all applicable EPA guidance. The EPA quality assurance (QA) manager assigned by EPA is Gina Grepo-Grove, or designee.

A4.2.2 Teck Organization and Responsibilities

Marko Adzic will serve as Teck's project coordinator and will have the primary responsibility for ensuring that Teck meets all the requirements and associated deliverables specified within the June 2, 2006, Settlement Agreement (Agreement) (USEPA 2006a). Dr. Markus Hecker will be responsible for overseeing all technical aspects of this task, and managing the overall task schedule.

A4.2.3 Key Task Personnel

Task Manager—Dr. Markus Hecker (ENTRIX; U of S) will oversee all task activities, review quality assurance reports, approve final project quality assurance needs, and communicate status of the work to the technical team and the Teck project manager. Dr. Hecker will be responsible for compiling summary results and project final reports. He will also be responsible for statistical analysis and data interpretation.

U of S Principal Investigator—Professor John P. Giesy (U of S) will advise the task manager in overseeing and approving all project activities, review quality assurance reports, approve final project quality assurance needs, and authorize necessary actions and adjustments related to U of S activities to accomplish program quality assurance objectives.

Study Team Leader—David Vardy (U of S), under the supervision of Dr. Hecker, will oversee research activities and supervise study crews. The study team leader will ensure that proper sample collection, preservation, storage, transport, and chain-of-custody (COC) procedures are followed; will inform the project quality assurance manager when problems occur; and will communicate and document corrective actions taken. As study team leader, Mr. Vardy will discuss study activities with the task manager.

Analytical Chemistry Laboratory Coordinator—Kris McCaig (Teck) is responsible for coordinating with the testing laboratory and tracking the laboratory's progress; verifying that the laboratory has implemented the requirements of this QAPP; addressing quality assurance issues related to the laboratory analyses; ensuring that laboratory capacity is sufficient to undertake the required analyses in a timely manner; and addressing scheduling issues related to laboratory analyses. As chemistry laboratory coordinator, Ms. McCaig will report directly to the project manager and will work closely with the study team leader and the quality assurance manager.

Quality Assurance Coordinator—Dr. Shaun Roark (ENTRIX) is responsible for providing overall QA support for the work and ensuring that the QAPP and FSP contain all components necessary to meet EPA guidelines (USEPA 2002a). As quality assurance manager, Dr. Roark will review program quality assurance activities, quality problems, and quality-related requests. In response to experimental and bioanalytical findings, he will approve corrective actions. Dr. Roark will also report quality non-conformances to the Teck project coordinator and review all pertinent portions of the U of S and ENTRIX deliverables before they are transmitted to ensure conformance with quality assurance and quality control (QA/QC) procedures and quality work products.

Data Manager—Randy O'Boyle (Exponent) is the database administrator and will have primary responsibility for incorporating the results of the work into the project database, including establishment of storage formats and standards for coding of data. Dr. Hecker will coordinate data acquisition and storage during the execution of the studies, and will coordinate with Mr. O'Boyle on the establishment of formats for storage and uploading of information to the project database.

Data Validator—Rock Vitale (Environmental Services, Inc. [ESI]) is responsible for coordinating the validation of analytical laboratory data; communicating data quality issues to the data users; and working with data users to address any data limitations. Mr. Vitale will report to the analytical laboratory coordinator, and will work closely with the task manager and the database manager to ensure that the objectives of the QAPP are met.

A4.2.4 Analytical Contract Laboratory

The following responsibilities apply to the project manager and quality assurance manager at the analytical laboratory used for the analysis of samples collected during the work. The analytical laboratory will be Columbia Analytical Services (CAS).

Laboratory Project Manager—Jeff Christian (CAS) is responsible for the successful and timely completion of sample analyses, as well as the following actions:

- Ensure that samples are received and logged correctly, that the correct methods and modifications are used, and that data are reported within specified turnaround times
- Review analytical data to ensure that procedures were followed as required in this QAPP, the cited methods, and laboratory standard operating procedures (SOPs)
- Apprise the analytical chemistry laboratory coordinator of the schedule and status of sample analyses and data package preparation
- Notify the chemical laboratory coordinator if problems occur in sample receiving, analysis, or scheduling, or if control limits cannot be met
- Take appropriate corrective action as necessary
- Report data and supporting quality assurance information as specified in this QAPP.

Laboratory Quality Assurance Manager—Julie Gish (CAS) is responsible for overseeing the quality assurance activities in the laboratory and ensuring the quality of the data for this task. Specific responsibilities include the following:

- Oversee and implement the laboratory's quality assurance program
- Maintain quality assurance records for each laboratory production unit
- Ensure that QA/QC procedures are implemented as required for each method and provide oversight of QA/QC practices and procedures
- Review and address or approve non-conformity and corrective action reports
- Coordinate responses to any quality control issues that affect this task with the laboratory project manager.

A4.3 Problem Definition and Background

Poor recruitment of white sturgeon in the Columbia River between Grand Coulee Dam and Hugh L. Keenleyside Dam in Canada has been documented since the 1970s. While adult spawning and occurrence of embryos and larvae have frequently been reported, limited numbers of embryos and larvae have been captured in this stretch of the river (Howell and McLellan 2006). Furthermore, juveniles (9 to 10 months old) that have been released as part of the White Sturgeon Recovery Program appear to have good survival,

growth rates, and body condition (Howell and McLellan 2006). However, survival of one or more stages during the early development of larval and juvenile sturgeon appears to be limiting recovery of the population (Howell and McLellan 2006).

Given the epibenthic nature of white sturgeon, there is the potential for increased exposure to contaminated sediments within the UCR. Specifically, there are concerns about the potential toxicity of COPCs associated with granulated slag to white sturgeon ELS, including the early hiding stage where fry are in proximity to sediments. Exposure pathways may include pore- and surface water at the sediment-water interface. Water may move through the sediments either from upwelling from deeper sediments, downward movement from surface water, or lateral fluvial flow.

In addition to the above-mentioned factors and based on unpublished data, it would appear that there is a critical stage (15 to 50 days post hatch [dph]) where white sturgeon experience relatively high mortalities, and may be particularly sensitive to water-borne contaminants such as copper. As a result, to supplement work being conducted by the USGS, a limited number of water exposure acute toxicity tests using copper will be completed during this critical life-stage.

A5 EXISTING DATA

There are no data that evaluate sediment toxicity to white sturgeon ELS. A limited number of static acute fish toxicity tests using rainbow trout (*Oncorhynchus mykiss*) exposed to granulated slag were identified and are provided in Appendix A. A number of environmental investigations have, however, identified elevated slag-related COPC sediment concentrations within the Site (Johnson et al. 1988; Bortleson et al. 2001; Era and Serdar 2001; Majewski et al. 2003; Cox et al. 2005; USEPA 2003, 2006b; Paulson et al. 2006). As noted in the site-specific Screening Level Ecological Risk Assessment (SLERA) (Teck 2010a), these COPCs indicate the potential for adverse ecological effects and warrant further investigation.

In addition to studies identifying elevated COPC sediment concentrations, unpublished data are available regarding the location of spawning grounds, nursing grounds, and movements of white sturgeon within the UCR. Based on these data, researchers have identified that the greatest abundance of white sturgeon larvae are located upstream of Marcus Flats (Howell and McLellan 2006; see Map A-1) with Marcus Flats representing a likely habitat for fry and juveniles (Golder 2007). As illustrated in Maps A-2 and A-3, slag-related COPC sediment concentrations within these white sturgeon habitats cover a wide range of concentration gradients and are at concentrations that warrant further

investigation (Johnson et al. 1988; Bortleson et al. 2001; Era and Serdar 2001; Majewski et al. 2003; Cox et al. 2005; USEPA 2003,2006a; Paulson et al. 2006). To assess the risk to white sturgeon ELS from exposure to COPCs through sediments in the UCR, toxicological information specific to this matrix must be collected and evaluated.

A6 DATA GAPS

Although numerous independent studies have been conducted to assess COPC concentrations in sediments and the distribution of white sturgeon ELS within the UCR, there are no data that evaluate the potential toxicity of these sediments to white sturgeon ELS. Given the epibenthic nature of white sturgeon, the potential exists for exposure of ELS of this species to contaminated sediments within the UCR. Specifically, there is concern that white sturgeon ELS (including the hiding stage) can be affected by the potential toxicity of COPCs associated with sediments containing granulated slag. In addition, potential uncertainties associated with the relative sensitivity of white sturgeon during the 15 to 50 dph critical life-stages remain and must be addressed.

A7 DATA QUALITY OBJECTIVES, CRITERIA, AND DESIGN RATIONALE

EPA's seven-step data quality objective (DQO) process (USEPA 2006b) was used to guide the design rationale for the 2010 sturgeon ELS studies. The DQO process is a tool to determine the type, quantity, and quality of data that are needed to address specified risk questions. This process establishes performance and acceptance criteria for the data to promote achievement of study goals.

A7.1 Step 1—State the Problem

White sturgeon is a species of special interest in the UCR ecological risk assessment because of three factors: 1) its poor recruitment over the past decades; 2) it may be sensitive to metals; and 3) its epibenthic habit potentially increases the likelihood of exposure to metals through contact with sediments. The third factor may be especially true during the early hiding stage of white sturgeon fry where they live in close proximity to sediments.

Poor recruitment of white sturgeon in the Columbia River between Grand Coulee Dam and Hugh L. Keenlyside Dam in Canada has been documented since the 1970s. While both spawning of adults and occurrence of embryos and larvae have been reported frequently during the past years, limited embryos and larvae have been captured in nets set from the U.S.-Canadian border to Evans, Washington (Howell and McLellan 2006).

Furthermore, juveniles (9 to 10 months old) that have been released as part of the White Sturgeon Recovery Plan appear to have good survival, growth rates, and body condition (Howell and McLellan 2006). Therefore, survival of one or more stages during the early development of larval and juvenile sturgeon appears to be limiting recovery of the population (Howell and McLellan 2006).

This study seeks to evaluate if COPCs associated with granulated slag in sediments in the UCR Site present an unacceptable risk to the survival and growth of white sturgeon during the first 2 months of life. This QAPP focuses on the sediment pathway; however, a limited number of acute water-only laboratory exposure studies will be conducted during certain life-stages (15 to 50 dph) before and after the transition to feeding as described in Appendix B.

A7.1.1 Team Members and Roles

One of the goals of Step 1 of the DQO process is to establish a planning team and identify decision makers. The planning team will consist of personnel from ENTRIX and U of S, and will be supported by the Teck technical team. A detailed overview of the specific personnel and their roles is provided in Section A4.2.

A7.1.2 Resources and Timelines

To conduct the work identified in this QAPP, toxicity testing will be conducted at the Aquatic Toxicology Research Facility of the Toxicology Centre at the U of S. Details are provided in Section B1.

Timing of toxicity tests will be dictated by the spawning season of white sturgeon in the Columbia River, which is expected to take place sometime between June and July (the only window when embryos are available for testing). Controlled laboratory studies to be conducted at the Aquatic Toxicology Research Facility will include ~60 day subchronic toxicity testing of sturgeon fry with sediments collected in the vicinity of Deadman's Eddy, China Bend, and Marcus Flats in Washington; and at a reference site (e.g. Birchbank[2]) and a laboratory control sediment.

[2] At the time of writing, the exact reference sediment has not yet been confirmed (i.e. neither the reference site nor the laboratory control sediment) as it is dependent on observations and findings outlined within the draft QAPP entitled *Methods Development for Sediment Sampling and White Sturgeon Sediment Toxicity Studies* as submitted on March 3, 2010 (Teck 2010b). For the purposes of this document however, the upstream reference location will be assumed to be Birchbank, as it has historically served as a sediment bioassay reference.

In addition, a limited number of focused 96-hour acute copper toxicity tests with white sturgeon ELS and rainbow trout will be conducted to provide a level of replication based on work being completed by the USGS at their Columbia, Missouri, laboratory.

A7.1.3 Pilot Studies to Inform Work Described in this QAPP

At the time this document was written, some of the information required to make definite decisions regarding the study design were not available. This information is being developed under a separate QAPP entitled *Methods Development for the White Sturgeon Sediment Toxicity Study* as approved on April 26, 2010 (Teck 2010b). Test conditions that are being evaluated as part of the methods development work are listed in Table A-1. Results from the method development work will be presented to EPA in a technical memorandum. Agreement on the study design changes, if any, will be documented in an amendment to this QAPP.

A7.2 Step 2—Identify the Goal of the Study

There are two goals associated with this work. The primary goal of the study is to determine if COPCs associated with granulated slag in UCR sediments are chronically toxic to white sturgeon ELS (including the hiding stage). A secondary goal is to refine our understanding of acute toxicity of copper at critical life-stages relative to a standard test organism (rainbow trout) in water. The latter will be accomplished in the form of 96h acute toxicity tests at two critical life-stages with white sturgeon and rainbow trout. The water and sediment studies are both part of an ongoing multiple lines-of-evidence approach for assessing the potential toxicity of granulated slag COPCs in UCR matrices to white sturgeon ELS. Specific risk-related questions that will be addressed during the studies are:

- Are there significant differences in acute and/or subchronic effects on survival, growth, and biomass on white sturgeon ELS raised on Site and reference sediments? If significant differences occur
  - What is the magnitude of these effects?
  - Are these effects due to slag-associated COPCs as measured in sediments, porewater, and overlying water?
- What is the relative sensitivity of white sturgeon ELS compared to rainbow trout as determined in acute toxicity tests with copper at 15 and 45 dph? (Additional details presented in Appendix B).

A7.2.1 Testable Null Hypotheses

Null hypotheses to be evaluated in independent studies will provide the following conclusions on white sturgeon ELS at the UCR Site.

1. There are no statistically significant differences in survival, and growth of white sturgeon ELS reared on Site and reference sediments.
2. There are no dose-dependent differences in survival, and growth of white sturgeon ELS as a function of COPC concentrations in sediments from the Site and at the point of exposure.
3. There are no life-stage specific and dose-dependent toxicities after acute exposure to copper under controlled laboratory conditions; moreover, the sensitivity to acute exposure of white sturgeon ELS is no different than that of rainbow trout. (Additional details are presented in Appendix B).

A7.3 Step 3—Identify Information Inputs

Step 3 of the DQO process (USEPA 2006b) requires consideration of the following:

- The types and potential sources of information (e.g., site characteristics or variables) that should be measured to provide estimates or resolve decisions
- Information to provide a basis for specifying performance or acceptance criteria
- Information on the performance of appropriate sampling and analytical methods.

The decision process regarding the white sturgeon toxicity assessment will be supported by analytical measurements detailed in Table A-2.

In addition to the analytical measurements, determination of biological endpoints in white sturgeon ELS during the sediment exposure studies will include:

- Fry/juvenile mortality—NOAECs and LOAECs
- Growth (length and mass)—NOAECs and LOAECs.

Determination of biological endpoints in white sturgeon ELS during the acute exposure studies with copper will include:

- Fry/juvenile mortality—NOAECs, LOAECs, and LC50s
- Growth (length and mass).

Developmental and behavioral abnormalities will also be observed daily during the sediment toxicity and the acute copper toxicity tests.

A7.4 Step 4—Define the Boundaries of the Study

This step specifies the population of interest for the study, the geographical boundaries of the site, and any temporal considerations that may be required.

A7.4.1 Target Populations for Risk Evaluation

Target populations of interest for risk evaluation are white sturgeon ELS from the parent population living in the Columbia River between Hugh Keenleyside Dam in Canada and Grand Coulee Dam in the U.S.

A7.4.2 Geographic Boundary of the Site

The Site encompasses the UCR from the U.S.-Canadian border (River Mile [RM] 745) to the Grand Coulee Dam (approximately RM 596). However, with the distribution and occurrence of white sturgeon limited to areas upstream and including Marcus Flats (Howell and McLellan 2006), sediment toxicity tests using white sturgeon ELS will be limited to sediments collected upstream of RM 700 (Marcus Flats). In addition and based on sediment data collected to date (Johnson et al. 1988; Bortleson et al. 2001; Era and Serdar 2001; Majewski et al. 2003; Cox et al. 2005; USEPA 2003,2006a; Paulson et al. 2006), elevated sediment COPC concentrations associated with granulated slag have been identified within this portion of the Site.

A7.4.3 Temporal Considerations

The temporal boundaries of the studies are defined by the reproductive cycle of white sturgeon. Typically, spawning of white sturgeon in the UCR occurs sometime in June or July depending on temperature and hydraulic conditions. Thus, it is anticipated that exposure chambers will be operational at the end of the first two weeks of June, so that the test can be initiated as soon as fertilized sturgeon eggs are available in June/July.

A7.5 Step 5—Develop the Analytical Approach

Risks of exposure of white sturgeon ELS to UCR sediments will be assessed in a deterministic fashion using a multiple lines-of-evidence approach: 1) exposure of white sturgeon ELS to sediments collected in the Site and reference samples, and 2) comparison of metal concentrations measured in porewater and overlying water to chronic ambient water quality criteria.

The first line of evidence will be derived from a controlled study with sediments collected in the UCR and reference sediments. Long-term (> 60 days overall exposure time), exposures of white sturgeon larvae, fry, and juveniles to these sediments will be tested in fluvial flow-through systems. The tests will begin with newly hatched fry and

continue to 60 dph. Effects on survival, growth, and development (external morphology) will be measured and recorded. Details on the experimental design are provided in Section B1.4[3]. If the testing reveals significant effects from exposure to sediments collected downstream of the U.S.-Canadian border vs. reference samples, then the data will be reviewed to determine if the results can be explained by the presence of COPCs in porewater or water overlying sediments. This will be determined by the presence of a relative response, the statistical vigor of that response, and the correlation of exposure to the response (i.e., dose response curve).

All data generated from the herein described toxicity tests will be submitted to the EPA for review (see Section C-2).

A7.6 Step 6—Specify Performance and Acceptance Criteria

Specifying limits on decision errors involves defining the possible decision errors and the consequences of making these errors. Typically, this is done by describing the decisions in terms of hypothesis tests or other objective decision criteria and by specifying the hypotheses to be tested using an appropriate statistical model. Limits can also be specified by identifying the decision errors as false-positive and false-negative errors. In this study, the target value for the type I error (the false positive decision error; α) will be set at 0.05. The target value for the type II error (the false negative decision error; β) will be set at 0.2.

Sediment collection, transport, and storage conditions will meet or exceed requirements listed in:

- ASTM (American Society for Testing and Materials). 2009. *Standard guide for collection, storage, characterization, and manipulation of sediments for toxicology testing and for selection of samples used to collect benthic invertebrates.* E1391-03 (2008). Annual Book of ASTM Standards, Volume 11.06, ASTM International, West Conshohocken, PA.

Detailed descriptions regarding the collection, transport, storing and processing of sediments are provided in SOPs 4 and 8. Details on performance criteria for the sediment exposure assessed in the subchronic studies are provided in Section B1.4 In

[3] At the time of writing, the experimental design has not been finalized as it is dependent on observations and findings outlined within the draft QAPP entitled *Methods Development for Sediment Sampling and White Sturgeon Sediment Toxicity Studies* as submitted on March 3, 2010 (Teck 2010b).

brief, these criteria include acceptable ranges for mortalities in the control sediment treatments, which are not to exceed 20 percent during a specific life-stage with the exception of the transition-to-feed life-stage, during which mortalities up to 50 percent can naturally occur (target value for mortality during this life-stage is not to exceed the upper 95 percent confidential interval of mortality reported for the same batch of fish cultured at the Kootenay Trout Hatchery, Fort Steele, British Columbia, Canada). Overall, mortality in the control sediment treatments should not exceed 36% for the duration of the exposure (i.e., survival should be at least 64%). Initial seeding densities for all treatment chambers will ensure that fish density will not confound overall survival rates. Coefficients of variation for mortalities among replicate chambers of the same treatment group are not to exceed 20 percent.

A7.6.1 Optimized Study Design

Details concerning how data will be collected for assessing the potential toxicity of COPCs present in sediments to white sturgeon are provided in Section B and the SOPs associated with this QAPP (Appendix C). These methods will be tailored to the physical and logistical constraints associated with obtaining the most effective endpoint measurements. The remainder of this section describes the methods that will be used to determine sample sizes needed to meet the objectives of the sediment exposure study.

The method that will be used to calculate statistical power of the studies to be conducted is the hypothesis testing method. This method uses estimates of variance (S^2) to determine the optimal sample size (n). One of the key parameters in determining the required sample size is the relative difference to be demonstrated. The relative difference (relative to the mean) is the property that affects sample size. To demonstrate differences with the same power, a larger sample size would be required to demonstrate a relative difference of 5 percent of the mean than to demonstrate a 20 percent difference. Furthermore, the ability to demonstrate a particular difference relative to the mean is dependent on the variance of the population. As an example, a greater sample size would be required to demonstrate the difference between two means when the coefficient of variation is 40 percent than when it is 20 percent.

Statistical power of the proposed study design was calculated using the "Means Routine" of the PASS software of NTCC (NTCC, Kaysville, Utah). This method allows the specification of the level of type II error (β). It determines the required number of samples (replicates of the basic experimental unit, which in this study is the replicate exposure system) necessary to achieve adequate statistical power in testing the null hypothesis, H_0, using a fixed significance level α (i.e., the type I or false-positive error)

and a fixed, specific alternative hypothesis, H_1. This fixed alternative may be for hypothesis tests involving a single mean (e.g., comparing the mean number of mortalities for one sample to COPC concentrations) or for problems involving two means (e.g., comparing the mean mortality between two treatment groups). The key to this method is determining the relative magnitude of difference to be demonstrated, relative to the variance of the mean or means. Thus, it is the relative magnitude that is important, not the absolute difference between means. To effectively use this method, in addition to selecting values for the probability of committing type I and type II errors, the method requires estimates of the variance of each population as well as the magnitude of difference to be demonstrated for a particular mean value.

Based on the above method and the information collected during previous unpublished surface water toxicity studies (means and variance), the number of replicate groups required to detect significant differences among sites for α values of 0.05 and 0.1 and β values of 0.2 and 0.1 were calculated (Table A-3). Based on these calculations, three or more replicate groups provide sufficient power to detect 10 percent or greater increases in overall mortality among treatment groups. Given the logistical constraints of the studies proposed herein, it was decided to use three true replicate groups, which should permit detection of a 10 percent increase in mortality with a power of greater than 80 percent and an α value of 0.05. However, it should be noted that the above calculations of power are based on variances and other information obtained during studies with aqueous exposure media, and thus, may not completely apply to the tests with natural sediments proposed here.

Based on previous experience with the culture and exposure of white sturgeon ELS (Tompsett et al., submitted for publication), it is expected that there will be naturally elevated mortalities during the transition to exogenous feeding. Therefore, several statistical approaches will be used to evaluate the data and to determine dose-response relationships (if any), with associated LCx [4] (survival) and ECx [5] (growth and development). Data will be aggregated for the entire exposure period of > 60 days and also will be stratified by key life-stages, yolk sac larvae, transition to feeding, and juvenile. Thus, separate LC/ECx endpoints can be calculated for life-stage periods of interest such as 0 to 21 dph, 29 to 50 dph, and for the entire exposure period.

[4] Lethal concentration for "x" percent of the test population.

[5] Effective concentration for "x" percent of the test population.

Additionally, comparisons among treatment and control groups (e.g., using analysis of variance) will compensate for transition-phase mortality because all groups are assumed to have at least as much mortality as the control groups; therefore, data can be statistically adjusted to account for this period of decreased survival. However, over the entire course of the continuous exposure, control survival should be greater than or equal to 64% (in accordance with the sturgeon Level of Effort document) and control survival should be greater than or equal to 80% in each of the 0-21 and 28-56 day posthatch exposure periods. The 0-21 and 28-56 day posthatch periods are meant to represent the endogenous and exogenous feeding stages; actual days are approximate.

A7.7 Step 7—Develop the Study Plan

Detailed discussions of the various study components are presented in Section B1 of this QAPP.

A8 SPECIAL TRAINING/CERTIFICATES

The project manager is responsible for assembling a project team with the necessary experience and technical skills. Part of the process is to identify special training requirements or certifications necessary to execute the project successfully. Project-specific requirements include training specific to the analytical methods to be conducted, handling, and health assessment methods for white sturgeon larvae, usage of the flow-through exposure systems, and health and safety training for personnel engaged in on-site and laboratory activities.

All project personnel will receive training before commencing work to ensure they are familiar with the required SOPs (Appendix C) and safety and emergency procedures and are adequately skilled at collecting data and operating and maintaining the exposure system. Personnel training records will be maintained by the laboratory manager.

In addition, all personnel working at the Site should have the appropriate health and safety training identified in the General Site Health and Safety Plan (Teck 2009a) and U of S laboratory personnel will follow the laboratory safety guide (Appendix D). Field sampling will follow the approved Cultural Resource Plan (Appendix E).

A9 DOCUMENTATION AND RECORDS

This section identifies on-site and laboratory records to be maintained for this project, information to be included in project reports, data reporting format for data report packages, and document control procedures to be used.

A9.1 Required Records

Critical records required for this project are identified below with descriptive or supporting information as appropriate. The records will include documents and electronic deliverables related to field sampling (field notebook, sample logs, COC, etc.), toxicity testing and chemistry lab documentation (lab records, data packages, project reports, electronic deliverables, etc.), data validation, and data reports and other applicable project reports.

A9.1.1 Field sampling

Documentation is described in SOPs 2 and 6 and summarized here.

- Field notebooks (hardcopy).
- Field sampling forms.
- Photographs.
- COC records.
- Corrective action reports.

A9.1.2 Toxicity Testing

Documentation is described in SOPs 4 and 12 and summarized here.

- Sample receipt dates.
- Exposure system maintenance logs.
- Equipment maintenance and QA logs.
- Laboratory notebooks.
- Photographs.
- Raw data sheet.

A9.1.3 Chemistry Laboratory

Documentation is described in Appendix F and will include in the electronic data documentation package, at a minimum, the following items.

- Sample receipt and analysis dates.

- Final analyte concentration including reporting limit, laboratory qualifiers, and reanalyses.
- Percent recovery of each compound in the matrix spike sample.
- Matrix spike recovery control limits.
- Relative percent difference (RPD) for all matrix spike/matrix spike duplicate (MS/MSD) and/or laboratory control sample (LCS)/LCS duplicate (LCSD) results.
- RPD control limits for MS/MSD and/or LCS/LCSD reports.
- Laboratory control sample results when analyzed.
- Recovery control limits for LCS or standard reference material recoveries and relative standard deviation.
- Blank results for method blanks, experimental blanks, and equipment blanks.
- Method blank summary indicating associated samples.
- Case narrative.

A9.1.4 Data validation

For data validation, the following additional data will be reported:

- Calibration information, including initial calibration, concentration response data of the calibration check standards, continuing calibration check data, instrument tunes, and associated samples.

All raw data and logs will include the following information:

- Analyst's initials and date
- Initial and final sample and extract volumes or weights and/or dilutions
- Condition of instrument
- Documentation linking sample analysis to instrument calibration (where appropriate)
- Analysis start time of all experimental and quality control samples
- Instrument run log showing analytical sequence
- Dilutions performed and amount of sample analyzed
- Experimental samples, quality control samples, and blanks clearly labeled
- Quantification reports
- Sample preservation (where applicable).

Paper copies of all these records will be retained. In addition, data will provided as: 1) an electronic deliverable in Microsoft Excel format for all test results, and 2) an electronic backup for all on-site and laboratory data generated.

Procedures for project control, archiving, and storage of laboratory records are described in Section B10 of this QAPP.

A9.2 Project Reports

The task manager will prepare summary reports for investigations described herein as required under the terms of the Agreement.

A9.3 Record Maintenance and Storage

All documents relating to the project will be controlled to ensure proper distribution, filing, and retrieval, and to ensure that revisions are properly recorded, distributed, and filed.

Project records will be stored and maintained by ENTRIX. The task manager and office staff are responsible for organizing, storing, and cataloging all project information and for collecting records and supporting data from project team members. Once project records are cataloged, ENTRIX will ensure that the project records are appropriately filed by category in the correct project file. Filed documents will be available to U of S and ENTRIX staff through checkout procedures developed to ensure the integrity of the project file. Individual project team members may maintain separate files or notebooks for individual tasks. These files or notebooks will be transferred to the task manager as part of project closeout. The archived files will be stored and maintained by ENTRIX. Newly created documents will be transmitted to Teck quarterly in accordance with its document retention policy. Additional information on record management can be found in Section B9 of this QAPP.

SECTION B: DATA GENERATION AND ACQUISITION

This section describes all aspects of measurement, design, and implementation and discusses the methods that will be used for sampling, analysis, data handling, and quality control in support of the studies that will be conducted in 2010 to assess the potential toxicity of sediments from the UCR to white sturgeon ELS. These studies will include the following experiments:

- Chronic sediment exposure studies with field-collected sediments from a reference location in Canada (e.g., Birchbank) and three locations in the UCR (i.e., Deadman's Eddy, China Bend, and Marcus Flats)
- Water-only acute copper toxicity studies (96-hour exposures) at 15 to 45 dph (refer to Appendix B for additional details).

The approach is designed to collect data that supports characterization of the nature and extent of potential sediment-related toxicity to white sturgeon ELS from hatch through 60 dph, as well as to augment the database on acute toxicity from water-only exposures.

The following specific aspects of measurement and data acquisition are discussed in this section:

- Sampling process design and rationale
- Sampling method requirements
- Sample handling and custody requirements
- Analytical method requirements
- Quality control requirements
- Instrument/equipment inspection and maintenance requirements
- Instrument calibration and frequency
- Acceptance requirements for supplies and consumables
- Data management.

B1 SAMPLING PROCESS DESIGN AND RATIONALE

B1.1 Test Species—Numbers, Source, Strain, and Life-Stages

Species: White sturgeon (*Acipenser transmontanus*)

Strain: Offspring from wild sturgeon caught in the Columbia River

Age: Fry, and juveniles

Number: 25,000 eggs from 2 to 4 breeding pairs

Source: Kootenay Trout Hatchery, Fort Steele, British Columbia, Canada

Freshly fertilized white sturgeon eggs from at least two breeding pairs from the Columbia River will be obtained from the Kootenay Trout Hatchery in British Columbia. Fertilization of eggs will be harmonized in the hatchery by injecting adult riverine sturgeon with a gonadotropin analog on 2 subsequent days. Eggs will be transported to the exposure facilities between 4 and 12 hours after fertilization. Arrangements with the Kootenay Trout Hatchery will be made to retain a contingent of fish from the same fertilization event at the hatchery (~3,000 fry) as a backup if mortality rates in the controls are too great to be able to reinitiate studies at a later life-stage.

B1.2 Sampling Locations and Rationale

Four locations between the trans-boundary reach and Kettle Falls (upstream of RM 700) will be sampled (see Maps A-2 and A-3), representing a sediment exposure gradient for slag-associated COPCs such as cadmium (Cd), copper (Cu), lead (Pb), and zinc (Zn). Specifically, these locations include: 1) Deadman's Eddy (RM 737), 2) China Bend (RM 723), and 3 & 4) Upper and Lower Marcus Flats (RM 706 and 704) (Table B-1). In the event that sediment samples cannot be retrieved from the aforementioned areas, due to unspecified reasons, alternative sampling locations have been identified. These include Northport and the Little Dalles (Table B-1). As illustrated in the above-referenced maps, the proposed locations not only represent a gradient of slag-associated COPCs between locations but also within locations.

In addition to a gradient of slag-associated COPC sediment concentrations within the proposed locations, the greatest abundance of white sturgeon ELS (larvae) has been reported in the vicinity of Little Dalles and China Bend downstream of Northport, which represent an important nursing ground for sturgeon. The reference location (Birchbank) is located approximately 12 km upstream of the city of Trail and Teck's Trail smelter (Table B-1).

Within each of the four primary sampling areas (Deadman's Eddy, China Bend, Upper and Lower Marcus Flats) there will be three sites to capture potential local gradients of COPC concentrations.

The rationale for the selected locations is as follows:

1. Reference site—It is located upstream of the Trail smelter facility and has historically been used as a reference for a wide range of environmental studies (e.g., sediment bioassays and surface water quality).
2. Deadman's Eddy (RM 737)—This site is located downstream of a known spawning site within the Columbia River (Waneta, British Columbia, Canada) and, as such, there is the potential that white sturgeon ELS would be exposed to these sediments. In addition, preliminary toxicity tests on white sturgeon ELS (i.e., 30 dph) have been performed by the USGS (2009) using sediment from this area.
3. China Bend (RM 723)—Early larvae abundances appear to be greatest upstream and at this location (MacLellan and Howell 2006). Thus, it is assumed that later life-stages such as fry during the hiding stage will be present at and downstream of this site.
4. Marcus Flats (RM 706)—Marcus Flats has been hypothesized to serve as the farthest downstream sink for slag-containing sediments and as a nursing ground for white sturgeon.

B1.3 Support Facilities for Chemical Analysis

The primary laboratory for analysis of samples for COPCs will be Columbia Analytical Services, Kelso, Washington, USA. Non-COPC measurements (e.g., dissolved oxygen, ammonia, pH) will be monitored by the Aquatic Toxicology Research Facility of the Toxicology Centre at the U of S during the course of the work to maintain appropriate biological conditions in the exposure chambers.

B1.4 Experimental Setup and Sampling Strategy

The following sections describe the experimental set-up and design anticipated during the work. It is important to note however, that specific details presented below are subject to change based on observations and findings outlined within the draft *Methods Development for Sediment Sampling and White Sturgeon Sediment Toxicity Studies* QAPP (Teck 2010b). Observations recorded during the methods development work will be summarized in a technical memorandum submitted to EPA for review and concurrence; and will serve as an addendum to this QAPP.

B1.4.1 Sediment Sampling, Transport, and Storage

A total of approximately 50 gallons of sediment per sediment sampling site (or less, depending upon final fluvial system design as determined during methods development) will be needed for the exposure studies (Table B-2). Samples will be collected in accordance with protocols described in Appendix C. After collection, sediments will be stored and transported immediately to the test facilities using decontaminated 5-gallon resealable polyethylene buckets. Transport will occur at approximately 4°C, and samples will be stored at the same temperature until use in experiments.

B1.4.2 Chronic Sediment Toxicity Studies (U of S)[6]

Exposures to UCR sediments will be conducted in specifically designed flow-through fluvial simulation systems at the Aquatic Exposure Laboratory at the U of S (Figures B-1 and B-2). Prior to adding sediments to the exposure chambers, sediments from each sampling site that were stored separately in 5-gallon buckets will be mixed and homogenized in a commercial grade concrete mixer (SOP-8) or a >50-gallon polyethylene barrel by means of thorough mixing/tumbling (SOP-8). Homogenization of a sediment will be assessed by visual observations, including uniformity of color and consistency (as determined in methods development work). Sediments then will be evenly layered in exposure chambers at a thickness of 5.08 cm (2 inches), or as otherwise determined during the methods development work. A sample for bulk sediment chemistry analysis as specified in Tables B-3 and B-4 will be taken from each replicate exposure chamber. Eight to 12 suction devices (e.g., airstones) that span the entire width of the exposure chambers will be distributed at 4- to 3-inch intervals throughout each chamber to enable porewater sampling during the experiments without disturbing the sediment (number and exact placement to be determined during the methods development work). Diffusive membrane devices (such as "peepers", diffusive gradient thin-film (DGT) probes, or other similar diffusive devices) may also be deployed for collection of porewater and/or sediment-water interface water, depending upon the results of methods development work. After addition of sediments to each chamber,

[6] The procedures described herein and design elements are "envisioned" based on the current state of knowledge, and may not represent the final conditions and design under which the tests will be conducted. Definite conditions will be established based on the current pilot activities as described in the draft QAPP entitled *Methods Development for Sediment Sampling and White Sturgeon Sediment Toxicity Studies* as submitted on March 3, 2010 (Teck 2010b).

test systems will be slowly filled with artificial river water (laboratory water that was adjusted for hardness, pH, dissolved organic carbon [DOC] and alkalinity similar to that observed in the UCR [Teck 2009b]; SOP-13). On top of the sediment layer collected from the river and placed into exposure chambers, medium size gravel (1 to 2 cm diameter) will be placed at a density of ~5 to 7 stones per 100 cm^2 to improve habitat in accordance with earlier observations (Little et al. pers. comm.), with size and distribution optimized during methods development. Water flows and velocities will be determined during methods development, but will be less than those occurring at some of the upstream locations in the river to ensure proper sturgeon culture. Prior to initiation of exposure studies, all systems will be equilibrated for a minimum of 4 weeks, or as otherwise determined during methods development, although timing of sediment collection will ultimately dictate the available timeframe. Although water in the UCR may move through the sediments either from upwelling from deeper sediments, downward movement from surface water, or lateral fluvial flow, it is not possible to recreate these conditions within the laboratory exposure chambers.

Exposures of white sturgeon will begin immediately after hatching at the Aquatic Exposure Laboratory at the Toxicology Centre at the U of S; and will continue through approximately day 60 post-hatch. Eggs will be incubated in McDonald-type incubation jars in artificial river water. After hatching, larvae from the different breeding pairs will be combined and randomly assigned to treatment chambers. Sturgeon will be sampled at day 1 and at termination of the 60-day exposure period (Figure B-2). Number of individuals per treatment and replicate group and general test design are provided in Table B-5.

Exposure of sturgeon fry and juveniles will be conducted in a facility that allows control of light and temperature regimes. Each sediment sample group and the controls/reference will be tested in three replicate exposure systems (Figure B-1). Each exposure system is temperature controlled. All criteria meet or exceed requirements listed in ASTM guidelines for testing fish ELS (Table B-6; ASTM 2005). Summaries of test conditions for conducting the work, the general activity schedule, and the test acceptability requirements are provided in Tables B-7 through B-9, respectively. Fish densities for specific life-stages during the exposure period were calculated based on optimum seeding densities for this species under fluvial conditions (Tompsett et al., submitted for publication).

B1.5 Sample Types

Samples to be collected during the duration of this work are listed in Table B-10 and sample analytical methods are listed in Table B-11. In general, two types of samples will be taken during the studies described herein:

1. Samples for exposure assessment. Three different matrices will be sampled for assessment of exposure

    a. Overlying water—Water samples will be collected at weekly intervals during the course of the exposure studies. Water will be taken initially (for the first two weeks) at the inflow and outflow of the test chambers, and will be tested at the U of S for DOC, alkalinity, hardness, total suspended solids (TSS), pH, dissolved oxygen (DO), conductivity and temperature. If no significant differences between the initial samples taken from inflow and outflow occur, only outflow samples will be taken during the remainder of the study.

    b. Porewater—Porewater will be collected at the same intervals as overlying water using suction devices (see Section B1.4.2). Samples will be taken at multiple locations within each treatment chamber, and will be preserved for dissolved metal analysis, cation, anion, DOC, and other parameters listed in Table B-10 using the methods listed in Table B-11, as appropriate for application of the biotic ligand model (BLM) to the data.

    c. Sediment-water interface water – Water at the interface of the sediment surface and overlying water will be collected by either suction, by means of a "peeper" or a similar sampling device, as determined during the methods development studies. Sampling will occur at the same intervals as pore- and overlying water, and samples will be subjected to the same analysis as porewater.

    d. Sediment—Prior to initiation of the studies, COPC concentrations in whole sediments will be measured, and sediment samples will be characterized for grain size, organic carbon content, acid volatile sulfides (AVS), simultaneously extracted metals (SEM), target analyte list (TAL) metals, and other parameters as appropriate (e.g., pH).

2. Samples for biological analysis. Following measurements of length and biomass (weight), all sturgeon will be preserved at intervals/life-stages specified above to

ensure, if necessary, later analysis of gross and histological morphological alterations (fixation in 10 percent buffered formalin).

B1.6 Study Contingencies

The study design is intended to meet the DQOs and satisfy the data needs of the RI/FS and ecological risk assessment specific to white sturgeon. It is possible that complications may arise due to mortalities in the controls (> 50 percent during transition to feeding). The following is a description of possible contingencies or alternative approaches to be followed in the event control mortalities are unacceptably high (i.e., exceed 25 percent for yolksack larvae or 50 percent for post-swim-up stage). Use of contingency plans will be approved by EPA prior to implementation.

Contingency #1—Maintain backup batch of fish at hatchery

Arrangements with the Kootenay Trout Hatchery will be made to retain a contingent of fish from the same fertilization event at the hatchery (~3,000 fry) as a backup if mortality rates in the controls are too great to be able to reinitiate studies at a later life-stage.

Contingency #2—Obtain fertilized eggs/fry from possible second spawning event

In past years, there often has been more than one spawning event at Waneta, and in such occasions ripe females and males were collected and transported to the Kootenay Trout Hatchery at different time points for stripping and fertilization of eggs. In case there would be a second later spawning event at the hatchery in 2010, a potential second batch of eggs could be obtained for reinitiation of the experiments.

B2 SAMPLING METHOD REQUIREMENTS

B2.1 Sample Processing and Laboratory Analyses

For water quality and COPC analyses conducted at CAS, current EPA analytical methods for analysis of dissolved TAL metals, conventional parameters, and major ions will be used, in addition to *Standard Methods for the Examination of Water and Wastewater* (SM) (APHA 1998), as indicated in Table B-11. All sample processing procedures will be entered into the appropriate forms, and dated and initialed by the person who took the sample.

Water Quality Analysis for Fish Culture. Overlying and porewater quality will be measured a the U of S Aquatic Toxicology Research Facility as part of the daily/weekly

laboratory routine to ensure that water quality is appropriate for culture of fish (e.g., temperature, pH, dissolved oxygen, conductivity, hardness, alkalinity, ammonia; Table B-10)

Sediment Characterization. Sediment samples will be analyzed by CAS for TAL metals, pH, total organic carbon (TOC), AVS, SEM, and grain size at the initiation and end[7] of the study. All sediments will be stored appropriately after termination of the experiments to enable later analyses for additional COPCs if this should be deemed necessary.

Water Samples for COPC Analysis. Overlying and porewater samples for analysis of major cations and anions, dissolved TAL metal, and general water quality parameters (alkalinity, hardness, DOC, TDS) will be taken weekly and preserved as specified in Tables B-8 and B-9 for shipment to CAS. All procedures, including transport of samples to the analytical laboratory for analysis, will be recorded on the COC forms that will accompany the samples at all times. All methods for sample preparation and fixation will be in accordance with EPA methods on a performance basis.

Biological Samples. Subsets of white sturgeon ELS will be sampled throughout the course of the studies (Figure B-2). All samples will be measured and weighed immediately after collection, and observations of any behavioral and morphological abnormalities will be recorded on the appropriate forms (Table B-12). Behavioral observations will follow ASTM E1711-95 (2008). Also, sturgeon of an age >50 dph will be dissected and morphological anomalies (e.g., tissue color, liver size, gut/stomach filling) will be recorded. After 24 to 48 hours, depending on the size of the individual, the sample will be transferred into ethanol, and stored at room temperature. All sturgeon that die prior to termination of the experiments will be subjected to the same procedures as described in this paragraph.

B2.2 Sampling Documentation

Study team members at the Aquatic Toxicology Research Facility will maintain bound logbooks to provide a daily record of significant events, observations, and measurements during sampling and routine experimental maintenance procedures. Each data book will have a unique identifier and each page and carbon copy will include

[7] Grain size will not be determined at the end of the experiment.

this data book identifier. All information pertinent to sampling will be recorded in the logbooks. Each day's logbook entries will be signed and dated and will include:

- Name and title of author, date and time of entry, and experimental conditions during the activity (e.g., water quality parameters, health status of test organisms)
- Activities performed (e.g., water renewal, feeding of larvae)
- Sampled matrix
- Sample collection method
- Number of samples taken.

When activity-specific data forms are used, they will also include:

- Project name and number
- Treatment identifier
- Initials
- Analysis and sample collection method.

The following information will be recorded either in the logbook or on the activity-specific data forms:

- Date and time of collection
- Sample identification number(s)
- Sample destination (e.g., laboratory)
- Laboratory observations
- Experimental measurements
- Experimental handling (preservation).

All original data recorded in experimental logbooks, data forms, sample labels, and COC forms must be written with waterproof, indelible ink. None of these accountable, serialized documents are to be destroyed or discarded, even if one is illegible or contains inaccuracies requiring document replacement. If an error is made on an accountable document assigned to one individual, that individual will make all corrections simply by crossing a line through the error, initialing and dating the correction, and entering the correct information. The erroneous information will not be obliterated. The person who made the entry will correct any subsequent error discovered on an accountable document. All personnel will be trained in the proper use of notebooks prior to the start of the studies.

B2.3 Sample Identification

The analysis and sample identity information will be recorded in bound logbooks or recorded on data sheets while in the custody of the sampling team.

A sample label will be completed and attached to each sample container for every individual or composite sample collected. Labels consist of a waterproof material backed with a water-resistant adhesive. Labels are to be filled out using waterproof ink, and are to contain at least the following information:

- Sampling date and time
- Sample identification number
- Sampler's initials
- Sample matrix or matrix identifier.

Each sample to be analyzed for residues (COPCs) will be assigned a unique number consisting of an alphanumeric code that identifies the treatment group and sample type. These numbers will be tracked electronically, from collection through laboratory analysis and into the final reports.

The sample number will be cross-referenced with the treatment group name on the COC. Additional sample volume will be collected for samples identified for laboratory QC purposes (i.e., MS, MSD, DUP) and identified as "For Lab QC Use." Information to be included on COCs is specified in SOP-8 (Appendix C), titled "Sample Management: Receiving, Preservation, Storage, Documentation, Decontamination, and Disposal."

B2.3.1 Tissue Sample Handling Procedures

Appropriate sample containers will be sealed and labeled. In cases where tissue samples will be archived, samples will be placed on wet or blue ice in an insulated container for no longer than 30 minutes, and then stored appropriately (e.g., buffered formalin). Appropriate COC documentation will accompany the samples as required by the QAPP. Specific sample volumes, sample containers, preservatives, and replication of samples are detailed in the following sections. Any sampling equipment that will be reused will be decontaminated by rinsing with deionized water followed by dilute acid (e.g., nitric acid) between sampling.

B2.3.2 Decontamination Procedures and Materials

All equipment used during investigation activities that could come into contact with chemically affected materials will be thoroughly cleaned, before and after each use, by

washing with Liquinox® (a laboratory-grade detergent) and rinsing with deionized water followed by dilute acid (generally, nitric acid). Decontamination procedures may be modified and/or revised based upon the data obtained or the equipment used.

Decontamination waste is expected to consist of dilute acid. Decontamination solutions will be properly disposed through the Department of Health, Safety and Environment (DHSE) of the U of S.

B2.4 Sampling/Measurement Failure Response

If quality control surveillance and/or experimental audits result in detection of unacceptable conditions, procedures, or data, the task manager, in conjunction with the quality assurance manager, will be responsible for developing and directing implementation of corrective actions. Corrective actions will include one or more of the following:

- Identifying the root cause of the problem and implementing systems to prevent future occurrences
- Identifying the source of the violation
- Evaluating and amending sampling and/or analytical procedures
- Accepting data and flagging the data to indicate the level of uncertainty associated with failure to meet the specified quality control performance criteria.
- If necessary, studies will be restarted at a selected life-stage, by agreement with EPA.

Any finding requiring corrective action must be documented by the task manager on a corrective action form (Figure B-3). The project quality assurance manager will check to ensure that corrective actions have been implemented and that the problem has been resolved. Problems will be addressed and the corrective action noted in the appropriate notebook.

If an error is made on an accountable document assigned to one individual, that individual will make all corrections simply by crossing a line through the error, initialing and dating the correction, and entering the correct information. The erroneous information will not be obliterated. The person who made the entry will correct any subsequent error discovered on an accountable document.

B2.5 Sample Preservation and Holding Time Requirements

The sample containers, preservative requirements, and maximum holding times for analytical methods for this project are provided in Tables B-8 and B-9. All containers for samples submitted for chemical analyses will have screw-type lids to ensure adequate sealing. Pre-cleaned bottles will be provided by the analytical chemistry laboratory.

B3 SAMPLE HANDLING AND CHAIN-OF-CUSTODY REQUIREMENTS

Proper sample handling, shipment, and maintenance of COC are key components of building the documentation and support for data that can be used to make project decisions. It is essential that all sample handling and sample COC requirements be performed in a complete, accurate, and consistent manner. Sample handling and custody requirements must be followed for all samples collected as part of this project.

B3.1 Sample Custody

Sample custody and documentation procedures described herein must be followed throughout all sample collection activities. Components of sample custody procedures include the use of experimental logbooks, sample labels, custody seals, and COC forms. The COC form must accompany the samples during shipment from the collection sites to the Aquatic Toxicology Research Facility at the U of S (SOP-6 in Appendix C) and from the Aquatic Toxicology Research Facility at the U of S to the contract laboratory (CAS) (SOP- 7 through SOP-9 in Appendix C).

Samples will be delivered to the designated laboratories by the experiment personnel, laboratory courier, or by commercial shipping services (such as UPS or Federal Express). The method of sample shipment will be noted on the COC. During the experimental effort, the experiment team leader or a designee will inform the laboratory daily of planned shipments. Hard plastic ice chests or coolers with similar durability will be used for shipping samples. The coolers must be able to withstand a 4-ft drop onto solid concrete in the position most likely to cause damage. The samples will be packed to prevent the least amount of damage if such a fall were to occur.

A sample is under custody under the following conditions:

- It is in one's actual possession
- It is in one's view, after being in his or her physical possession

- It was in one's physical possession and that person then locked it up to prevent tampering
- It is in a designated and identified secure area.

The following procedures must be used to document, establish, and maintain custody of samples:

- A sample label will be completed and attached to each sample container for every sample collected. Labels consist of a waterproof material backed with a water-resistant adhesive. Labels are to be filled out using waterproof ink, making sure that the labels are legible and affixed firmly on the sample container. Sample labels are to contain at least the following information: sampling date and time; sample identification number; treatment group identifier; and sampler's initials.
- All sample-related information must be recorded in the project logbook or on activity-specific data forms.
- The sampler must retain custody of samples until they are transferred or properly dispatched.

To simplify the COC record and minimize potential problems, as few people as possible should handle the samples or other physical evidence. For this reason, one individual from the experiment crew should be designated as the responsible individual for all sample transfer activities. This investigator will be responsible for the care and custody of the samples until they are properly transferred to another person or facility.

A COC record will accompany all samples. This record documents the transfer of custody of samples from the investigator to another person, to the laboratory, or other organizational entities; a signature for relinquishment and receipt of the samples must accompany each change of possession. The COC record will be prepared for groups of samples collected at a given location on a given day.

The COC form makes provision for documenting sample integrity and the identity of any persons involved in sample transfer. Information entered on the COC will consist of the following:

- Project name and number
- Logbook number
- COC serial number
- Treatment group

- Sample numbers
- Sampler/recorder's signature
- Date and time of collection of each sample
- Sample type
- Analyses requested
- Inclusive dates of possession
- Name of person receiving the sample
- Date of receipt of sample
- Name, address, and telephone number of laboratory
- Name, address, and telephone number of person to whom laboratory report will be sent
- Method of delivery and courier.

Completed COC forms will be inserted into a resealable plastic bag, sealed, and taped to the inside cover of the shipping container used for sample transport from the experimental site to the laboratory, or from the laboratory to the analytical laboratory, when a courier or shipping company is used. The shipping company will not sign for custody of the samples.

When samples are relinquished to a courier for transport, the tracking number from the shipping bill or receipt must be recorded on the COC form or in the site logbook.

The recipient for the samples must be notified of the date of shipment and anticipated time of arrival. The shipping bill number must also be provided to the recipient to enable tracking of samples. It must be clearly established prior to shipment who will be responsible for ensuring that timely sample delivery occurs and who will track the samples in case of shipping delays. The recipient of the samples must inform the sender when the samples are delivered. Custody seals must be affixed on shipping containers or individual sample containers[8] when samples are shipped to the laboratory to prevent sample tampering during transportation. In cases of delivery delay or packing damage,

[8] Samples that are shipped to Canada from the United States, or from Canada to the United States, are subject to custom inspection which may break the seal on the shipping container. Therefore, sample containers must be sealed individually.

all details of damage and sample condition must be recorded and, if necessary, photographed for documentation.

B3.1.1 Laboratory Sample Handling and Custody

This section is applicable to all samples sent to the U of S laboratory or to CAS. The task manager or field team leader will notify the laboratory project manager of upcoming sampling activities and the subsequent transfer of samples to the laboratory. This notification will include information concerning the number and type of samples to be shipped, analyses requested, and the expected date of arrival. The laboratory project manager will notify appropriate laboratory personnel about the expected shipment including the sample custodian.

Upon arrival at the laboratory, the samples will be received and logged in by a trained sample custodian in accordance with the laboratory's sample handling program. A description of the laboratory's general program is provided in SOP-7 through SOP-9 and is summarized below.

Upon sample receipt, the sample custodian is responsible for performing the following activities during sample receipt where appropriate:

- Examining the shipping containers to verify custody seals, if used, are intact
- Examining all sample containers for damage
- Taking digital photographs of any custody seals used, before opening, and of any damage to the shipping container or individual sample containers
- Comparing samples received against those listed on the COC
- Verifying sample holding times have not been exceeded
- Determining sample temperature (from the temperature blank vial) and documenting variations from the acceptable range on the COC
- Verifying that all samples listed on the COC are present or accounted for
- Immediately signing and dating the COC after shipment is accepted
- Noting any sample receipt problems on the COC, initiating a condition-upon-receipt (CUR) report, and notifying the laboratory project manager
- Attaching laboratory sample container labels with laboratory identification number and test
- Placing the samples in proper laboratory storage.

The laboratory project manager is responsible for contacting the project liaison as soon as possible if any problems are identified during sample receipt. All identified sample receiving problems will be resolved before sample preparation and analysis.

Following sample receipt, the sample custodian is responsible for logging the samples in the laboratory sample log-in book, and/or the Laboratory Information Management System with the following information:

- Laboratory project number
- Sample numbers
- Type of samples
- Required tests
- Date collected
- Date received.

The sample custodian is also responsible for notifying the laboratory project manager and appropriate group/team leader(s) of sample arrival and placing completed COCs, waybills, and any additional documentation in the project file.

Samples will be stored appropriately within the laboratory to maintain any prescribed temperature, to protect against contamination, and to maintain the security of the samples.

B4 ANALYTICAL METHOD REQUIREMENTS

This subsection presents the analytical method requirements for chemical analyses that will be performed during the study including preparation/extraction procedures, where appropriate, and method performance requirements.

Analysis of samples for COPCs will be conducted by CAS according to the methods indicated in Table B-11. The laboratory's quality assurance protocols are in Appendix F and contain summary information about the analytical methods used, including the following (see also Tables B-3, B-4, and B-13 of this QAPP):

- Holding times
- Sample containers and preservatives
- Calibration requirements and acceptance criteria

- Laboratory quality control samples including acceptance criteria and corrective action
- Limits of detection.

More detailed information on the laboratory's analytical methods is presented in laboratory-specific SOPs, to be provided in Appendix F.

B4.1 Analytical Methods

B4.1.1 Biological Assessments

Prior to initiation of the exposure experiments, hatchability will be assessed by dividing the number of successfully hatched eggs by the total number of eggs seeded in each egg incubation jar. Results will be presented as percent hatchable eggs and used to evaluate test acceptability (see Tables B-7 and B-9). The following endpoints will be measured in the chronic sediment exposure test at the U of S Aquatic Toxicology Research Facility:

Mortality. Mortality data will be reported as the percentage of dead fry/juveniles compared to original seeding density. In addition, life-stage specific mortality will be calculated by dividing the number of fish at the end of a specific life-stage period by the number of fish present at the beginning of the same life-stage. Dead fish will be discerned from live ones by immobility (e.g., in response to gentle prodding) and absence of respiratory movement in older individuals.

Growth. Growth will be determined by measuring length and weight of the fish. All dead fish will be measured when removed from the tanks. A sample of newly hatched fry will be taken from the hatching tank and weighed (in groups of ten) and measured (individually) to document size at start of the test. Photo documentation will be used to measure length of live fish throughout the duration of the experiment, measuring the fish to the nearest 0.5 mm if permitted by resolution of photograph. At the end of the test, all fish will be weighted to the nearest 0.01 g and measured to the nearest 0.5 mm; individuals will be blotted dry prior to determining weight. Growth will be reported as weight and length gain over time.

Other observations. Throughout the experiment, fish will be monitored for alterations in behavior (Table B-12; ASTM E1711-95 (2008)). Furthermore, animals will be inspected for gross morphological alterations (e.g., fin aberrations, skeletal deformities) at the time of sampling. All of the individuals removed from the experiments will be fixed in formalin.

B4.1.2 Chemical Analysis

A summary of the chemical analyses to be performed during this work is presented in Table B-10. As indicated within the table, routine water quality measurements to ensure proper fish culture will be conducted by the U of S Aquatic Toxicology Research Facility; while COPC measurements and associated measure of metal bioavailability (e.g., BLM parameters) will be determined by CAS. To determine the reporting limit goals, available water and sediment guidelines and benchmarks were compiled and compared to the expected reporting limit (Table A-2). For aquatic ecological receptors, reporting limit goals were developed using the EPA national aquatic life chronic criteria (USEPA 2006c).

Screening values and required method reporting limits (MRLs) for samples collected during the 2010 sturgeon ELS studies are provided in Table A-2. The goal is for MRLs to be equal to or below one-fifth of the EPA national aquatic life chronic criteria (USEPA 2006c) for each analyte. MRLs are generally equivalent to the concentration of the lowest calibration standard (i.e., the practical quantification limit) and represent the low end of the calibration range. Analytes that are detected at concentrations below the reporting limit but above the detection limit will be reported, but will be qualified as estimated (i.e., a "J" qualifier will be applied to the result by the laboratory).

Laboratory methods for sample preparation and analysis are summarized in Table B-11. Sample containers, preservation, and holding times are provided in Tables B-8 and B-9.

B4.2 Laboratory Corrective Action

Laboratories have formal corrective action systems in place to ensure that prompt action is taken when an unplanned deviation from a procedure or plan occurs and that, whenever possible, corrective actions include measures to prevent the reoccurrence of deviations. Specific corrective actions will be taken and documented when a quality control sample does not meet acceptance criteria. Following is a description of how information from the laboratory's corrective action system is communicated to the project team.

Corrective action procedures include prompt notification of the project contact (quality assurance manager) for any significant problems or discrepancies. The laboratory project manager is responsible for reporting any significant problems or discrepancies that occur as analyses are conducted to the project liaison or other identified project contact. The laboratory project manager is also responsible for ensuring that corrective action is taken, where appropriate, to prevent the reoccurrence of similar problems or

discrepancies. In addition, each analytical data report will include a case narrative that discusses any problems or discrepancies, and sufficient calibration and quality control information to verify that the method was within control limits at the time that the samples were analyzed. The case narrative will also include a discussion of any corrective action taken by the laboratory to prevent the reoccurrence of similar problems or discrepancies.

B5 EXPERIMENTAL QUALITY CONTROL REQUIREMENTS

This section presents the quality control checks that will be performed by the analytical (chemistry) laboratory during investigations including a discussion of quality control samples with frequency and acceptance criteria and corrective action procedures.

B5.1 Quality Control Samples

Quality control samples for equipment and field blanks will be prepared at CAS and shipped with the sample containers to the U of S exposure facility and the field crew, respectively, for monitoring the bias and precision of the sample collection and analysis procedures. The type and frequency of quality control samples to be collected during investigations are summarized in Table B-13 and are described below.

B5.1.1 Equipment Rinsate Blank Samples

Equipment rinsate blanks (ERBs) are samples of weak acid (e.g., 1 percent nitric acid) passed through and over the surface of decontaminated sampling equipment. The rinsate is collected in sample bottles, preserved, and handled in the same manner as the samples. ERBs are used to monitor the effectiveness of the decontamination process. The planned frequency for ERBs is one per week per equipment type. If more than one type of equipment is used to collect samples for a particular matrix, then an ERB is collected and submitted for each representative group of equipment. Typically, ERBs are analyzed for the same analytes as the corresponding samples collected that day.

B5.1.2 Blanks

Two types of field blanks will be used: 1) sample containers filled with DI water that are transported to and returned from the experimental location unopened and 2) sample containers that are opened at the experimental location, filled with DI water, and then closed. Typically, at least one blank per lot number of collected samples will be analyzed.

B5.1.3 Duplicate (Blind) Samples

"Blind" duplicate samples are collected to monitor the precision of the sampling process. The use of replicates to assess precision is discussed in Section B5.2.1. Experimental duplicates of overlying water in the exposure chambers will be collected weekly and submitted to the laboratory for analysis. However, given the limits in volume that can be taken for porewater and sediment-water interface water analysis, no duplicate samples will be collected during porewater sampling events in the laboratory.

B5.1.4 Certified Reference Materials

Certified reference materials are purchased by the analytical laboratory from nationally recognized, reputable vendors that have fulfilled the requirements of ISO 9001 certification and/or are accredited by A_2LA. These standards ensure the accuracy of the analytical measurements. Further details are provided in Appendix F *Quality Assurance Manual of Columbia Analytical Services*.

B5.1.5 Reference Toxicant

Sensitivity of the fish from the Kooteney hatchery will be assessed through the use of a reference toxicant in water-only exposures (see Appendix B).

B5.2 Method Performance Objectives

Method performance requirements for analytical laboratory methods are expressed in terms of precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS). Summarized below are brief definitions for each PARCCS parameter, with calculation equations as appropriate.

B5.2.1 Precision

Precision is an estimate of the variability between individual measurements of the same physical or chemical property under prescribed similar conditions.

Experimental Precision

Experimental precision is usually assessed through the collection and measurement of duplicate samples from each treatment. The duplicate sample is submitted "blind" to the laboratory, and sample results are compared to check for the overall variability introduced by sampling and analytical procedures. The experimental duplicate approach is generally not applicable to systems where the experimental unit is the single organism because each individual represents a sampling replicate. Similarly, when a

single test solution sample is collected and divided into additional blind samples, these replicate samples represent analytical replicates.

Analytical Precision

Precision in the laboratory is assessed through the calculation of the RPD for two replicate samples. The precision of the analysis can be inferred through the use of one of the following: 1) standard reference materials (SRMs) and duplicate SRM (SRMD) samples; 2) MS/MSD samples, which are project samples spiked with known analyte concentrations; or 3) duplicate analyses of unspiked project samples. The laboratory analyzes one or more of the aforementioned types of duplicate samples at a rate of one per batch of 20 or fewer investigative samples per matrix.

The MS/MSD samples provide information about the effect of the sample matrix on extraction and measurement methodology. An MS/MSD pair will be analyzed at a rate of one per 20 per analytical batch or fewer investigative samples per matrix.

Calculating the RPD for each pair of duplicate analyses (e.g., MS/MSD, LCS spike duplicates, unspiked duplicate samples) and the RPD for experimental duplicate sets using the following formula will assess the precision of laboratory analyses:

$$RPD = \frac{S - D}{(S + D)/2} x100 \qquad \text{(Eq. B5-1)}$$

Where:

RPD	=	Relative percent difference
S	=	First sample value (original or MS value or larger of the duplicate)
D	=	Second sample value (duplicate or MSD value or smaller of the duplicate)

B5.2.2 Accuracy

Accuracy is the degree of agreement between a measurement or observation and an accepted value.

Experimental Accuracy

Experimental accuracy is assessed through the collection and analysis of appropriate experimental blanks, and achieved through adherence to all sample handling, preservation, and holding time requirements. Experimental blank samples are analyzed to check for procedural contamination that may cause sample contamination. Equipment rinse blanks are used to assess the adequacy of decontamination of sampling

equipment between collections of individual samples. Accuracy of instruments will be assessed by using weekly instrument calibration and calibration checks. Experimental blank and equipment rinsate blank analysis frequencies are given in Table B-13.

Analytical Accuracy

Laboratory accuracy is assessed by the analysis of method blanks and matrix spikes, LCS, and/or SRM or certified reference materials. The results are expressed as percent recovery. Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Surrogate compounds are used in analyses for inorganic contaminants and are specified in the analytical methods described in the 2008/2009 surface water study QAPP (Teck 2009b).

Method blanks, matrix spike, LCS, and/or SRM samples will be analyzed at a rate of one per analytical batch of 20 or fewer investigative samples/matrix.

The percent recovery (percent R) of spike samples will be calculated using the formula:

$$R = \frac{A - B}{C} x100 \qquad \text{(Eq. B5-2)}$$

Where:

R	=	Recovery (percent)
A	=	The analyte concentration determined experimentally from the spiked sample, units
B	=	The background level determined by a separate analysis of the un-spiked sample, units
C	=	The amount of the spike added, units.

B5.2.3 Representativeness

Representativeness is a qualitative measure of the degree to which sample data accurately and precisely represent a characteristic experimental condition. Representativeness is a subjective parameter and is used to evaluate the efficacy of the study plan design. Representativeness is demonstrated by providing full descriptions of the sampling design and its rationale in the project planning documents.

There cannot be a target numerical goal for a qualitative parameter such as representativeness or comparability. Therefore, this criterion is completed and evaluated subjectively rather than quantitatively. The measure for representativeness is answered during the preparation of the sampling and analysis approach and rationale, and then reassessed during the data usability process. For example, an integral part of developing

the sampling and analysis approach and rationale is to answer the question "How many samples are needed to fully evaluate x?" Then, during the data usability process, the question "Were enough data collected to answer the original question?" must be answered. Thus, it is not possible to construct a table with numerical goals that can be used to evaluate these subjective measures. The criteria to make these decisions can be based on power analysis conducted after initial information has been collected or during data interpretation to determine if additional samples are necessary to fully describe the nature and extent.

B5.2.4 Comparability

Comparability expresses the confidence with which one data set can be compared with another data set obtained during parallel or previous investigations. Comparability can be related to precision and accuracy because these parameters are measures of data reliability.

Results are generally considered comparable if the same procedures for collecting and analyzing the samples are employed, if the samples comply with the same QA/QC procedures, and if the units of measurements are the same.

The study protocols for the determination of biological effects for this study were designed such that the data obtained during these studies are comparable with data collected during previous studies as outlined in Section A5 where applicable. Furthermore, comparability will be assessed by the parallel assessment of three true replicates for each sediment in the experiments.

The quality objective for data from the study's exposure experiments and analytical tasks is to achieve a level of comparability that allows for the comparison of data collected within and among all experiments. To accomplish this goal, all data generated during the tasks included in this investigation will be subject to strict QA/QC procedures as specified in this QAPP. Furthermore, comparability will be assessed by including a separate control using laboratory water in the field. Key water quality parameters known to influence availability and toxicity of metals and/or that are of importance for larval development and growth will be adjusted in the acute toxicity studies for comparability reasons. These parameters include temperature, pH, hardness, and dissolved oxygen.

B5.2.5 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was planned to be obtained under normal conditions. Data completeness will be calculated by using Equation B5-3.

$$\%\,\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \text{x}100 \qquad \text{(Eq. B5-3)}$$

Experience on similar projects has shown a reasonable goal considering combined historical field and laboratory performance is 90 percent completeness. All valid data will be used. During the data validation process, an assessment will be made of whether the valid data are sufficient to meet project objectives. If sufficient valid data are not obtained, the project manager will initiate corrective action. Where invalid data are generated, all documentation and the reasons for the invalidation of the data will be provided.

B5.2.6 Sensitivity

Sensitivity is the measure of the concentration at which an analytical method can positively identify and report analytical results. The sensitivity of a given method is commonly referred to as the detection limit. Although there is no single definition of this term, the following terms and definitions of detection limits will be used for this project:

- **Instrument Detection Limit.** Defined as the minimum mass of analyte that can be measured above instrument background noise under ideal conditions.
- **Analytical Detection Limit.** Method detection limits (MDLs) are statistically derived and reflect the concentration at which an analyte can be detected in a clean matrix with 99 percent confidence that a false positive result has not been reported. The laboratory conducting the analysis will determine a method detection limit for each analyte, as required by USEPA (2004). The analytical laboratory will have established MRLs at levels above the MDLs for the task analytes. These values are based on the laboratory's experience analyzing environmental samples, reflect the typical sensitivity obtained by the analytical system, and represent the level of analyte above which concentrations are accurately quantified. Analyte concentrations for this study will be reported to the MDL. Analytes detected at concentrations between the MRL and the MDL will be reported with a "J" qualifier to indicate that the value is an estimate (i.e., the analyte concentration is below the calibration range). Nondetected

values will be reported at the MRL and will be adjusted by the laboratory as necessary to reflect sample dilution or matrix interference.

B6 EQUIPMENT INSPECTION AND MAINTENANCE REQUIREMENTS

Maintenance and inspection of both experimental and analytical equipment are described in the following sections.

B6.1 Experimental Instruments/Equipment

Preventive maintenance of instrumentation and equipment will be performed according to manufacturer's instructions. The laboratory staff is responsible for ensuring that all instrumentation is operating properly prior to use. If problems are encountered, they will be documented in a bound notebook. The faulty instrumentation/equipment will be scheduled for repair and sequestered and tagged until repaired and qualified for reuse.

B6.2 Analytical Instrument/Equipment

Analytical instrument/equipment testing, inspection, and maintenance will be conducted in accordance with the procedures specified in the manufacturers' directions. The quality assurance manual discusses the schedule, procedures, criteria, and documentation in place at the laboratory to prevent instrument and equipment failure and to minimize downtime. For each instrument or piece of equipment, the laboratory maintains the following:

- Instrument/equipment inventory list
- Instrument/equipment major spare parts list or inventory
- External vendor service agreements (if applicable)
- Instrument-specific preventive maintenance logbook or file.

The laboratory documents all preventive maintenance and repair for each instrument or piece of equipment in dedicated logbooks or files.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

Calibration and frequency of calibration of both experimental and analytical equipment are described in the following sections.

B7.1 Experimental Instruments

The experimental equipment that will need calibration is listed below:

- Water quality meters
- Balance
- Pipettes
- Pumps
- Flow meters.

Proper maintenance, calibration, and operation of each instrument will be the responsibility of experiment personnel assigned to a particular activity. All instruments and equipment used during the investigations will be maintained, calibrated, and operated according to the manufacturer's guidelines and recommendations. If an individual suspects an equipment malfunction, the device must be removed from service and tagged so that it is not inadvertently used, and the appropriate personnel notified so that a recalibration can be performed or a substitute piece of equipment can be obtained. An extra or backup meter will be available at all times to replace the inoperable unit.

Results of measurements performed using equipment that has failed recalibration will be evaluated. If the measurement results are adversely affected, the results of the evaluation will be documented, the data qualified appropriately, and the data users notified.

B7.2 Analytical Equipment and Instrumentation

All laboratory equipment and instruments used for quantitative measurements are calibrated in accordance with the laboratory's formal calibration program as described in the quality assurance manual. A summary of the laboratory instrument/equipment calibration program is presented in that manual. Detailed calibration procedures specific to each analysis are included in method-specific SOPs, which can be obtained from the laboratory.

Whenever possible, the laboratory uses recognized procedures for calibration such as those published by EPA or ASTM. If established procedures are not available, the laboratory develops a calibration procedure based on the type of equipment, stability, characteristics of the equipment, required accuracy, and the effect of operation error on the quantities measured. Equipment requiring only periodic calibration such as pumps,

balances, thermometers, and micropipettes are listed along with their respective calibration requirements in the quality assurance manual. Whenever possible, physical reference standards are used, which are associated with periodic calibrations such as weights or certified thermometers with known relationships to nationally recognized standards. Where national reference standards are not available, the basis for the reference standard is documented.

Other instruments that require initial and/or continuing calibration as a part of instrument usage are listed along with their respective calibration requirements in the quality assurance manual. Initial calibrations are verified and documented for each constituent by analysis of laboratory-prepared certified independent standard solutions.

All calibration standards will be obtained from either the EPA repository or a commercial vendor, and the laboratory will maintain traceability back to the National Institute of Standards and Technology. Stock standards will be used to establish intermediate standards and calibration standards. Special attention will be given to expiration dating, proper labeling, proper refrigeration, and prevention of contamination. Documentation relating to the receipt, mixing, and use of standards will be recorded in a laboratory logbook. All calibration and spiking standards will be checked against standards from another source, as specified in the methods and the laboratory quality assurance manual.

B8 ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

Supplies and consumables that may be used during the investigations include sample bottles, petri dishes for filter storage, hoses, filters, nitric acid, formalin, ethanol, materials for decontamination activities, potable water, deionized water, ASTM Type II water, MS 222, *Artemia salina* eggs, marine salts, worms, and water quality test kits. Project team members obtaining supplies and consumables are responsible for assuring that the materials obtained meet the required specifications, are intact and in good condition, are available in adequate supply, and are stored appropriately until use. Project team members will direct any questions or identification of any problems regarding supplies and consumables to the experiment team leader for resolution.

B9 DATA MANAGEMENT

Data management procedures will be established and applied during the investigations to record, document, track, and compile investigative data into an overall project database. Data generated during the investigations, as well as historical data, will be used to form the basis for conclusions and recommendations. Efficient utilization and comprehensive consideration of available data require that the data be properly organized for review. Organization of the data shall be planned prior to actual collection to ensure the generation of identifiable and usable data. This section contains procedures necessary to ensure the collection of sufficient data for accurate validation of raw data and transfer of validated data to the project data management system. This section also describes the operating practices to be followed by personnel during the collection and reporting of data.

B9.1 Purpose and Background

Data collected during the investigations will include analytical chemistry data from aqueous and sediment samples; data on survival, and growth; and observations of development and behavior of white sturgeon ELS in each treatment group. The data will be used to analyze sediment-related toxicity in the UCR stretch of interest to these life-stages, and the potential contribution of slag-related COPCs to any potentially occurring toxicity. Data will be collected, managed, and stored in a way that models the inherent structure of the data and facilitates its usage for the RI/FS.

B9.2 Data Recording

Observations made and measurements taken during toxicity testing experiments will be recorded using appropriate hard copy (e.g., data sheets or logbooks) or electronic formats (e.g., laboratory electronic deliverables). Data recorded in hard copy will be transcribed into electronic forms and proofed before use or integration with other data.

For the chemical analytical portions of the study, a variety of manually entered and electronic instrument data will be generated. Data will be manually entered into:

- Standard logbooks
- Storage temperature logs
- Balance calibration logs
- Instrument logs
- Sample preparation and analysis worksheets

- Maintenance logs
- Individual laboratory notebooks
- Results tables for conventional analyses (e.g., grain-size distribution, percent moisture).

All data manually entered into the laboratory information management system will be proofed at each laboratory prior to being released. All data collected from each laboratory instrument, either manually or electronically, will be reviewed and confirmed by analysts before reporting. A detailed description of procedures for laboratory data management, data review, and verification is provided in the laboratory quality assurance plans.

Both chemical analytical and biological laboratory data will be entered directly into the project database from the electronic data deliverable (EDD). EDDs for chemical analytical data will be checked against the hard copy with 10 percent QA/QC for all detected analytes and other data where appropriate. For all data collected at the U of S, ENTRIX will perform a comparison of electronic data with the hard copy report prior to submittal to ensure that the EDD and hard copy data are identical. The EDDs will be uploaded to the project database as Microsoft Excel and pdf files.

Toxicity testing and analytical chemistry data will be entered into the project database management system (DBMS). This database will facilitate the following processes:

- Tracking COC and sample identification data
- Reviewing and evaluating analytical data against project-specific QAPP criteria
- Producing data tables.

Data will be tabulated for evaluation and presentation in the investigation report. Copies of the original data records will be attached to the report as appendices.

B9.3 Data Validation

Data validation is an integral part of the quality assurance program and consists of reviewing and assessing the quality of data. Data validation provides assurance that the data are of acceptable quality as reported. Data usability is the determination of whether or not a data set is sufficiently complete and of sufficient quality to support a decision or action in terms of the specific DQOs.

Chemical Analytical Data. For validity of these types of data, the characteristics of importance are precision, accuracy, representativeness, comparability, and completeness.

The data validation process includes:

- Evaluating against blank criteria
- Evaluating against accuracy criteria such as holding times, surrogates, LCS, and matrix spikes
- Evaluating against precision criteria such as MS/MSDs, and experimental and analytical duplicates
- Confirming that data qualifiers are assigned appropriately
- Uploading sample data only to the central database.

Biological Data. Any data generated from biological measurements will be validated based on test acceptability criteria, precision, representativeness, comparability, and completeness.

The data validation process includes:

- Evaluating against reference values specific to this species of batch of fish (e.g., hatchery average values and confidence intervals for survival, and growth at specific temperature)
- Evaluating against test acceptability criteria such as temperature, DO, pH, etc.
- Evaluating against precision criteria such as measurement duplicates (e.g., dependent and/or independent confirmation of weight, length, etc.)
- Confirming that data qualifiers are assigned appropriately
- Uploading sample data only to the central database.

The data validation process is described more fully in Section D.

B9.4 Data Transformation

If data transformation is performed for this study, then conversion procedures will be described in detail in the associated technical report.

B9.5 Data Transmittal

Laboratory data will be uploaded to the project database in Microsoft Excel format. Laboratory log sheets will be provided in *.pdf format and uploaded into the project database. The electronic data will be reviewed for completeness and accuracy by the project liaison before uploading to the project DBMS.

B9.6 Data Analysis

Data analysis (e.g., computation of summary statistics, standard errors, confidence intervals) will be conducted for this project. Percent survival will be calculated based on number of fry/juveniles at the start of each exposure period of interest (i.e., at 0 dph or 21 dph or 29 dph, depending upon which day represents the start of an exposure period of interest). Specifically, data will be analyzed by both analysis of variance and regression approaches. After assessing the distribution of data and homogeneity of variance, data will be tested for statistically significant differences among treatment groups using parametric or non-parametric tests, as appropriate, to determine NOAECs and LOAECs. Statistical comparisons will be made among treatment groups and relative to the controls and reference sediments. Where appropriate, effects on mortality and growth parameters will be assessed using regression approaches such as Probit or Logit analyses (e.g., calculation of LCs). To separate potential effects and interferences due to increased mortalities during the transition to feeding life-stage, the data will be evaluated by considering the entire exposure period of >60 days stratified by key life-stages, namely yolk sac larvae, transition to feeding, and juvenile.

B9.7 Data Tracking

The project manager is ultimately responsible for all activities conducted during experimental activities, including data management with the exception of data generated by the external analytical laboratory CAS. The project manager has the authority to enforce proper procedures as outlined in this plan and to implement corrective procedures to assure the accurate and timely flow and transfer of data. The project manager will review the final data reports.

Data will be generated from the measurements made during the course of the experiments and during sampling and analysis activities. The generators of data will be responsible for accurate and complete documentation of data required under the task, and for ensuring that these data are presented to their supervisor in a timely manner.

The study team leader or his designees will be responsible for the day-to-day monitoring of data during the conduct of the experiments. They will ensure that data are collected in the format specified in this QAPP and then routed to ENTRIX to be placed in the project files at the end of the experimental activities. Original documents will be maintained in the ENTRIX central project file.

The study team leader will also be responsible for evaluating biological data. The study team leader or his designees will review biological data for accuracy and completeness.

The project manager will assure that representations of current experimental conditions are accurate and complete for each component of the study.

The analytical chemistry lab coordinator (Kris McCaig) will be responsible for the day-to-day monitoring of activities related to the generation and reporting of chemical data. Ms. McCaig ensures that samples are analyzed according to the specified procedures; that data are validated; and that the data are properly coded, checked for accuracy, and entered into the project data management system.

B9.8 Data Storage and Retrieval

A project file will be established for the storage of original data, historical data, written documents, and data collected or generated during the experiments. The format for the file will follow the central filing system procedure list, which consists of the following categories:

- Correspondence
- Budgets
- Contracts
- Experimental data
- General data
- Notes/comments
- Raw data
- Figures and maps
- Permits
- Paper and electronic copies of data collected—both paper and personal digital assistant (PDA) data
- Laboratory data and QA/QC documents
- Chains of custody
- Photographs
- Reports
- Schedules
- Background.

All materials will be dated, carry the initials of the person responsible for the preparation of the document, and bear the project number. The file copies will include

peer review sign-off on the calculation sheets and editing review sheets where applicable.

Access to the project files will be limited to those personnel assigned to this project. The project manager will maintain overall responsibility for the project files and will ensure that appropriate documents are filed. All documents relating to the project shall be controlled to ensure proper distribution, filing, and retrieval. The project manager will also ensure that revisions are properly recorded, distributed, and filed. ENTRIX staff will maintain the project files.

ENTRIX staff will handle all documents submitted to the project file and will ensure that the documents are appropriately filed by category and placed in the correct project file. Once filed, documents are available to ENTRIX staff and may be removed from the file for use by signing out the material.

SECTION C: ASSESSMENT AND OVERSIGHT

This section presents the internal and external checks (assessments) that have been built into this project to ensure that

- Elements of this QAPP have been correctly implemented as prescribed for all investigations conducted
- The quality of the data generated is adequate and satisfies the DQOs that have been identified in this QAPP
- Corrective actions, when needed, are implemented in a timely manner and their effectiveness is confirmed.

Assessment activities may include surveillance, inspection, peer review, management systems review, readiness review, technical systems audit, performance evaluation, and data quality assessment.

C1 ASSESSMENT ACTIVITIES

The following subsections identify the planned assessment and oversight activities to ensure the objectives identified above are attained for experimental and analytical operations. The quality assurance manager and/or the project manager may also identify additional assessment activities to be performed during the course of the project based upon findings of the planned assessment activities described below.

C1.1 Assessment of Experimental Operations

The quality assurance manager and/or other designated members of the Aquatic Toxicology Research Facility project team will conduct internal assessments of experimental operations, where appropriate, at least once during the in-life portion of the test. The assessment activities will evaluate performance issues related to the experimental operations such as:

- Are sampling and monitoring operations being conducted in accordance with the QAPP?
- Are the sample labels being filled out completely and accurately?
- Are the COC records complete and accurate?
- Are the experimental notebooks being filled out completely and accurately?

- Are the sampling and monitoring activities being conducted in accordance with the SOPs?

Planned assessment activities to evaluate these and other performance issues include surveillance (frequent review) of sample collection documentation, sample handling records (COC forms), experiment notebooks, and study measurements, as well as the performance of unannounced experimental operations audits.

The team member conducting the assessment activity will report the results of any assessment activities to the project manager. Assessment activity reports will include the findings and identification of any corrective actions taken or planned.

C1.2 Assessment of Analytical Operations

The Chemical Laboratory Project Manager (Jeff Christian) will be in contact with the Analytical Chemistry Laboratory Coordinator (Kris McCaig) on a weekly basis while samples collected during this investigation are being analyzed. This will allow assessment of progress in meeting DQOs and the identification of any problems requiring corrective actions early in the investigative process. The Chemical Laboratory Project Manager will promptly report problems identified, corrective actions taken, and recommendations as appropriate for additional corrective action to the Analytical Chemistry Laboratory Coordinator who will review the problem and provide for the swift implementation of any outstanding corrective actions. In addition, contact between the Chemical Laboratory Quality Assurance Manager and the independent data auditor could result in the need for a laboratory audit. The Chemical Laboratory Quality Assurance Manager will report the audit findings and any recommendations for corrective action to the Analytical Chemistry Laboratory Coordinator, and the laboratory. The Analytical Chemistry Laboratory Coordinator will be responsible for working directly with the laboratory to ensure the prompt resolution of any problems identified.

C2 REPORTS TO EPA

As required by the Agreement, validated data will be provided electronically to EPA within 90 calendar days of completion of the study. This includes analytical chemistry results (sediment, porewater, overlying water) and biological observations (survival and growth), with a draft data summary report to follow shortly thereafter.

SECTION D: DATA VALIDATION AND USABILITY

Data generated at the laboratories will be verified and validated according to criteria and procedures described in this section. Data quality and usability will be evaluated, and a discussion will be included in the data validation report. Implementation of this section will determine whether the data conform to the specified criteria, thus satisfying the project objectives.

D1 DATA REVIEW, VERIFICATION, AND VALIDATION

Data validation is the process of reviewing data and accepting, qualifying, or rejecting data on the basis of sound criteria using established EPA guidelines. The laboratory will report laboratory data generated during the investigations in the form of data packages. All of these data will be subjected to full data validation conducted by an independent data validator as discussed below in Section D1.1.

D1.1 Independent Data Validation Protocols

All data generated for decisional purposes (i.e., to address the question of toxicity of sediments to sturgeon ELS) will be independently reviewed for completeness and accuracy as specified within this QAPP. Daily or weekly monitoring of water quality for maintenance of the exposure chambers will not be subjected to similar scrutiny as these measurements are intended only to keep the system within the specified limits.

D1.1.1 Aquatic Toxicology Research Facility

Biological data collected at the Aquatic Toxicology Research Facility at the U of S during the course of the study will be validated by Dr. Shaun Roark, ENTRIX QA Coordinator, or his designee. While the actual procedures used will be determined by the validator, the validation approach will consist of a systematic review of the analytical results, associated quality control methods and results, supporting data, and biological observations and measurements. Specific data package review procedures can be found in SOP-17, "Data Package Review," included in Appendix C. Best professional judgment will be used, as necessary, in any area not specifically addressed by EPA guidelines and described in the usability assessment portion of the data validation report.

D1.1.2 Columbia Analytical Services (CAS)

Mr. Rock Vitale and associates at ESI will be responsible for validating analytical data generated by CAS. Where applicable and/or appropriate, data will be validated

according to guidelines set forth in the following sources and guidelines to ensure compliance with the Federal Information Quality Act:

- SOP-17 Data package review (ETL-SOP#4039). Environmental Toxicology Laboratory, Toxicology Centre, University of Saskatchewan, Saskatoon, SK S7N 5B3
- USEPA. 1992. *Guidance for data usability in risk assessment (Part A)*
- USEPA. 2002b. *Guidelines for ensuring and maximizing the quality, objectivity, utility, and integrity of information disseminated by the Environmental Protection Agency*
- Federal Register, 67, No. 36, pp8451-8460. *Guidelines for ensuring and maximizing the quality, objectivity, utility, and integrity of information disseminated by Federal Agencies*, February 22, 2002
- USEPA. 2002c. *Guidance on environmental data verification and validation*.
- USEPA. 2004. *USEPA contract laboratory program national functional guidelines for inorganic data review*.

Data validations will include a data completeness check of each data package, a transcription check for sample results, and a thorough review of all laboratory reporting forms and the associated raw data for QA/QC issues. Specifically, this review will include:

- Review of data package completeness
- Review of the required reporting summary forms and all associated raw data to determine if the quality control requirements were met and to determine the effect of exceeded quality control requirements on the precision, accuracy, and sensitivity of the data
- Review of the overall data package to determine if contractual requirements were met
- Review of raw data and all calculations associated between 1 and a minimum of 10 percent of all samples to determine if the sample results and quantification limits were correctly calculated and reported
- Review of additional QA/QC parameters, such as blank contamination, to determine technical usability of the data
- Application of standard data quality qualifiers to the data.

In addition, each data validation will include a comprehensive review of the following QA/QC parameters:

- Holding times (to assess potential for degradation that will affect accuracy)
- Inductively coupled plasma/mass spectrometry (ICP/MS) instrument check (to assess accuracy and sensitivity of method)
- Initial calibration (to assess method sensitivity)
- Continuing calibration (to assess method sensitivity)
- Blanks (to assess contamination for all compounds)
- System monitoring compounds (to assess method accuracy)
- MS/MSD or laboratory fortified blanks (to assess accuracy of the methods and precision of the method relative to the specific sample matrix)
- Internal standards (to assess method accuracy and sensitivity)
- Target compound identification
- Compound reporting limit and MDL (to assess sensitivity as compared to project-specific requirements)
- System performance (to assess accuracy and precision).

D2 VERIFICATION AND VALIDATION METHODS

The data validation process is conducted to assess the effect of the overall experiment, sampling, and analysis process on the usability of the data. There are three areas of review: assessment of validity of biological measurements conducted, laboratory performance evaluation, and the effect of matrix interferences.

Biological measurements will be conducted in accordance with the procedures specified in the study protocols, and will be verified by means of randomized and blind (unknown sample ID) measurements. During sampling events, a randomly selected subset of fish (1 percent) will be measured twice by the analyst and the validator without either knowing the identity of the sample or the data recorded by the other. The results of these validation efforts will be recorded and reported in a data package and report. The Task QA Coordinator is responsible for ensuring that all required measurements are complete and valid (e.g., correct instruments were used to conduct the measurements, all instrumentation was properly calibrated, and test performance is within required guidelines).

Evaluation of laboratory performance is a check for compliance with the method requirements and is a straightforward examination. This evaluation will determine if the laboratory did or did not analyze the samples within the quality control limits of the analytical method and according to protocol requirements. The assessment of potential matrix effects consists of a quality control evaluation of the analytical results and also the results of testing blank, duplicate, and matrix spike samples, and then assessing how, if at all, the matrix effect will affect the usability of the data.

All bioanalytical data will be supported by a data package. The data package contains the supporting quality control data for the associated samples. The data validation report deliverables will include the following information:

- A comprehensive narrative detailing all quality control exceedances and explaining qualifications of data results. In cases where data are qualified due to quantifiable quality control exceedances, the bias (high or low) will be identified.
- Data summary tables in Microsoft® Excel format reporting all data results with the qualifiers that were added during the data validation review. These tables will include sample ID, laboratory ID, date sampled, sample type (e.g., experimental duplicate, experimental blank), units, concentration of analytes or biological measurements, and validation qualifiers. These tables may be modified to report other information as needed (such as date analyzed, dilution factor).
- Resubmittal requests sent to the laboratory indicating missing information and verification of analytical information, etc.
- EDDs compatible with the project database. These electronic deliverables will contain the validated results and qualifications as presented in the data summary tables of the validation reports. In addition, the validation reports can be submitted in electronic format for inclusion in interim remedial investigation data deliverables.

Before the laboratory releases each data package, the laboratory must carefully review the sample and laboratory performance quality control data to verify sample identity and also the completeness and accuracy of the sample and quality control data. This evaluation is performed through three levels of laboratory data review starting with 100 percent verification performed by the laboratory analyst, followed by a second-level review performed by a peer, supervisor, or designee. The laboratory project manager

performs the third and final laboratory review to assure that project requirements are met for the analyses performed.

Data validation is at times based on best professional judgment. In order to achieve consistent data validation, data worksheets will be completed for each data validation effort. A data review worksheet is a summary form on which the data validator records data validation notes and conclusions specific to each analytical method. The worksheets will help the validator to track and summarize the overall quality of the data. Sample results will then be qualified, as appropriate, following EPA protocols (EPA 2010). Samples that do not meet the acceptance limit criteria will be indicated with a qualifying flag, which is a one-letter or two-letter abbreviation that indicates a problem with the data (Table D-1).

The data verification process begins once the data packages for each project have been validated. During verification, the entire data set will be verified for overall trends in data quality and usability. Information summarized as part of the data quality verification will include percent variation between replicate biological measurements, frequencies of detection, dilution factors that might affect data usability, and patterns of target compound distribution. The data set also will be evaluated to identify potential data limitations or uncertainties in the laboratory. The trend analysis results will be included in the validation summary report, which will be submitted to the project manager at the end of the study efforts. The validation report and notes will be archived with the analytical data.

D3 RECONCILIATION WITH USER REQUIREMENTS

An assessment of the usability of the validated data compared to the data validation criteria and DQOs will be provided. The usability assessment will be performed in accordance with USEPA (1992) and best professional judgment. The data auditor will delineate major and minor deficiencies in the data, their effects on the reported results, and determination of usability for each compound reported in each sample included in the data package. The usability assessment will provide an overall summary of data quality. It will define acceptability or problems with accuracy, precision, sensitivity, and representativeness of the results and will provide clear guidance regarding uncertainties in the data that have been qualified as estimated (J) and a quantification of these uncertainties (e.g., bias high by a maximum of 80 percent), wherever possible. The data auditor may determine specific results to be unusable because of cumulative effects of quality control exceedances (i.e., an "R" qualifier will be applied to the result).

Alternatively, based upon the EPA guidelines and best professional judgment, the data auditor may determine specific results to be usable for DQOs when they are not significantly outside the quality control criteria.

The final activity of the data validation process is to assess whether the data meet the DQOs. The final results, as adjusted for the findings of any data validation/data evaluation, will be checked against the DQOs and an assessment will be made as to whether the data are of sufficient quality to support the DQOs. The decision as to data sufficiency may be affected by the overall precision, accuracy, and completeness of the data as demonstrated by the data validation process. If the data are sufficient to achieve project objectives, the project manager will release the data and work can proceed. If the data are insufficient, corrective action will be required.

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