Quality Assurance Project Plan

Assessing Sediment and Toxic Chemical Loads from the Green River, WA to the Lower Duwamish Waterway

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Table of Contents

List of Figures

List of Tables

Abbreviations and Acronyms

ADCP	Acoustic Doppler current profiler
ADVM	Acoustic Doppler velocity meter
BCM	Bed composition model
cPAH	Carcinogenic polycyclic aromatic hydrocarbon
CVO	USGS Cascades Volcano Observatory Sediment Laboratory
DOC	Dissolved organic carbon
DL	Detection limit
Ecology	Washington State Department of Ecology
EIM	Environmental Information Management
FNU	Formazin nephelometric unit
FFY	Federal fiscal year
HRMS	High-resolution mass spectrometry
LCS	Laboratory control sample
LDW	Lower Duwamish Waterway
MS	Matrix spike
NWIS	National Water Information System
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PSD	Particle size distribution
RL	Reporting limit
RKM	River kilometer
RPD	Relative percent difference
SLEDS	Sediment Laboratory Environmental Data System
SRM	Sediment reference material
SSC	Suspended sediment concentration
STM	Sediment transport model
TOC	Total organic carbon
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey
WAWSC	USGS Washington Water Science Center

Background

The Lower Duwamish Waterway (LDW) in Seattle, Washington is the site of intense current and historical anthropogenic influence, including numerous industrial, commercial, and residential uses. The land uses in the drainage basin include: residential (35 percent) such as the towns of South Park and Georgetown; industrial (18 percent) and commercial (11 percent) including marinas, boat manufacturing, concrete manufacturing, food processing, and airplane parts manufacturing; rights-of-way (18 percent) such as roads and highways; and open or undeveloped areas (17 percent) including parks. Decades of intense anthropogenic activities have resulted in contaminated sediments in the LDW. In 2001-2002, the U.S. Environmental Protection Agency (USEPA) and the Washington State Department of Ecology (Ecology) required remedial investigations and feasibility studies on the 5-mile, 441-acre LDW under the federal Superfund law and Washington's Model Toxics Control Act due to concern over human health risks from exposure to contaminated sediments. The main contaminants of concern for human health include polychlorinated biphenyls (PCBs), dioxins/furans, carcinogenic polycyclic aromatic hydrocarbons (cPAHs), and arsenic. The USEPA's final cleanup plan for the LDW was released in November 2014, and includes using combinations of dredging, capping, natural sedimentation and enhanced natural recovery.

To support the implementation of a cleanup plan of contaminated sediments in the LDW, sources of sediment to the site were evaluated. Three sources of sediment to the LDW were identified: upstream sources that are transported by the Green River to the LDW, lateral sources from land adjacent to the LDW, and re-suspended bed sediment within the LDW. The Sediment Transport Model (STM), developed for the LDW, predicts that every year more than 185,000 MT of sediment enters the LDW, and greater than 99 percent of that originates from upstream sources while approximately 0.5 percent originates from lateral sources and 0.2 percent originates from bed sediment within the LDW (LDWG 2008). There is substantial uncertainty in the average annual upstream sediment load because of large inter-annual variations in precipitation and sediment transport dynamics. The STM predicts that approximately 90 percent of the total bed area in the LDW receives 10 cm of new sediment within 10 years or less. Therefore, the sediment and contaminant transport and loading dynamics from the Green River to the LDW will determine, in large part, the sediment recovery potential of remediated areas in the LDW.

Research Problem and Objectives

Limited field data are available regarding sediment and contaminant transport and loading dynamics from the Green River to the LDW. The STM estimated suspended and bed sediment loading into the LDW from upstream sources using grain size information and a flow-rating curve for the Green River based on discharge data from 1960-1980 and 1996-1998. That physical model was then coupled with contaminant concentration data to create a Bed Composition Model (BCM). The upstream contaminant data was extrapolated from five historic data sets from King County, Ecology, and the U.S. Army Corps of Engineers. Only one of those data sets (Gries and Sloan, 2009) measured contaminants on suspended sediment (the other studies measured surface sediment or whole water). The sample size of the Gries and Sloan data set was relatively small (n=7) and samples were not collected during the rising limb of high flow events. The upstream data that were used in the BCM primarily originated from surface bed-sediment data, and those values were estimates of actual contaminant concentrations because the suspended sediment fraction was not fully represented. In addition, suspended sediment-associated chemical loadings are expected to vary over time as affected by a number of variables including precipitation, streamflow, seasonality, sediment organic carbon content and particle size distribution. Therefore, better estimates of annual sediment loading and toxic chemical loading from suspended sediment in the Green River to the LDW are needed.

The objective of this project is to quantify sediment and toxic chemical loads associated with upstream sources in the Green River to the LDW, including high flow/high turbidity events that may contribute more to the annual loading than average flow conditions. The activities described in this document are an expansion and continuation of USGS activities performed from 2013-2015 under previous USGS-Ecology agreements. Project information, including previous QAPPs and reports, are available at: http://wa.water.usgs.gov/projects/riverloads/. This QAPP includes all of the main elements of a complete Ecology QAPP as described in "Guidelines for Preparing Quality Assurance Project Plans for Environmental Studies (Ecology, 2004). These improved measurements will aid in assessing the potential for future re-contamination of remediated sediment in the LDW and will leverage ongoing U.S. Geological Survey (USGS) efforts to quantify sediment and chemical loading from large rivers to Puget Sound.

Organization and Timeline

The roles and responsibilities of key personnel involved in this project are provided in this section.

Mahbub Alam, Washington State Department of Ecology. Provide technical management of the project to ensure that activities are conducted in accordance with Department of Ecology guidelines and standards.

Kathy Conn and Bob Black, USGS Washington Water Science Center. Implement project objectives including coordination of field sampling, processing, transport of samples for physical and chemical analysis, and data retrieval. Ensure that the collected data are correctly loaded in EIM. Analyze data and provide interpretive findings to Ecology. Ensure that the project is conducted according to USGS guidelines and standards including quality assurance and quality control standards.

Fu-Shin Lee, Washington State Department of Ecology, Quality Assurance Specialist, Toxics Cleanup Program. Review sampling plan and data for adherence to Ecology quality assurance and control standards, including those required for input into the Environmental Information Management (EIM) database.

Joel Bird, Washington State Department of Ecology, Director, Manchester Environmental Laboratory. Oversee analysis of samples at Manchester and manage analytical chemistry contracts and agreements for remaining parameters. This includes the development of the Statement of Work, evaluation of bidding laboratories, and payment.

Ginna Grepo-Grove, Washington State Department of Ecology, Quality Assurance Manager. Provide EPA Level 4 validation of analytical data as described in Data Validation section.

Contract Laboratories and Consultants.
Ecology will contract with Washington State accredited laboratories for analytical chemistry not analyzed by Ecology. Ecology will coordinate the analytical laboratory contracts and agreements, and those analytical service costs are not included in the overall agreement between Ecology and the USGS. The USGS will utilize a USGS sediment laboratory for physical analysis of suspended sediment in water samples and a USGS research laboratory for mineral analysis of suspended sediment. The USGS will manage all data from both the USGS sediment lab and the contract analytical laboratory (see USGS Washington Water Science Center responsibilities below).

USGS Washington Water Science Center (WAWSC)
Kathy Conn, Water Quality Specialist
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The WAWSC will be responsible for overseeing the collection, transport, shipping, and interpretation of all physical and chemistry data related to this project. This includes water, suspended sediment, XAD-2 resin, and glass fiber filter samples. The WAWSC will also be responsible for payment of physical sediment analysis conducted by USGS labs. USGS analytical guidelines and quality parameters will be reviewed and compared for compliance and a data quality evaluation (see USGS Office of Water Quality Technical Memorandum 2014.01: http://water.usgs.gov/admin/memo/QW/qw2014.01.pdf) will be the responsibility of the WAWSC. The WAWSC will review all field and USGS lab data and conduct data analysis and report preparation. In addition, the publication and transmittal of all final reports and the long-term storage of data in Ecology (EIM) and USGS (NWIS) databases will be the primary responsibility of the WAWSC.

USGS Cascades Volcano Observatory Sediment Laboratory (CVO)
Dan Gooding, Laboratory Chief
1300 SE Cardinal Court,
Building 10, Suite 100
Vancouver, WA 98683
Phone: (360) 993-8917
FAX: (360) 993-8980
dgooding@usgs.gov
URL: http://vulcan.wr.usgs.gov/Projects/SedLab/framework.html

The CVO will be responsible for the analysis of water samples for physical characterization of suspended sediment, including particle size distribution (PSD) and suspended sediment concentration (SSC). SSC is a measure of the amount of sediment in a given volume of water, reported as milligrams per liter (mg/L). Water samples collected using two techniques will be compared: (1) from the bridge using depth- and width-integrated techniques that ensure the sample is representative of the river's entire cross-section (as the samples for water chemistry will be collected) and (2) from the bank using a pump from a point source (as the samples for suspended sediment chemistry will be collected). See the Field Sampling section for more details.

USGS National Research Program X-ray Diffraction Laboratory
Kate Campbell-Hay
3215 Marine St. Suite E-127
Boulder, CO 80303
Phone: (303) 541-3035
kcampbell@usgs.gov

Split samples of the glass-fiber filters and suspended sediment (when available) will be analyzed for mineralogical content by X-ray diffraction at the USGS National Research Program laboratory in Boulder, Colorado to determine the mineralogy of the suspended sediment to which contaminants are sorbed. Results will be transmitted in an electronic format for review by the USGS project manager, and will be released as part of the Data Release publication for long-term publicly-accessible storage.

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Ecology's Manchester Lab will be responsible for the chemical analysis of water, suspended sediment, glass fiber filters, and XAD-2 resin for all analytes listed in Appendix A. Analyses for organic carbon and inorganic compounds including low-level mercury will be performed at Manchester. The remaining analyses will not be performed at the Manchester Lab and will be performed by one or more contract laboratories (see below). Ecology's Manchester Lab will manage the analytical laboratory contract(s), which will include the development of the Statement of Work, evaluation of bidding laboratories, and payment for analytical services. The Manchester Laboratory will also be responsible for conducting laboratory data validation of contracted results comparable to USEPA Level 4 validation. See Data Validation section for more details.

Contract Laboratories

The contract laboratories will be responsible for the chemical analysis of water, suspended sediment, glass fiber filters, and XAD-2 resin samples as specified in the analytical contract or agreement between the laboratory and Ecology. The laboratory(s) will provide a designated project manager for direct communication with Ecology and the USGS. The laboratory will provide bottles, coolers, preservatives, filters, and chain of custody forms for each sampling event. They also will provide an USEPA Level 4 data package deliverable to the USGS and Ecology, which includes a summary narrative and raw data. The data also will be transmitted in an electronic format that is compatible with Ecology's EIM database.

The following contract laboratories will perform analyses not performed by Ecology's Manchester Environmental Lab:

AXYS Analytical, Ltd
Analysis of dioxins/furans and 209 PCB congeners
Mike Elliott – account manager, melliott@axys.com, (530) 521-8476
Georgina Brooks – project manager, gbrooks@axys.com
2045 Mills Road W.
Sidney, BC Canada V8L 5X2

Analytical Resources, Inc. (ARI)
Analysis of semivolatile compounds, low-level PAHs, and butyl tins
Mark Harris, project manager, markh@arilabs.com, (206) 695-6210
4611 S. 134th Pl., Suite 100
Tukwila, WA 98168-3240

King County Environmental Lab
Analysis of PAHs in water by large-volume injection (LVI)
Colin Elliott, project manager, colin.elliott@kingcounty.gov
322 W. Ewing St.
Seattle, WA 98119

The timeline for the project is shown in Table 1.

Table 1. Timeline of project tasks. [The Federal Fiscal Year (FFY) begins on October 1 and ends on September 30.]

Task	FY 2016	FY 2017		
	July-Sept	Oct-Dec	Jan-Mar	Apr-June
Gage Operation and Maintenance	🟦	🟦	🟦	🟦
Study Design and Preparation	🟦			
Water and Sediment Sampling	🟦	🟦	🟦	
Tidal Dynamics Monitoring	🟦	🟦	🟦	🟦
Data Review and Analysis	🟦	🟦	🟦	🟦
Report Preparation			🟦	🟦

Data Quality Objectives

The overall data quality objective is to ensure that data of known and acceptable quality are generated. To achieve this goal, data must be reviewed for 1) precision, 2) accuracy (or bias), 3) representativeness, 4) completeness, 5) comparability, and 6) sensitivity.

1) Precision- is a measure of mutual agreement among individual measurements of the same property, under prescribed similar conditions. For this project, sampling precision from field samples will be addressed by collecting and submitting for chemical analysis a concurrent field replicate sample obtained during the same sampling event. One concurrent field replicate sample of unfiltered water, filtered water, colloid material (on the glass fiber filter), and XAD-2 resin will be collected for chemical analysis. The concurrent field replicate sample of unfiltered and filtered water will be collected in a second, identical Teflon churn, filled on the bridge from the river immediately after the first churn. The concurrent field replicate sample of colloid material will be collected from a second glass-fiber filter in parallel with the first filter receiving a split of centrifuge effluent. The concurrent field replicate sample of XAD-2 resin will be collected from a second XAD-2 resin deployed in parallel to the first XAD-2 resin receiving a split of filtrate. A field replicate will not be collected for suspended sediment because of the mass limitations. Results from the field replicate samples will be included in the final report.

Precision of continuous or discrete field parameters is specific to the instrumentation. Quality objectives for field parameters are:

- An Argonaut SL 1500 or similar acoustic Doppler velocity meter (ADVM) will be used to measure:
    - Bi-directional velocity up to ±6 m/s, accurate to ±1% of measured velocity or ±0.5 cm/s (whichever is greater).
- A Teledyne Rio Grande or similar acoustic Doppler current profiler (ADCP) will be used to measure:
    - Water depth from 0.5 to 30 m, accurate to ±1 cm, and
    - Instantaneous velocity up to 20 m/s, accurate to 0.2 cm/s.
- A Forest Technology Systems DTS-12 or similar turbidity sensor will be used to measure:
    - Turbidity up to 1600 Formazin Nephelometric Units (FNU), accurate to ±2% of reading or 0.2 FNU from 0-399 FNU, whichever is greater, and ±4% of reading from 400-1,600 FNU.
- A YSI 6280 V2 sonde or similar multi-parameter sonde will be used to measure:
    - Water temperature between -5 and 50 °C, resolution 0.01 °C, accuracy ±0.15 °C
    - Dissolved oxygen between 0 and 50 mg/L, resolution 0.01 mg/L, accuracy ±0.1 mg/L or 1%, whichever is greater
    - pH between 0 and 14 units, resolution 0.01 unit, accuracy ±0.2 unit
    - Specific conductance between 0 and 100 mS/cm, resolution 0.001 mS/cm, accuracy ±0.5% of reading

    - Turbidity between 0 and 1000 NTU, resolution 0.1 NTU, accuracy ±2% or 0.3 NTU, whichever is greater.

Quality objectives for analysis of SSC and PSD at CVO are:

- SSC: For concentrations of 0-50 mg/L, accuracy ±15%, detection limit of 0.5 mg/L
- SSC: For concentrations >50 mg/L, accuracy ±5%, detection limit of 0.5 mg/L
- PSD: Size fractions reported to the nearest 1%, accuracy ±5%

The analytical laboratory(s) will conduct laboratory blank, laboratory control samples (LCS), and laboratory control replicates according to their quality assurance and control plan (with every batch of approximately 20 samples). In addition, laboratory replicates and matrix spikes (MS) of environmental samples from this project will be requested at approximately a 10% frequency. Laboratory replicates and MS of environmental water samples will be prepared by submitting an additional two 1-L samples. Laboratory replicates and MS of environmental sediment samples will be prepared by splitting a sample in the laboratory, if sufficient mass is available. The replicates and MS then will be carried through the entire analytical process. Precision is expressed as the relative percent difference (RPD). Method control limits for individual compounds will be used where available. Where no limits are published, the following limits for both water and sediment will be used:

- 60% to 135% recovery or better of LCS and MS for high-resolution organic analyses.
- 30% to 160% recovery or better of LCS and MS for organic analyses.
- 75% to 125% recovery of LCS and MS for general chemistry analyses (i.e. total organic carbon, TOC).
- 80% to 120% recovery of LCS for metals analyses.
- 75% to 125% recovery of MS for metals analyses.
- RPD between lab replicates ≤40% for high-resolution organic analyses.
- RPD between lab replicates ≤40% for organic analyses.
- RPD between lab replicates ≤20% for inorganic analyses (general chemistry and metals).

2) Accuracy- is a measure of the bias of a system or measurement. It is the closeness of agreement between an observed measurement value to the expected value or to the most-probable value. Quality-assurance check measurements on the ADVM were performed after the instrument was first acquired. Quality-assurance check measurements on the ADCPs will be performed annually, after an instrument is first acquired, after factory repair, or after firmware or hardware upgrades. Quality-assurance check measurements on the continuously-deployed turbidity sensors will be performed at the WAWSC laboratory before and after the sensor is deployed in the field and before and after the sensor is shipped from the manufacturer for annual calibration and inspection. Field checks of deployed water-quality sondes will be performed during site visits according to Wagner and others (2006). The multi-parameter sonde used for

discrete measurements will be calibrated at the WAWSC laboratory or in the onsite mobile laboratory on the day of each sampling event.

Quality assurance of SSC and PSD data produced by the USGS CVO is assessed through the Sediment Laboratory Quality Assurance Project. Historic results from annual single-blind studies are available at http://bqs.usgs.gov/slqa/.

Accuracy of chemical analysis will be assessed through laboratory matrix spikes and matrix spike duplicates requested at approximately a 10% frequency, as specified in the analytical contract. Accuracy will also be assessed through continuing calibration data generated by each laboratory. When isotope dilution methods are available, they will be used, from which analyte concentrations are adjusted based on the extraction recovery and analytical performance of its isotope.

At one time during the project, suspended sediment samples will be batched with a relevant sediment reference material. The sediment reference material(s) may include the Puget Sound Reference Material (QATS catalog # PS-SRM) for analysis using high-resolution mass-spectrometry (HRMS) methods of low-level dioxins/furans and PCB congeners. The sediment reference material(s) also may include the NIST SRM 1944 which is certified for the analysis of PAHs, PCBs, and trace elements and provide reference values for dioxins/furans. All other analyses of suspended sediment samples will be conducted as they are collected within their respective holding times.

3) Representativeness- expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. For this project, representativeness will be determined by the station selection, timing of the sampling events, sample collection methods, acceptance criteria, and sample handling and storage. To ensure samples for chemical analysis are representative, they will be collected:

- From a location in the Duwamish River that is close to the LDW but far enough upstream to minimize potential tidal influences determined during previous studies (Gries and Sloan 2009, Embrey and Frans 2003, Santos and Stoner 1972), and to minimize the probable resuspension, advection, and deposition of the sediment mass that oscillates landward and seaward in the transitional regime of the estuarine river (Ganju and others, 2004).
- During a range of flow and sediment conditions, including high-flow, high-turbidity events; capturing the rising limb of the turbidity signal (often caused by precipitation and/or increased flow from the Howard Hanson dam) when logistically possible.
- From one or more locations within the water column at the sampling station that represents average conditions, as determined by water quality parameters (specific conductance, turbidity, pH, dissolved oxygen), flow, and visual observations.
- Using USGS field sampling protocols for representative samples when available and appropriate (Mueller and Wagner, 2009; U.S. Geological Survey, 2006; Wilde and others, 2004; Davis, 2005; Edwards and Glysson, 1999).

Specifically, samples of water (for chemical and physical parameters) and suspended sediment (for physical parameters only) will be cross-section weighted average (in other words, samples are collected from multiple stations in the river's horizontal cross-section and depth-integrated), and will be collected using samplers (nozzles, bottles, and bags) that have been tested for non-biased sampling (the velocity through the nozzle into the sampler is the same as the velocity of the river, so as not to bias the sediment representation). Water samples will be composited in a Teflon churn prior to bottle filling to minimize sample variability between bottles. Previous equipment blank testing indicated minimal contamination of PCBs (less than 80 pg/L) and other analytes from the field equipment such as Teflon and silicon tubing (Conn and others, 2015).

The USGS has developed a protocol for the collection and concentration of suspended sediment using a continuous-flow centrifuge summarized in Appendix B for chemical analysis. For this project, suspended sediment samples collected for chemistry will be pumped from a point source in the river at approximately 0.8 times the depth at the thalweg. See the Field Sampling section and Table 2 for more details.

4) Completeness- is a measure of the amount of acceptable analytical data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Target completeness values are 10 storms or dam release events (of 15 total events) as defined as peak discharge and/or turbidity values at least two times greater than recent baseline values. Of those 10 events, a target of five events will capture the rising limb of the turbidity signal. The remaining events will capture smaller storms and low-flow conditions. A dam release is defined as a doubling of discharge within 24 hours at USGS 12105900 – Green River below Howard A Hanson dam, WA, located at river mile 63.8. Travel time from this station to the sampling station is approximately 15 hours. Target completeness values are 90% for chemical analyses of water, filters, and XAD-2 resin. Due to suspended sediment mass limitations during low flow events, only prioritized analytical methods may be performed (see Analytical Methods). Target completeness values are 90% for priority methods in suspended sediment (dioxins/furans, PCB congeners, metals, and PAHs).

5) Comparability- expresses the confidence with which one data set can be compared to another. For this project, comparability will be achieved through the use of standard USEPA-approved laboratory methods. In addition, standard techniques to collect and analyze representative samples will be used. This will allow comparison to previous (for example, Embrey and Frans 2003) and ongoing (for example, http://wa.water.usgs.gov/projects/riverloads/) USGS data sets. There are differences in suspended sediment field collection and processing protocols between this project, the Gries and Sloan (2009) project, and the King County project (to be published here: http://www.kingcounty.gov/environment/wastewater/Duwamish-waterway/PreventingPollution/PollutionSources.aspx#3). However, both this project and the Gries and Sloan projects utilize continuous-flow centrifugation for concentration of suspended sediment. Both projects include measures of centrifuge sediment capture efficiency, TOC, SSC and PSD, which will aid in comparing the suspended sediment chemistry results between projects. Both this project and the King County project collected suspended sediment samples from the same location during the same event using different field techniques, which will allow a qualitative comparison of different field techniques.

6) Sensitivity- is a measure of the analytical capability of the methods to meet the project objectives. The analytical detection limit (DL) and reporting limit (RL) goals for each compound in water, sediment, filters, and XAD-2 resin are presented in Appendix A. The

contract analytical laboratory will be selected specifically because of its ability to meet these low-level limits. One unfiltered water and filtered water equipment blank sample will be collected. The equipment blank will be lab-provided organic- or inorganic-free water transported from the WAWSC in its original container and processed through the field sampling equipment (Teflon nozzle, collection bag or bottle, and churn) before bottle filling. One XAD resin field blank will be collected. The field blank will be XAD resin prepared by the lab and transported in a column to the field station, then opened to the atmosphere at the field site during sample collection. Each XAD resin will be spiked with PCB surrogates to assess "wash out" during the large-volume sampling process. The lab will test the purity of each batch of purchased resin and each batch of glass fiber filters following the lab's cleaning and packing procedures, reported as pre-field resin blank and filter blank samples, respectively. Laboratory blank samples of XAD resin and glass fiber filters will be included in each batch of environmental analyses of the same matrix type. Laboratory analyses will include a solvent rinsate of the sample jar used for each parameter to confirm that the sample jars are clean and are not contributing low levels of contamination to the sample. Results from the field and equipment blanks will indicate if the equipment cleaning, sampling collection, handling, and processing procedures introduce contamination that could increase the low reporting limits. Results from the analysis of blank samples at this station collected from 2013-15 indicated that the field collection and processing techniques are appropriate for the detection levels of the parameters being analyzed (Conn and others, 2015).

Sampling Design

<u>Approach:</u> In this third agreement between the USGS and Ecology, the USGS will continue some tasks from Phase 1 and 2 and initiate some new tasks, for a total of eleven tasks. The eleven tasks are summarized in Table 2 and described in detail in the subsequent sections. Briefly, the USGS will continue to operate and maintain the stream gaging station at USGS 12113390 (Task 1). Between approximately July 1, 2016 and March 31, 2017, the USGS Washington Water Science Center will collect 12-15 additional samples of water (Tasks 2 and 3) and suspended sediment (Task 4) from USGS 12113390 during a range of hydrological conditions representing seasonal, storm-, and dam-related variations in flow and turbidity. In particular, summer and early autumn storms will be targeted, which are conditions under-represented in the current data set. The amount and size fraction of suspended sediment in representative water samples will be determined. Samples will be analyzed by Washington State-accredited laboratories for a large suite of compounds, including the 209 PCB congeners, dioxins and furans, PAHs and other semivolatile compounds, metals including arsenic and mercury, butyltins, and total and dissolved organic carbon. Owing to limited sample mass and low frequency of detection, the following compound groups that were included for analysis in Phases 1 and 2 will not be analyzed during Phase 3: volatile organic compounds (VOCs), PCB Aroclors by low-resolution mass spectrometry, hexavalent chromium, and pesticides.

A new task (Task 5), described further below, is to collect two additional field samples (a "colloid" sample and a "dissolved" sample from the water column) during each of the 12-15 sampling events for PCB congener analysis to support partition and loading estimates.

The USGS will continue to operate and maintain the downstream gaging station at USGS 12113415 to collect continuous water-quality information (Task 6). The downstream station is in an estuarine setting affected by tidally-cycling bi-directional saltwater flows that potentially could complicate chemical and sediment loading calculations. It is unknown to what extent sediment from the LDW is re-suspended and transported upstream during incoming tides, vessel turning, and dredging activities. If re-suspension and transport does occur, it is also unknown what conditions are necessary to flush that sediment back down into the LDW. To inform these unknowns, vertical and cross-sectional profiles of specific conductance, temperature, pH, dissolved oxygen, and turbidity will be collected simultaneously from multiple bridges within the estuary, including at the downstream station, over tidal cycles at different times of the year to determine the frequency, extent, shape and dynamics of saltwater intrusion (Task 7).

Project management (Task 8) will occur throughout the project, including field sampling preparation and implementation, USGS review of all data, interfacing with the analytical laboratories, and uploading the data to Ecology’s Environmental Information Management (EIM) database and the USGS National Water Information System (NWIS). Three final products will be prepared: Sediment load estimates (Task 9), Open-access publication of new Phase 3 data (Task 10), and Chemical Load estimates (Task 11).

Table 2. Summary of 11 tasks to be completed during Phase 3, July 2016 – June 2017.

Task #	Task	Description	Timing	Notes
		At USGS 12113390 (Golf Course)		
1	Operate and maintain stream gage	Continue real-time gage operation for bi-directional velocity, gage height, turbidity, and water temperature. Includes record review and publication.	Duration of agreement (12 months)	The discharge rating curve developed during Phase 2 will be confirmed or updated.
2	Sediment concentration and percent fine sediment	12-15 depth- and width-integrated water samples for determination of suspended sediment concentration (SSC) and percent fine sediment <63 µm.	Event-driven, targeting summer/first flush, 7/1/2016 - 3/31/2017	Additional SSC samples will confirm/update SSC/turbidity regression established in Phases 1 and 2. Percent fine sediment is needed to calculate chemical load estimates.
3	Water chemistry	12-15 depth- and width-integrated water samples for chemical analysis (see Table 4 for parameter list).	Event-driven, targeting summer/first flush, 7/1/2016 - 3/31/2017	-
4	Suspended sediment chemistry	12-15 samples of centrifuged suspended sediment for chemical analysis (see Table 5 for parameter list).	Event-driven, targeting summer/first flush, 7/1/2016 - 3/31/2017	Concurrent collection with water chemistry (Task 3).
5	Dissolved PCB sampling to support partition and loading estimates	In addition to the 12-15 suspended sediment samples (Task 4), 12-15 "dissolved" samples (0.45 µm-filtered centrifuge effluent) and 12-15 "colloid" samples (the filter) will be analyzed to determine site-specific, in-situ partition coefficients for PCB congeners.	Event-driven, targeting summer/first flush, 7/1/2016 - 3/31/2017	Concurrent collection with suspended sediment chemistry (Task 4). The dissolved sample will be concentrated on XAD-2 resin for analysis.

At USGS 12113415 (E. Marginal Way)				
6	Operate and maintain stream gage	Continue real-time gage operation for gage height, turbidity, water temperature, and specific conductance. Includes record review and publication.	Duration of agreement (12 months)	Gage was in start-up mode when project was suspended. The long-term viability of each sensor will be determined in Phase 3.
7	Assess tidal dynamics	Over 3-5 different tidal cycles, conduct vertical and cross-section profiles of specific conductance, temperature, pH, dissolved oxygen, and turbidity at bridges between RM 5 and 11 to determine extent and shape of salt wedge in river.	Event-driven, 7/1/2016 - 3/31/2017	An improved understanding of the extent, shape, and duration of the salt wedge is needed to address sediment and chemical transport in the future.

At USGS Washington Water Science Center Laboratory, Tacoma, WA				
8	Project management	USGS review of all data, interface with labs, upload to EIM.	Duration of agreement	-
9	Publication of sediment load	Publication (as USGS Open-File Report, or similar) of sediment rating curve and resulting annual sediment loads.	Prepared by 12/31/2016	This will be the peer-reviewed version of the sediment loads reported in the April 2016 Progress Report.
10	Publication of new data	Publication (as USGS Data Release in ScienceBase or similar) of new data collected in Phase 3.	Prepared by 6/30/2017	This will include QA/QC data, data from the tidal studies, and other data that is not able to be stored in Ecology's EIM database or the USGS NWIS database.
11	Publication of chemical loads	Publication (as USGS Scientific Investigations Report or similar) of chemical loads and other interpretive results.	Prepared by 6/30/2017	The focus is on chemical load estimates, but will also include other interpretive results including the tidal dynamics at the downstream station.

Site Description: The field activities will be conducted at the two new USGS stream gaging stations installed for this project in tidally-influenced reaches of the Duwamish River. The upstream station is located at the Foster Golf Links footbridge at RKM 16.7, which is approximately 8 RKM upstream of the LDW boundary (Figure 1). It has a USGS Station ID of: USGS 12113390 - Duwamish River at Golf Course, Tukwila, WA. Field activities at the upstream station will build on the USGS data set collected for this project since February 2013 as well as historic USGS water quality data collected at this station through the National Water Quality Assessment program between 1995 and 2004. The upstream station is tidally influenced: reverse (upriver) streamflow occurs regularly during the summer low tides. The station is upstream of the salt wedge, which has been documented during high tide-low flow times by Gries and Sloan (2009) at RKM 10.8 and by Santos and Stoner (1972) as far upstream as the Foster Bridge (RKM 14.0). The upstream station also has an existing bridge (Figure 2) that is safe, secure, and well suited for sample collection. Those features will maximize the potential to collect complete high-quality suites of data during the severe weather conditions that generate the high-flow, high-turbidity events during the rising limb of a storm or dam release hydrograph that are a primary target for the sampling. The stream gaging station was installed in November 2013 with an ADVM, temperature and turbidity sensor. The USGS has an on-going amicable agreement with the City of Tukwila golf course allowing access to the stream gaging station, the golf cart footbridge, and the riverside centrifuge hutch (Figure 3). The site location provides safe sampling access because it is a wide bridge with limited golf cart and foot traffic only. After hours, the bridge can only be accessed through a locked gate (for which the USGS has a key), providing heightened security compared to other sites.

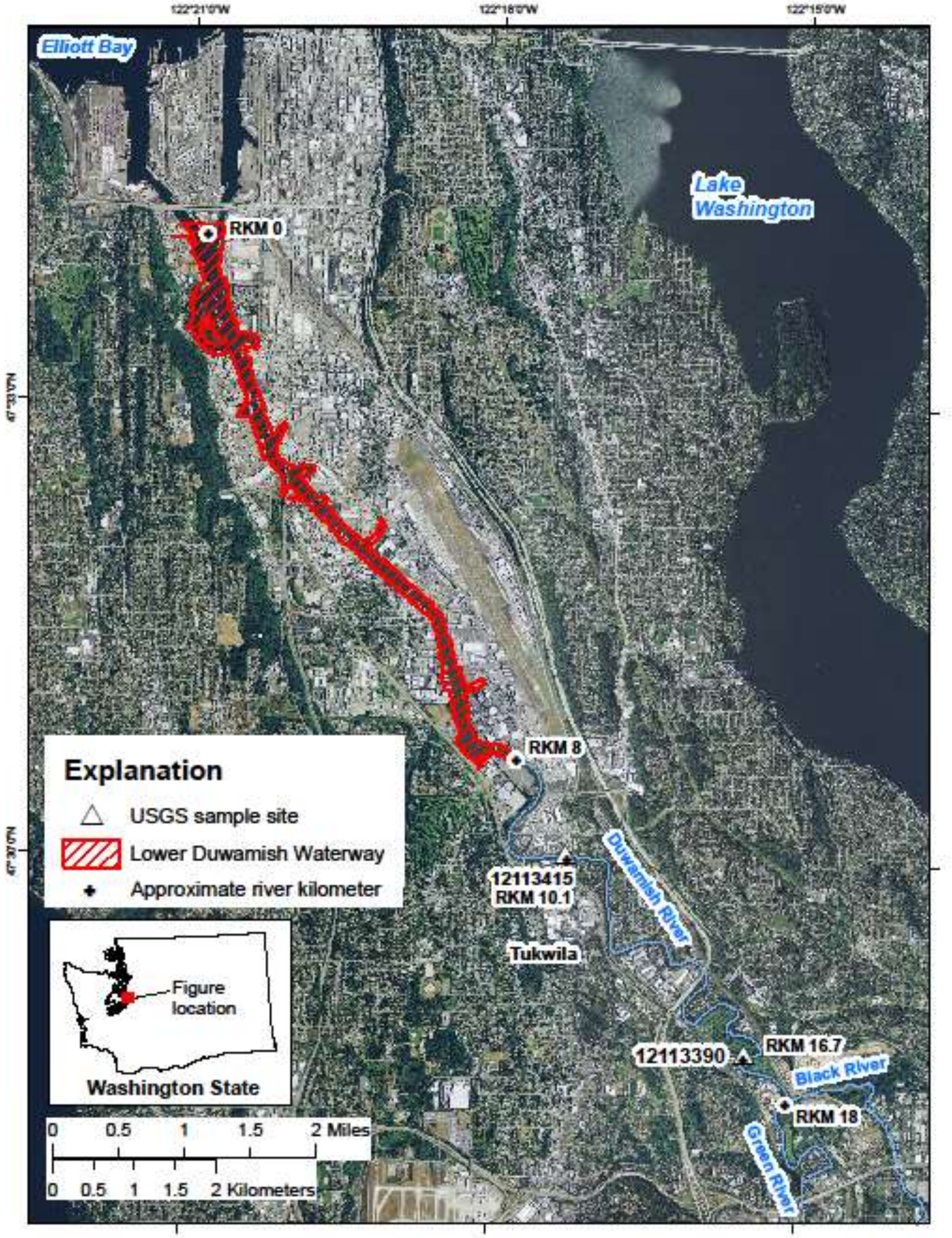

Figure 1. Map of upstream (12113390) and downstream (12113415) USGS sampling stations relative to the Lower Duwamish Waterway Superfund Site in Seattle, WA.

Figure 2. Photo of the upstream station, USGS 12113390 – Duwamish River at Golf Course at Tukwila, WA, located at River Kilometer 16.7.

Figure 3. Site photographs of the upstream station (USGS 12113390) including (A) the stream gaging station, (B) the golf cart footbridge, and (C) the riverside centrifuge hutch.

The downstream stream gaging station is located at the East Marginal Way bridge at River Kilometer (RKM) 10.1, which has a USGS Station ID of: USGS 12113415 – Duwamish R at E Marginal Way Br at Duwamish, WA (Figure 4). This station, which is approximately 2 RKM upstream of the LDW boundary (Figure 1), is in the estuarine portion of the Duwamish River and is affected by tidally-driving bi-directional saltwater flows and may also be affected by navigational/dredging effects occurring in the LDW. No historic USGS water- or sediment-quality data is available for this station. The bank and shoreline are accessible, there is ample off-road parking, and a pedestrian sidewalk will be used for bridge-based measurements (Figure 5). Permits and approvals were received from all relevant agencies (City of Tukwila, WA Department of Fish and Wildlife, Ecology, and the U.S. Army Corps of Engineers). Installation occurred during the first half of 2015 as part of this project, and included velocity, turbidity, temperature, and specific conductance sensors.

Figure 4. Photo of the downstream station: USGS 12113415 – Duwamish R at E Marginal Way Br at Duwamish, WA, located at River Kilometer 10.1.

Figure 5. Site photographs of the downstream station (USGS 12113415) including (A) the pedestrian sidewalk, (B) off-road parking and bank access, and (C) shoreline access.

The cross-sections at both stations promote good mixing of the water column as the bridge supports are on the bank and there are no large bridge abutments or other disruptions to flow and mixing in the water (Figures 2 and 4). A traffic control plan will be in place for both stations to direct pedestrian traffic around the work zone.

Continuous Real-Time Monitoring

The USGS has recently published approved methods for reporting discharge in tidally-influenced river reaches using ADVM instrumentation (Levesque and Oberg, 2012). By measuring particle backscatter through acoustic Doppler principles, an ADVM can provide powerful information regarding forward and reverse flow throughout the entire vertical and horizontal river cross-section at a station. Both USGS stations in this project were instrumented in Phase 2 to measure velocity, stage, and water temperature (and specific conductance at the downstream estuarine station) for determination of continuous, real-time discharge data. A turbidity sensor is co-located at each station and continuous, real-time turbidity data also is available. The turbidity sensor (DTS-12, Forest Technology Systems, Inc.) uses Nephelometric geometry to measure backscattered light, reported as turbidity.

During Phase 2, a discharge rating curve was developed at the upstream station. The real-time discharge, temperature and turbidity data has been available since November 2013 for the upstream station: USGS 12113390 – Duwamish River at Golf Course at Tukwila, WA, at http://waterdata.usgs.gov/wa/nwis/uv?site_no=12113390. Real-time provisional data has been available since February 2015 for the downstream station: USGS 12113415 – Duwamish R at E Marginal Water Br at Duwamish, WA, at http://waterdata.usgs.gov/nwis/uv?site_no=12113415. It has since been determined that a discharge rating curve cannot be developed at this site owing to large bedload transport that affects performance of bed-mounted instrumentation such as the ADVM (i.e. the instrument is recurrently buried). Stage, turbidity, temperature, and specific conductance at the downstream station will be transmitted for the duration of this project.

The instrumentation at both stations will continue to be operated and maintained by the USGS for the duration of the agreement (Tasks 1 and 6, Table 2). The provisional real-time data will continue to be publicly available and the data records will be compiled, reviewed, and approved by the USGS in a timely manner consistent with USGS protocols (Levesque and Oberg, 2012). The real-time data at both stations will be valuable in informing USGS discrete sampling events and will improve the ability to capture a representative range of flow and sediment conditions. In addition, the real-time data may be useful to other agencies, tribes and the public to inform other river-related activities such as flood management, sediment management decisions, and habitat restoration.

Field Sampling Methods

Between approximately July 1, 2016 and March 31, 2017, 12 to 15 discrete bridge-based sampling events will be conducted at the upstream station (USGS 12113390, RKM 16.7). This will allow continued opportunity to capture high-chemical loading events including summer and autumn “first flush” events, and to better represent the seasonal and inter-annual variability of the river system. Sampling will occur approximately two times per month, targeting high flow and (or) high turbidity events due to storms and dam releases (see sampling scheme in Table 3). A target of 10 of the 15 events will capture storms or dam releases of varying sizes with discharge and (or) turbidity values at least double recent baseline values. The remaining events will capture baseline conditions including summer and winter conditions. These targets will guide the sampling scheme, though actual sampling will be determined by real-time data, personnel availability, and safety.

Table 3. Proposed sampling scheme. Actual sampling scheme will be determined by the occurrence of high flow and (or) high sediment events.

Matrix	Number of Environmental and Quality Control (QC) Samples									
	2016					2017			Project Total	
	August	Sept	Oct	Nov	Dec	Jan	Feb	Mar	Environmental	QC
Water	1	2 + 1 AXYS and 1 ARI field equipment blank	2 + 1 KC and 1 MEL field equipment blank	2	2	2 + 1 concurrent field replicate (all parameters)	2	2	15	2 [a]
Suspended sediment	1	2	2	2 + 1 SRM	2	2	2	2	15	1
Glass-fiber filter(s)	1	2	2	2 + 1 concurrent field replicate	2	2	2	2	15	1
XAD-2 resin	1	2	2 + 1 trip blank	2	2 + 1 concurrent field replicate	2	2	2	15	2

[a] The 2 water QC samples are composed of 1 field equipment blank for all parameters and 1 concurrent field replicate for all parameters. The field equipment blank will be submitted to each of the 4 analyzing labs during September and October 2016.

The study design from the first two phases of this project (Conn and Black, 2014; Conn and others, 2015) will be followed, including monitoring of field parameters, the collection of water samples (Tasks 2 and 3, Table 2) and a suspended sediment sample (Task 4, Table 2) during each of the 12-15 bridge-based sampling events at the upstream station. Two additional samples will be collected during each event – a “colloid” sample and a “dissolved” sample (Task 5, Table 2) - for PCB analysis. Therefore, six activities will be conducted during each of the 12-15 sampling events:

- Monitor field water-quality parameters (pH, temperature, dissolved oxygen, turbidity, specific conductance);
- Collect a depth- and width-integrated WATER sample for determination of SSC and percent fine sediment < 63 µm;
- Collect a depth- and width-integrated WATER sample for chemical analysis;
- Collect a point sample of SUSPENDED SEDIMENT for centrifugation and chemical analysis;
- Collect a COLLOID sample on a filter from the water exiting the centrifuges for PCB analysis;
- Collect a DISSOLVED sample on XAD-2 resin from the water exiting the filter for PCB analysis.

General water quality: Environmental parameters, including pH, water temperature, specific conductance, dissolved oxygen, and turbidity, will be measured during each sampling event using a multi-parameter sonde (YSI Inc., Yellow Springs, OH) by USGS field personnel according per USGS protocols (Wilde, variously dated). The sonde will be deployed in a representative location in the cross-section, for example, at 60 percent of depth in the centroid of flow.

Suspended sediment physical parameters: A USGS team will collect depth-integrated samples from a minimum of 5 cross-section stations and composite them to characterize the abundance and size distribution of suspended sediment (Task 2, Table 2) using standard USGS protocols (Edwards and Glysson 1999). This sampling technique collects a depth- and width-integrated sample that is representative of the entire river cross-section at that sampling station. Briefly, a sampler (Figure 6) is lowered at a consistent transit rate from the surface to the bottom and back to the surface of the water column at each station. The process is repeated as necessary to obtain sufficient sample. Approved nozzles, containers, and samplers, such as the D-96 sampler (Figure 6; Davis, 2005) will be used. The volume of water collected will depend on current sediment conditions, and is expected to range between 5 and 20 L. These samples will not receive any chemical analyses, but will be used to characterize the abundance and size distribution of suspended sediment at the time of concurrent suspended sediment chemistry sampling (Task 4, Table 2). An accurate representation of the abundance and size distribution of suspended sediment will be combined with the suspended sediment chemistry sampling results to estimate the potential load of suspended sediment-bound chemicals being transported downstream. The samples will be stored until transport to CVO for analysis of SSC and percent fine sediment < 63 µm using published USGS methods (Guy 1969).

Figure 6. Photo of example USGS sampling equipment, including a crane, reel, and sampler for representative collection of depth- and width-integrated samples.

Water chemistry: After completing the cross-section described above, a team will immediately collect a second cross-section of depth-integrated samples from a minimum of 5 cross-section stations and composite them for water chemistry analysis (Task 3, Table 2) using standard USGS protocols (U.S. Geological Survey, variously dated) including those specific for sampling of trace organic chemicals (Wilde and others, 2004). Water samples will be collected from each cross-section station in Teflon bottles or bags using an approved sampler, such as the D-96 (Davis, 2005). The water samples will be composited in a 14-L Teflon churn and immediately processed in an on-site mobile laboratory (see Sample Processing) into sample bottles appropriate for the chemical parameters listed in Appendix A.

Suspended sediment chemistry: The chemical analysis of suspended sediment requires more material (~70 g) than is routinely collected in a depth-integrated sample and therefore a pump will be used concurrent with Tasks 2 and 3 to collect sufficient water into Teflon-lined containers (Task 4, Table 2) for centrifugation and concentration. The same point-sampling location from the second phase of the project will be used, which is located at a depth approximately 0.8 times the main channel depth in the centroid of flow (approximately 30 feet from the left edge of water). Cleaned Teflon tubing will be deployed during each sampling event through permanently installed rigid housing running from the bank along the bed to the centroid of flow. The tubing will be attached to a high-flow peristaltic pump (~4 L/min) such as an Isco autosampler pump (Teledyne Isco, Lincoln, Nebraska) or similar unscreened, high-flow pumping device. Water will be collected in Teflon-lined containers on the river bank for on-site centrifugation. In the second phase of the project, a centrifuge hutch was installed on the river bank near the in-river rigid tubing (see Figure 3C), which allows continuous, onsite, unattended pumping and centrifugation during times when it otherwise may not be safe to sample from the bridge (for example, in the dark or in the presence of large, floating debris). The volume of water collected will depend on the current river sediment conditions, and likely will be between 1000 and 2000 liters (four to eight hours of continuous pumping) during high-sediment storms and dam releases. During low-sediment baseline periods, larger volumes of water may be collected (~5,000 to 10,000 liters) and centrifuged continuously over multiple days. Pumping for suspended sediment will continue until sufficient suspended sediment has been collected for chemistry analysis, until the flow returns to pre-storm conditions, or until pumping is no longer feasible due to chemical holding times, resource limitations or safety concerns. Sediment collected from the centrifuge will be analyzed for those compounds listed in Appendix A.

Two additional samples will be collected from the centrifuge field set-up (Figure 7) to support PCB partition and loading estimates. In addition to the suspended sediment sample captured in the settling basins and centrifuge bowls (Task 4, Table 2), the water exiting the centrifuges will be passed through parallel 0.45 µm glass-fiber filters followed by concentration on an XAD-2 resin (Task 5, Table 2). The particulates captured on the filters will be called the “colloid” sample (particles larger than 0.45 µm that were not captured by the centrifuges). One filter will be analyzed for PCB congeners and the other by X-ray diffraction for mineralogical composition. The results may better describe the mineralogical composition of the fine-grained sediments to which PCBs prefer to sorb. For example, the analysis will provide the types of minerals (iron oxides, quartz, clays like illites) and amorphous material (such as organic carbon) present in each sample and their relative abundance, which may relate to the amount of PCB measured on the particles. The sample concentrated on the XAD-2 resin will be called the “dissolved” sample (freely dissolved or less than 0.45 µm) and will be analyzed for PCB congeners.

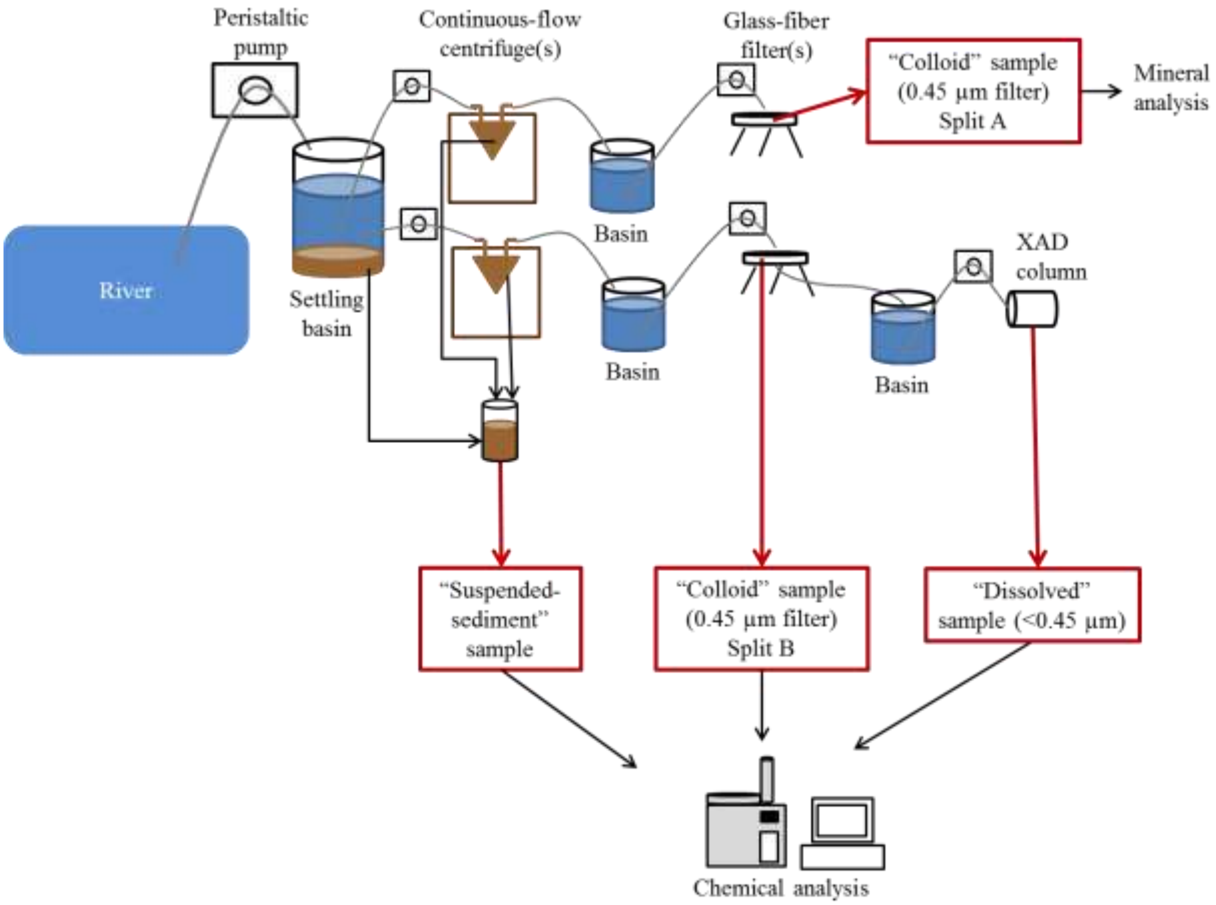

Figure 7. Schematic of centrifugation field set-up. In addition to the suspended sediment sample, two additional samples will be collected: the "colloid" sample, which is the particulate matter captured from the centrifuge effluent on a 0.45 µm glass fiber filter, and the "dissolved" sample, concentrated from the filtrate on an XAD-2 resin. A colloid split also will be analyzed by X-ray diffraction for mineral content.

A sampling effort, separate from the chemical sampling at USGS 12113390, will be conducted within the Duwamish estuary, including the downstream gaging station (USGS 12113415). Over three to five tidal cycles at different times of the year, vertical and cross-sectional profiles of water quality parameters (specific conductance, water temperature, turbidity, pH, and dissolved oxygen) will be conducted simultaneously at multiple bridges between RKM 8 (upstream end of the LDW) and RKM 16 (Foster bridge) to determine the frequency, shape, and extent of saltwater intrusion and mixing dynamics (Task 7, Table 2). An improved understanding of the extent, shape and duration of the salt wedge, particularly at the downstream stream gaging station, is needed to inform future sediment and chemical sampling designs.

Sample Processing

Water Chemistry Sample Processing (see Task 3, Table 2)
In the mobile laboratory, the composited water sample will be churned in a closed chamber according to USGS protocols (Wilde and others, 2004) to minimize contamination and ensure sample homogenization prior to bottle filling. Bottles will be filled for analysis of unfiltered-water samples for dioxins/furans, PCB congeners, PAHs, semivolatile compounds, butyltins, trace elements, low-level mercury, and TOC (Table 4). A sub-sample of churned water will be filtered through a 0.45 μm filter for analysis of trace elements and DOC. Pre-acidified bottles will be used for samples requiring preservation (See Table 4). Samples will be stored on ice until transportation within 48 hours to Ecology's Manchester Environmental Laboratory, ARI, and KCEL, or to the USGS Washington Water Science Center laboratory refrigerator for storage at 4 °C until shipment to AXYS.

Table 4. Analytical parameter group, method, analyzing lab, sample container, preservative, holding time, and number of samples for water samples.

[EPA, U.S. Environmental Protection Agency ; PCB, polychlorinated biphenyl; PAH, polycyclic aromatic hydrocarbon; AG, amber glass; FLPE, fluorinated polyethylene, with complete certification for mercury by EPA Method 1631; HDPE, high-density polyethylene; H_2SO_4, sulfuric acid; HNO_3, nitric acid.]

Analytical Parameter	EPA SW846 Method / Reference	Analyzing Lab	Sample Container	Preservative	Holding Time	Number of Samples [a]	
						Unfiltered	Filtered
Dioxins/furans	1613B	AXYS	2 x 1 L AG	Cool ≤ 6 °C	1 yr, chilled	17	0
209 PCB congeners	1668C	AXYS	2 x 1 L AG	Cool ≤ 6 °C	1 yr, chilled	17	0
Organic carbon	Standard Methods 5310B	MEL	250 mL AG	Cool ≤ 6 °C + pH <2 w/ 2 mL H_2SO_4	28 d	17	17
Trace Elements [b]	6020B	MEL	500 mL HDPE	2.5 mL 1:1 HNO_3	6 mo	17	17
Low-Level Mercury	1631E	MEL	250 mL FLPE	5 mL 1:1 HNO_3	28 d	17	17
Low-level PAHs	8270D SIM	ARI	2 x 1 L AG	Cool ≤ 6 °C	7 d	17	0
Low-level semi-volatile compounds	LL-8270D	ARI	2 x 500 mL AG	Cool ≤ 6 °C	7 d	17	0
Butyltins	8270D	ARI	2 x 500 mL AG	Cool ≤ 6 °C	7 d	17	0
Ultra low-level PAHs	8270D SIM, Large-volume injection	KCEL	3 x 1 L AG	Cool ≤ 6 °C	7 d	17	0

[a] Explanation of the number of samples: 15 environmental, 1 equipment blank, 1 concurrent field replicate

[b] Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Lead, Manganese, Molybdenum, Nickel, Selenium, Silver, Thallium, Vanadium, and Zinc

Suspended Sediment Chemistry Sample Processing (see Task 4, Table 2)
Water pumped from the river for suspended sediment chemistry will be stored on-site in Teflon-lined 30-gallon drums (see Figure 3C). Some sediment will settle in the drums, and the overlying water will be pumped through a floating Teflon tube into a continuous-flow centrifuge (Figure 7, CFC Express, Scientific Methods, Inc., Granger, IN). The centrifuge runs at a fixed speed of 10,000 revolutions per minute, and testing indicates that inflow rates of 600 mL/min results in sediment capture efficiencies greater than 90 % (by weight). At a flow rate of 600 mL/min using a single centrifuge, approximately 25 to 50 hours of total centrifuge time will be required for each sampling event. Additional centrifuges may be used to reduce processing time. Every eight hours or less, captured sediment from the centrifuge bowl(s) will be composited in a pre-tared glass jar and stored quiescently at 4 °C. The settled sediment at the bottom of each drum also will be transferred to the glass jar and composited with the bowl sediment. The centrifugation will occur in an enclosed hutch on the river bank at USGS 12113390. See Appendix B for a summary of the sampling and processing procedure for suspended sediment. Excess overlying water in the glass jar will be decanted by pipette and centrifuged on a traditional centrifuge at the WAWSC Laboratory. Any additional spun sediment will be added to the composite sample. A final wet weight of the suspended sediment sample will be determined at the WAWSC laboratory as the difference between the empty jar and the jar containing the sample. Each laboratory will determine moisture content on the sub-sample to report results on a dry weight basis.

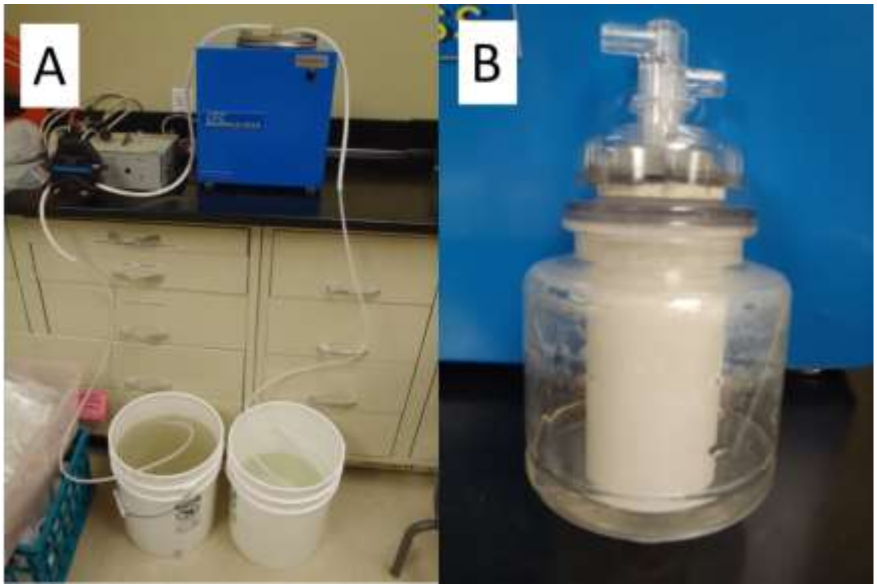

Figure 8. (A) Continuous-flow centrifuge containing (B) a centrifuge bowl for capturing sediment.

Suspended-sediment samples will be stored in jars for analysis of dioxins/furans, PCB congeners, PAHs, semivolatile compounds, butyltins, trace elements, low-level mercury, and TOC (Table 5). Samples will be stored on ice until transportation to Ecology’s Manchester Environmental Laboratory or ARI. A homogenized sub-sample for dioxins/furans and PCB congeners will be stored frozen until transportation to the analyzing laboratory.

Table 5. Analytical parameter group, method, sample container, minimum mass required, preservative, holding time, and number of samples for suspended-sediment samples.

[EPA, U.S. Environmental Protection Agency; PCB, polychlorinated biphenyl; PAH, polycyclic aromatic hydrocarbon; PSEP, Puget Sound Estuary Program; WMG, wide-mouth glass. The parameters are listed in priority order, from top to bottom, in the event there is insufficient mass collected to complete all analyses. Each analyzing lab performs a total solids (moisture content) analysis on the lab's sub-sample to report results on a dry weight basis; the mass required is less than a gram.]

Analytical Parameter	EPA SW846 Method / Reference	Analyzing lab	Sample Container	Minimum Dry Mass Required	Preservative	Holding Time	Number of Samples [a]
Dioxins/furans	1613B	AXYS	4 oz. WMG (amber)	15 g (co-extraction)	Cool ≤ 6 °C	1 yr, frozen	16
209 PCB congeners	1668C	AXYS					16
Organic carbon	PSEP 1986	MEL	8 oz. WMG	2 g	Cool ≤ 6 °C	14 d	16
Trace Elements [b]	6020A (or 200.8)	MEL		5 g	Cool ≤ 6 °C	6 mo	16
Low-Level Mercury	7471A	MEL		2 g	Cool ≤ 6 °C	28 d	16
Low-level PAHs	8270D SIM	ARI	8 oz. WMG	15 g	Cool ≤ 6 °C	14 d	16
Semivolatile compounds	Dual-scan 8270D	ARI		15 g	Cool ≤ 6 °C	14 d	16
Butyltins	8270D	ARI		10 g	Cool ≤ 6 °C	14 d	16

[a] Explanation of the number of samples: 15 environmental, 1 sediment reference standard.

[b] Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Lead, Manganese, Molybdenum, Nickel, Selenium, Silver, Thallium, Vanadium, and Zinc

Glass-fiber filter and XAD-2 resin processing (see Task 5, Table 2)

Water exiting the centrifuge(s) will be collected in a Teflon-lined settling basin and pumped through two parallel 0.45 µm nominal pore size, 142-mm diameter glass-fiber filters in stainless steel housing to capture particles greater than 0.45 µm that are not captured in the centrifuge bowl(s). Filters will be replaced when clogging occurs, as indicated by a decrease in flow rate to less than half of the original flow rate. Glass-fiber filters will be pre-prepared by washing with organic-free blank water, drying, and pre-weighing. Filters from one of the filtration stands will be sent to the USGS National Research Program lab in Boulder, CO for analysis of sediment mineralogy of the particles captured on the filters. Filters from the other filtration stand will be stored frozen until shipment to AXYS for 209 PCB congener analysis (Table 6).

Water exiting the filtration stand designated for PCB analysis will be collected in a Teflon-lined settling basin and pumped through XAD-2 resin packed in a stainless steel column to capture freely dissolved PCBs and PCBs sorbed to particles less than 0.45 µm (Table 6). The flow rate will be maintained at less than 1.2 L/min, per AXYS protocols. If a sub-sample of the filtered water is processed through the XAD-2 resin rather than the entire filtered column (to avoid overloading the resin), then a time-averaged sub-sample of water will be processed (for example, 10 L every hour over the duration of centrifugation). The filters and XAD-2 resin will be acquired, prepared, blank-tested, and shipped to the USGS in advance of sampling by AXYS per lab protocols (available from AXYS upon request) and as determined in the analytical contract between Ecology and AXYS. The total volume processed through the filter and XAD column for PCB analysis will be recorded by USGS field personnel during each sampling event. The volume will be reported to AXYS so the filter and XAD results can be reported on a pg/L basis.

Table 6. Analytical parameter group, method, sample container, minimum mass required, preservative, holding time, and number of samples for glass-fiber filter and XAD-2 resin samples.

[EPA, U.S. Environmental Protection Agency; PCB, polychlorinated biphenyl; PAH, polycyclic aromatic hydrocarbon; PSEP, Puget Sound Estuary Program; WMG, wide-mouth glass; WMP, wide-mouth plastic.]

Analytical Parameter	EPA SW846 Method / Reference	Analyzing Lab	Sample Container	Preservative	Holding Time	Number of Samples [a]	
						Glass-fiber filter	XAD-2 resin
209 PCB congeners	1668C	AXYS	142 mm, 0.45 µm glass fiber filters (multiple per event)	Cool ≤ 6 °C	1 yr, frozen	16	17
			XAD-2 resin packed in stainless steel column	Cool ≤ 6 °C	1 yr, chilled	16	17

[a] Explanation of the number of samples: 15 environmental, 1 concurrent field replicate, 1 trip blank (XAD-2 resin only)

Analytical Methods

Composited depth-integrated water samples (Task 2) will be analyzed for SSC and percent fine sediment by the USGS Cascade Volcano Laboratory (CVO) located in Vancouver, WA using USGS methods (Guy 1969). This data will be comparable to the results collected in Phases 1 and 2 of the project as well as data currently being collected from other large rivers to assess sediment and/or chemical loading in Puget Sound, including the Puyallup, Elwha, Sauk, and Stillaguamish.

Samples of water and suspended sediment will be analyzed for a suite of toxic chemicals and total and dissolved organic carbon by Ecology's Manchester Environmental Laboratory or other Washington State-accredited laboratories. This includes high-resolution analysis for dioxins/furans and the 209 PCB congeners as well as analysis of PAHs, butyl tins, other semivolatile compounds, and metals including arsenic and mercury. Filtered water will be analyzed for metals and dissolved organic carbon. The analyte groups and methods are listed in Tables 4 and 5. Glass fiber filters (the colloid samples) and XAD-2 resin (the dissolved water samples) will be analyzed for PCB congeners only (Table 6). The individual analytes are listed in Appendix A.

The full criteria to be met by the lab(s) are described in the Statement of Work for each analytical contract. Ecology's Manchester lab will manage the analytical contract for any analyses not being conducted by Manchester, including the development of the Statement of Work, the posting, evaluation, and awarding of the contract, and payment. In addition, Ecology's Manchester lab will conduct data validation on all analytical chemistry data comparable to an USEPA Level 4 validation report. The analytical laboratory costs, including XAD-2 resin and column costs, and Level 4 validation costs are not included in the USGS-Ecology budget agreement.

Found in Appendix A, for water, sediment, filters, and XAD resin, are the analytes listed individually with the following information:

- Expected Reporting Limit (RL) – The lowest concentration that can be reliably achieved within specific limits of precision and accuracy during routine operating conditions. This is often synonymous with a Quantitation Limit (QL). For HRMS compounds including dioxins/furans and 209 PCB congeners, this can be reported as the Lower Method Calibration Limit, which is determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used: ((lowest level calibration standard) x (extract volume))/sample size,

- Expected Detection Limit (DL) – The lowest result that can be reliably distinguished from a blank with a false positive rate ≤ 1%. For HRMS compounds, this is often reported as a Typical Sample-specific Detection Limit, which is defined as the concentration equivalent to 2.5 times the estimated chromatographic noise height, determined individually for every sample analysis run.

The expected RLs and DLs listed in Appendix A are target levels identified by Ecology. The analytical laboratory(s) will be selected because of its ability to meet these low-level limits. Results for all analytes, with the exception of TOC and DOC, will be reported down to the DL. TOC and DOC will be reported down to the RL. When TOC or DOC is not detected in a sample above the RL, the RL will be reported with a "U" qualifier. Results for the other analytes that are greater than the DL but less than the RL will be reported as detections at the detected level with a "J" qualifier. When the compound is not detected at a concentration greater than the DL, the DL value will be reported with a "UJ" qualifier for HRMS compounds and with a "U" for ultra low-level PAHs (analyzed by King County). The RL value will be reported with a "U" qualifier for all other non-HRMS compounds (trace elements by Manchester and PAHs, butyl tins, and other semivolatile organic compounds by ARI).

The related common lab qualifiers used are defined as:

J	The analyte was positively identified. The associated numerical result is an estimate;
U	The analyte was not detected at or above the reported result;
UJ	The analyte was not detected at or above the reported estimate;
NJ	Disturbance of the mass ion used to monitor instrument performance (lock-mass) present;
K	A peak was detected that did not meet all the criteria for identification as the target analyte; the reported value is the estimated maximum possible concentration;
R	Result rejected as unusable, owing to gross contamination such as saturated peaks or a gross field or laboratory error.

A blank and at least five calibration standards shall be used to establish each calibration curve. At least one standard shall be at or below the RL, but above the DL. Labs will perform re-extractions or re-digestions (if within the holding time limits) or re-analyses of extracts (if outside the holding time limits) when gross laboratory contamination is present, for example, if target analytes are present in the method blank at concentrations exceeding the RL. In addition, for PCB congeners, extraction and analysis of a duplicate field sample and method blank are required if the total PCB concentration in the method blank sample is greater than or equal to 170 pg/L. This criterion was developed for the Spokane River, WA PCB study, and is being applied to this project. See Appendix C – the Method Blank Contamination Decision Rules for details. A 5X rule, instead of 10X as shown on the Decision tree, will be applied to this study (described further in the Data Management, Verification, and Validation section below).

A minimum of 65 g of dry suspended sediment is required to complete the chemical analyses (Table 5). During low-turbidity sampling events, even with consecutive days of water collection, there may be insufficient sediment concentrated from the centrifuge to complete all methods. In these cases, a priority list of analytical methods will be followed (with #1 being the top priority):

1. Percent solids (also called moisture content; always completed; required to report on a dry weight basis)
2. TOC (always completed; required to report a TOC-normalized concentration)
3. Dioxins/Furans and PCB Congeners (a single co-extraction by a high-resolution laboratory)
4. Metals (including mercury)
5. PAHs
6. Semivolatile compounds
7. Butyltins

All efforts will be made to collect sufficient sediment to complete all seven methods, and it is expected that there will be sufficient sediment to analyze the priority methods (#1-5) during all events.

A colloid split sample (filter) from each event will be analyzed for mineralogical composition by X-ray diffraction at the USGS National Research Program lab in Boulder, CO. If excess suspended sediment is available after chemical analysis, a sub-sample of suspended sediment also will be analyzed by X-ray diffraction. The samples will be compared to a reference mineral library (RockJock; see Eberl, 2003). A least-squares fitting will be utilized to match the spectra in the field sample to spectra of standard reference minerals and quantify the amount of each identified mineral present.

Quality Assurance and Control Procedures

USGS quality assurance procedures for surface-water measurements and water-quality sampling and analysis will be followed (Mastin, 2016; Wagner and others, 2007; Wagner and others, 2006; U.S. Geological Survey, 2006; Wilde and others, 2004; Wilde, variously dated). This includes the proper equipment selection, cleaning procedures, and sampling protocols for low level organic compounds and metals. Sampling equipment for chemical analyses will be Teflon and will be pre-cleaned with phosphate-free soap, rinsed three times with tap water, soaked in 5% hydrochloric acid, rinsed with deionized water, rinsed with high purity methanol, and air dried before being stored in clean bags for field transport. Field sampling techniques include various measures to avoid sample contamination including the 2-person “clean hands, dirty hands” technique for collecting low-level mercury samples and processing of water samples in a clean mobile laboratory. Hydrologists and hydrological technicians on this project have been trained at the USGS National Training Center in the collection of water quality samples, including samples for trace organic and low level mercury analyses.

The field folder will include copies of the QAPP and the protocols referenced within. Deviations from the QAPP will be noted on the field sheet. Results from field quality control samples (equipment blanks) will be reviewed by the project investigators. Field protocols will be modified to correct any identified contamination issues. Results from QA/QC samples analyzed in the first two phases of this project indicated that the equipment cleaning and sample processing protocols were appropriate and no major contamination issues were identified. Laboratory quality control samples (a blank, replicate, and matrix spike per batch of 20 samples) will be reviewed by laboratory personnel. If values exceed the control limits, then laboratory personnel will take appropriate corrective actions such as re-runs and re-extractions and/or discuss modifications to the protocol with the principal investigator, as described above in the Data Quality Objectives section and Analytical Methods section.

Audits include the review of proficiency testing results by the analytical labs of submitted standard reference sediments. The selected labs quality assurance and control procedures will be assessed and evaluated in accordance to the USGS Laboratory Evaluation Program (LEP) according to the guidance provided by the USGS Branch of Quality Systems (https://bqs.usgs.gov/labEvaluation.php) and in accordance with USGS Office of Water Quality Technical Memorandum 2007.01 and 2014.01. The Ecology Project Manager and Quality Assurance Specialist will accompany field personnel during a sampling event in the first half of the project to perform a qualitative audit of conformance to the QAPP and to suggest corrective actions as needed.

Data Management, Verification, and Validation

A field form, modified from the standard USGS Surface Water Quality Notes, will be completed during each sampling event (Appendix D). Field parameters recorded will include date, time, sampling team, field conditions, sampler types, sampling methods, meter and probe serial numbers and calibration information, number and type of quality assurance samples collected, and any deviations from the sampling protocol.

General water quality field parameters, including water temperature, specific conductance, dissolved oxygen, pH, barometric pressure and turbidity will be compiled on the field form and reviewed by one of the USGS principal investigators prior to entry into the USGS National Water Information System (NWIS). Instantaneous discharge records will be reviewed and approved according to standard USGS protocols (Mueller and Wagner, 2009). The continuous records will be reviewed and approved according to standard USGS protocols for the ADVM (Levesque and Oberg, 2012) and turbidity sensor (Wagner and others, 2006), which includes verification and validation by secondary and tertiary reviewers prior to entry into NWIS. Quality assurance procedures used by the WAWSC for activities related to the collection, processing, storage, analysis, and publication of surface-water data are described in detail in Mastin (2016).

Quality assurance procedures utilized by USGS sediment laboratories for analysis of suspended-sediment concentration are provided by Knott and others (1992; 1993) and Matthes and others (1992). Prior to sending samples to the laboratory, analytical services requests for determination of suspended-sediment concentration and particle-size analysis and sample site and other information are entered into the Sediment Laboratory Environmental Data System (SLEDS). The laboratory results are then added to the SLEDS system and automatically uploaded into the NWIS database for long-term, publicly-accessible storage.

All analytical results from the contract labs will be compiled and transmitted electronically as Level 4 data packages to the USGS Washington Water Science Center and Ecology's Manchester Environmental Laboratory. The Level 4 deliverable includes a written narrative, including any deviations from the methods, and all raw data needed to perform an independent review of the results (i.e. calibration reports, chromatograms and spectra for all calibration standards and samples, and bench sheets). In addition, the data will be delivered electronically in a format that is compatible for entry into Ecology's EIM database.

The Quality Assurance Manager at Ecology's Manchester lab will run USEPA Level 4 data validation on all of the chemical data as a 3rd-party independent reviewer, which includes Levels 1 through 4 as described in Table 7.

Table 7. Description of U.S. Environmental Protection Agency (EPA) data review validation levels, for the EIM field "Result_Validation_Level."

Valid Value	Description
EPA1	A verification and validation based only on completeness and compliance of sample receipt condition checks.
EPA2A	A verification and validation based on completeness and compliance checks of sample receipt conditions and ONLY sample-related QC results.
EPA2B	A verification and validation based on completeness and compliance checks of sample receipt conditions and BOTH sample-related and instrument-related QC results.
EPA3	A verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, AND recalculation checks.
EPA4	A verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, recalculation checks, AND the review of actual instrument outputs.

Data qualifiers may be applied to data by the laboratory (for example, those described in the Analytical Methods section). The Level 4 validator will translate the laboratory qualifiers or flags to the simplified EIM qualifiers consistent with Ecology's Toxics Cleanup Program data reporting protocols (Ecology, 2008) as outlined in the USEPA Functional Guidelines (USEPA 2016, 2014a, 2014b, 2014c, 2009). For example, EMPC results from HRMS methods (K-qualified) will be reviewed by the data validator and re-qualified as U or J-flagged results. Also, data will not be blank-corrected. Instead, a 5X rule (10X for common laboratory contaminants such as acetone, 2-butanone, methylene, chloride, toluene, and phthalate esters) will be applied to B-qualified results (indicating that the analyte was detected in the associated method blank). The result will be reported unqualified when it is greater than 5 times the level in the associated method blank. The result will be reported at the detected level with a U-containing qualifier when it is less than or equal to 5 times the level in the associated method blank. If there is more than one method blank associated with an environmental sample, the highest method blank concentration will be used.

In addition, an "R" qualifier will be allowed, indicating that "the sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control objectives. The presence or absence of the analyte cannot be verified" (USEPA 2016, 2014a, 2014b, 2014c, 2009).

Manchester's Quality Assurance Manager will submit to the USGS: 1) a Data Validation narrative describing the results of data validation review process, and 2) the EIM-compatible EDD (spreadsheet) updated with the Data Validator Qualifiers in the "Result_Qualifier" field. The original lab qualifiers will be retained in a "Result_Additional_Comment" field.

The USGS will review field parameters and data from USGS labs. The USGS data review and approval process follows the USGS' Fundamental Science Practices (http://www.usgs.gov/fsp/default.asp) to provide unbiased, objective, and impartial scientific information. The USGS will add field information to the EIM-formatted spreadsheet. Reviewed, validated, and approved chemistry data will be entered into the Ecology EIM database systems for long-term storage and public access. Reviewed, validated, and approved USGS-derived data (continuous stream gaging parameters, field parameters, SSC, and PSD) will be stored in NWIS for long-term storage and public access.

Reporting

Quarterly progress reports will be prepared by the USGS and submitted to the Ecology Project Manager. The data will be evaluated by the USGS to determine if the sampling design has been adequate and if it needs any modification for future use. For example, cumulative frequency diagrams of river discharge and turbidity during the project will be created and discrete sampling events will be plotted on each curve to assess how representative the sampling events were of the range of river conditions. Regression relations between continuous data (discharge and turbidity) and discrete data (instantaneous discharge, SSC, and chemical concentrations in water and sediment) that were developed in the first phases of the project will be refined through the addition of these 15 data points to provide a statistically-robust data set (approximately 35-40 data points). Estimates of sediment loading will be determined from the relation between discrete measurements of SSC and continuous turbidity and discharge records, according to USGS protocols (Rasmussen and others, 2009). Estimates of chemical loading may be determined using a variety of approaches including the USGS LOADEST program, multi-variate regression approaches, or alternate methods, such as Nonparametric Multiplicative Regression or Regression Trees, in which the chemical load is a function of the interaction of multiple environmental factors and does not assume any specific functional form (such as linear, logistic).

For models for which complete documentation cannot be provided in the report, a model archive will be created and stored at the USGS WAWSC. In addition, the archive will be provided to Ecology electronically. The model archive will fully describe and contain the model boundaries, input parameters, and statistical results to allow for reproduction of the model results, in accordance to the USGS Fundamental Science Practices and USGS Office of Water Quality Technical Memorandum 2015.01. The data used to develop the models and regressions will be stored in Ecology's publicly-available EIM database and NWIS for long-term storage and archiving. Data that is not able to be placed in EIM or NWIS for public access, such as the X-ray diffraction results and QA/QC results, will be released through a USGS Data Release for open and public access. The regressions will be used to provide instantaneous and annual estimates of loading of water, sediment, and sediment-bound contaminants from the Green River to the LDW.

Three final USGS citable products will be prepared, summarized in Table 8 and described below.

Table 8. Summary of final products.

Product	**Content**	**Date Prepared by**
USGS Open-File Report (or similar)	Sediment load estimates	12/31/2016
USGS Data Release	Phase 3 data	6/30/2017
USGS Scientific Investigations Report (or similar)	Chemical load estimates, tidal studies	6/30/2017

1) Sediment loading estimates, based on the regression between turbidity and SSC, will be prepared by December 31, 2016 in the form of an Open-File Report or similar on-line USGS citable product.

2) New data collected during Phase 3 that is not available for public and open access in Ecology's EIM database or the USGS NWIS database, will be prepared by June 30, 2017 in a USGS Data Release (also a USGS citable product).

3) Chemical load estimates and other interpretive results will be prepared by June 30, 2017 in the form of a USGS Scientific Investigations Report. The draft will contain loading calculations based on discharge, SSC and water chemistry concentrations (for unfiltered-water loading) or particulate-bound concentrations (for particulate-bound loading). For parameters that are not detected or detected infrequently during the study, statistics will be calculated to support loading calculations according to Helsel (2005). The results from this 2-year study, in combination with results from the previous 3 years of related research, will provide current estimates of sediment loading and chemical loading from the Green River to the LDW, and will inform future actions regarding watershed source control, remediation, and scientific investigations for improving these loading estimates.

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Appendix A. List of parameters and desired reporting limits (RL) and detection limits (DL) in water, sediment, filter, and XAD resin samples from the Duwamish River, WA.

Parameter Name	Water			Sediment		
	Unit	RL	DL	Unit	RL	DL
Organic Carbon, Total or Dissolved	mg/L	1.5	0.1	%	0.02	0.003
Low-level Mercury						
Mercury	ng/L	2	0.2	mg/kg	0.05	0.003
Metals						
Antimony	µg/L	0.2	0.01	mg/kg	0.5	0.04
Arsenic	µg/L	0.2	0.05	mg/kg	0.5	0.3
Barium	µg/L	0.5	0.02	mg/kg	1	0.2
Beryllium	µg/L	0.2	0.02	mg/kg	0.5	0.06
Cadmium	µg/L	0.1	0.01	mg/kg	0.3	0.04
Chromium	µg/L	0.5	0.04	mg/kg	1	0.12
Cobalt	µg/L	0.2	0.01	mg/kg	0.5	0.1
Copper	µg/L	0.5	0.2	mg/kg	1	0.1
Lead	µg/L	0.1	0.05	mg/kg	0.3	0.2
Manganese	µg/L	0.5	0.02	mg/kg	7	0.5
Molybdenum	µg/L	0.2	0.01	mg/kg	0.5	0.02
Nickel	µg/L	0.5	0.08	mg/kg	1	0.2
Selenium	µg/L	0.5	0.1	mg/kg	1.3	0.3
Silver	µg/L	0.2	0.01	mg/kg	0.5	0.03
Thallium	µg/L	0.2	0.004	mg/kg	0.5	0.05
Vanadium	µg/L	0.2	0.04	mg/kg	0.5	0.05
Zinc	µg/L	4	0.5	mg/kg	10	1
Ultra Low-level PAHs (LVI-SIM)				***Not measured in sediment***		
Naphthalene	µg/L	0.003	0.0015			
2-Methylnaphthalene	µg/L	0.001	0.0005			
1-Methylnaphthalene	µg/L	0.001	0.0005			
Acenaphthylene	µg/L	0.001	0.0005			
Acenaphthene	µg/L	0.001	0.0005			
Fluorene	µg/L	0.001	0.0005			
Phenanthrene	µg/L	0.001	0.0005			
Anthracene	µg/L	0.001	0.0005			
Fluoranthene	µg/L	0.001	0.0005			
Pyrene	µg/L	0.001	0.0005			
Benzo(a)anthracene	µg/L	0.001	0.0005			
Chrysene	µg/L	0.001	0.0005			
Benzo(a)pyrene	µg/L	0.001	0.0005			

Parameter Name	Water			Sediment		
	Unit	RL	DL	Unit	RL	DL
Indeno(1,2,3-cd)pyrene	µg/L	0.001	0.0005			
Dibenz(a,h)anthracene	µg/L	0.001	0.0005			
Benzo(g,h,i)perylene	µg/L	0.001	0.0005			
Dibenzofuran	µg/L	0.001	0.0005			
Benzo[b,j,k]fluoranthene	µg/L	0.001	0.0005			
Low-level PAHs (SIM)						
Naphthalene	µg/L	0.03	0.002	µg/kg	0.5	0.3
2-Methylnaphthalene	µg/L	0.01	0.001	µg/kg	0.5	0.1
1-Methylnaphthalene	µg/L	0.01	0.001	µg/kg	0.5	0.2
Acenaphthylene	µg/L	0.01	0.001	µg/kg	0.5	0.2
Acenaphthene	µg/L	0.01	0.001	µg/kg	0.5	0.2
Fluorene	µg/L	0.01	0.001	µg/kg	0.5	0.2
Phenanthrene	µg/L	0.01	0.001	µg/kg	0.5	0.2
Anthracene	µg/L	0.01	0.001	µg/kg	0.5	0.2
Fluoranthene	µg/L	0.01	0.001	µg/kg	0.5	0.2
Pyrene	µg/L	0.01	0.001	µg/kg	0.5	0.3
Benzo(a)anthracene	µg/L	0.01	0.001	µg/kg	0.5	0.3
Chrysene	µg/L	0.01	0.001	µg/kg	0.5	0.3
Benzo(a)pyrene	µg/L	0.01	0.001	µg/kg	0.5	0.1
Indeno(1,2,3-cd)pyrene	µg/L	0.01	0.001	µg/kg	0.5	0.2
Dibenz(a,h)anthracene	µg/L	0.01	0.001	µg/kg	0.5	0.2
Benzo(g,h,i)perylene	µg/L	0.01	0.001	µg/kg	0.5	0.2
Dibenzofuran	µg/L	0.01	0.001	µg/kg	0.5	0.2
Benzo[b,j,k]fluoranthene	µg/L	0.02	0.002	µg/kg	1.0	0.5
Semivolatiles						
Phenol	µg/L	0.2	0.010	µg/kg	5.0	3.67
Bis-(2-Chloroethyl) Ether	µg/L	0.2	0.028	µg/kg	20	6.6
2-Chlorophenol	µg/L	0.2	0.029	µg/kg	20	6.3
1,3-Dichlorobenzene	µg/L	0.2	0.031	µg/kg	5.0	1.30
1,4-Dichlorobenzene	µg/L	0.2	0.028	µg/kg	5.0	1.91
Benzyl Alcohol	µg/L	0.2	0.023	µg/kg	20	12.1
1,2-Dichlorobenzene	µg/L	0.2	0.033	µg/kg	5.0	1.32
2-Methylphenol	µg/L	0.2	0.027	µg/kg	5.0	1.92
2,2'-Oxybis(1-Chloropropane)	µg/L	0.2	0.028	µg/kg	20	5.5
4-Methylphenol	µg/L	0.2	0.029	µg/kg	5.0	2.53
N-Nitroso-Di-N-Propylamine	µg/L	0.2	0.035	µg/kg	20.0	15.1
Hexachloroethane	µg/L	0.2	0.037	µg/kg	20	5.5
Nitrobenzene	µg/L	0.2	0.027	µg/kg	20	7.7
Isophorone	µg/L	0.2	0.031	µg/kg	20	7.5
2-Nitrophenol	µg/L	1.0	0.036	µg/kg	20	6.7
2,4-Dimethylphenol	µg/L	1.0	0.27	µg/kg	25.0	10.2

Parameter Name	**Water**			**Sediment**		
	Unit	**RL**	**DL**	**Unit**	**RL**	**DL**
Benzoic Acid	µg/L	2.0	0.13	µg/kg	200	57
bis(2-Chloroethoxy) Methane	µg/L	0.2	0.030	µg/kg	20	6.2
2,4-Dichlorophenol	µg/L	1.0	0.10	µg/kg	99	31
1,2,4-Trichlorobenzene	µg/L	0.2	0.032	µg/kg	5.0	1.51
4-Chloroaniline	µg/L	1.0	0.042	µg/kg	99	33
Hexachlorobutadiene	µg/L	0.2	0.038	µg/kg	5.0	1.42
4-Chloro-3-methylphenol	µg/L	1.0	0.13	µg/kg	99	28
2-Methylnaphthalene	µg/L	0.2	0.029	µg/kg	20	5.5
Hexachlorocyclopentadiene	µg/L	1.0	0.14	µg/kg	99	40
2,4,6-Trichlorophenol	µg/L	1.0	0.16	µg/kg	99	25
2,4,5-Trichlorophenol	µg/L	1.0	0.13	µg/kg	99	26
2-Chloronaphthalene	µg/L	0.2	0.030	µg/kg	20	4.3
2-Nitroaniline	µg/L	1.0	0.17	µg/kg	99	29
Dimethylphthalate	µg/L	0.2	0.035	µg/kg	5.0	1.21
3-Nitroaniline	µg/L	1.0	0.15	µg/kg	99	37
2,4-Dinitrophenol	µg/L	2.0	0.22	µg/kg	200	40
4-Nitrophenol	µg/L	1.0	0.056	µg/kg	99	43
2,6-Dinitrotoluene	µg/L	1.0	0.17	µg/kg	99	26
2,4-Dinitrotoluene	µg/L	1.0	0.11	µg/kg	99	22
Diethylphthalate	µg/L	0.2	0.060	µg/kg	20.0	19.9
4-Chlorophenyl-phenylether	µg/L	0.2	0.020	µg/kg	20	6.8
4-Nitroaniline	µg/L	1.0	0.17	µg/kg	99	34
4,6-Dinitro-2-Methylphenol	µg/L	2.0	0.36	µg/kg	200	49
N-Nitrosodiphenylamine	µg/L	0.2	0.025	µg/kg	5.0	2.31
4-Bromophenyl-phenylether	µg/L	0.2	0.019	µg/kg	20	5.9
Hexachlorobenzene	µg/L	0.2	0.036	µg/kg	5.0	2.11
Pentachlorophenol	µg/L	1.0	0.14	µg/kg	20	10.4
Carbazole	µg/L	0.2	0.037	µg/kg	20	7.2
Di-n-Butylphthalate	µg/L	0.2	0.051	µg/kg	20	5.2
Butylbenzylphthalate	µg/L	0.2	0.066	µg/kg	5.0	2.18
3,3'-Dichlorobenzidine	µg/L	1.0	0.34	µg/kg	99	30
bis(2-Ethylhexyl)phthalate	µg/L	0.2	0.163	µg/kg	49	28
Di-n-Octyl phthalate	µg/L	0.2	0.045	µg/kg	20	8.5
Butyltins						
Tributyltin Ion	µg/L	0.2	0.05	µg/kg	3.7	1.5
Dibutyltin Ion	µg/L	0.3	0.1	µg/kg	5.6	3.6
Butyltin	µg/L	0.2	0.15	µg/kg	3.9	2.9
Dioxins/Furans						
2,3,7,8-TCDD	pg/L	3	0.6	pg/g	0.2	0.1
1,2,3,7,8-PECDD	pg/L	12	0.6	pg/g	1	0.1
1,2,3,4,7,8-HXCDD	pg/L	12	0.6	pg/g	1	0.1

1,2,3,6,7,8-HXCDD	pg/L	12	0.6	pg/g	1	0.1
1,2,3,7,8,9-HXCDD	pg/L	12	0.6	pg/g	1	0.1
1,2,3,4,6,7,8-HPCDD	pg/L	12	0.6	pg/g	1	0.1
OCDD	pg/L	25	0.6	pg/g	2	0.1
2,3,7,8-TCDF	pg/L	25	0.6	pg/g	0.2	0.1
1,2,3,7,8-PECDF	pg/L	12	0.6	pg/g	1	0.1
2,3,4,7,8-PECDF	pg/L	12	0.6	pg/g	1	0.1
1,2,3,4,7,8-HXCDF	pg/L	12	0.6	pg/g	1	0.1
1,2,3,6,7,8-HXCDF	pg/L	12	0.6	pg/g	1	0.1
1,2,3,7,8,9-HXCDF	pg/L	12	0.6	pg/g	1	0.1
2,3,4,6,7,8-HXCDF	pg/L	12	0.6	pg/g	1	0.1
1,2,3,4,6,7,8-HPCDF	pg/L	12	0.6	pg/g	1	0.1
1,2,3,4,7,8,9-HPCDF	pg/L	12	0.6	pg/g	1	0.1
OCDF	pg/L	25	0.6	pg/g	2	0.1
Parameter Name	**Water, Filter, XAD**			**Sediment**		
	Unit[a]	**RL**	**DL**	**Unit**	**RL**	**DL**
209 PCB Congeners						
PCB-001	pg/L [a]	5	2	pg/g	0.5	0.2
PCB-002	pg/L [a]	5	2	pg/g	0.5	0.2
PCB-003	pg/L [a]	5	2	pg/g	0.5	0.2
PCB-004	pg/L [a]	5	4	pg/g	0.5	0.2
PCB-005	pg/L [a]	5	3	pg/g	0.5	0.2
PCB-006	pg/L [a]	5	3	pg/g	0.5	0.2
PCB-007	pg/L [a]	5	3	pg/g	0.5	0.2
PCB-008	pg/L [a]	5	3	pg/g	0.5	0.2
PCB-009	pg/L [a]	5	3	pg/g	0.5	0.2
PCB-010	pg/L [a]	5	3	pg/g	0.5	0.2
PCB-011	pg/L [a]	5	3	pg/g	0.5	0.2
PCB-012/013	pg/L [a]	5	3	pg/g	0.5	0.2
PCB-014	pg/L [a]	5	3	pg/g	0.5	0.2
PCB-015	pg/L [a]	5	3	pg/g	0.5	0.2
PCB-016	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-017	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-018/030	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-019	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-020/028	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-021/033	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-022	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-023	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-024	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-025	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-026/029	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-027	pg/L [a]	5	1	pg/g	0.5	0.1

PCB-031	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-032	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-034	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-035	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-036	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-037	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-038	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-039	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-040/041/071	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-042	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-043	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-044/047/065	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-045/051	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-046	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-048	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-049/069	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-050/053	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-052	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-054	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-055	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-056	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-057	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-058	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-059/062/075	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-060	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-061/070/074/076	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-063	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-064	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-066	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-067	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-068	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-072	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-073	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-077	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-078	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-079	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-080	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-081	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-082	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-083/099	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-084	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-085/116/117	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-086/087/097/109/119/125	pg/L [a]	5	1	pg/g	0.5	0.2

PCB-088/091	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-089	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-090/101/113	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-092	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-093/095/098/100/102	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-094	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-096	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-103	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-104	pg/L [a]	5	1	pg/g	0.5	0.2
PCB-105	pg/L [a]	5	1	pg/g	0.5	1
PCB-106	pg/L [a]	5	1	pg/g	0.5	1
PCB-107	pg/L [a]	5	1	pg/g	0.5	1
PCB-108/124	pg/L [a]	5	1	pg/g	0.5	1
PCB-110/115	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-111	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-112	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-114	pg/L [a]	5	1	pg/g	0.5	1
PCB-118	pg/L [a]	5	1	pg/g	0.5	1
PCB-120	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-121	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-122	pg/L [a]	5	1	pg/g	0.5	1
PCB-123	pg/L [a]	5	1	pg/g	0.5	1
PCB-126	pg/L [a]	5	1	pg/g	0.5	1
PCB-127	pg/L [a]	5	1	pg/g	0.5	1
PCB-128/166	pg/L [a]	5	1	pg/g	0.5	1
PCB-129/138/160/163	pg/L [a]	5	1	pg/g	0.5	1
PCB-130	pg/L [a]	5	1	pg/g	0.5	1
PCB-131	pg/L [a]	5	1	pg/g	0.5	1
PCB-132	pg/L [a]	5	1	pg/g	0.5	1
PCB-133	pg/L [a]	5	1	pg/g	0.5	1
PCB-134/143	pg/L [a]	5	1	pg/g	0.5	1
PCB-135/151/154	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-136	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-137	pg/L [a]	5	1	pg/g	0.5	1
PCB-139/140	pg/L [a]	5	1	pg/g	0.5	1
PCB-141	pg/L [a]	5	1	pg/g	0.5	1
PCB-142	pg/L [a]	5	1	pg/g	0.5	1
PCB-144	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-145	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-146	pg/L [a]	5	1	pg/g	0.5	1
PCB-147/149	pg/L [a]	5	1	pg/g	0.5	1
PCB-148	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-150	pg/L [a]	5	1	pg/g	0.5	0.1

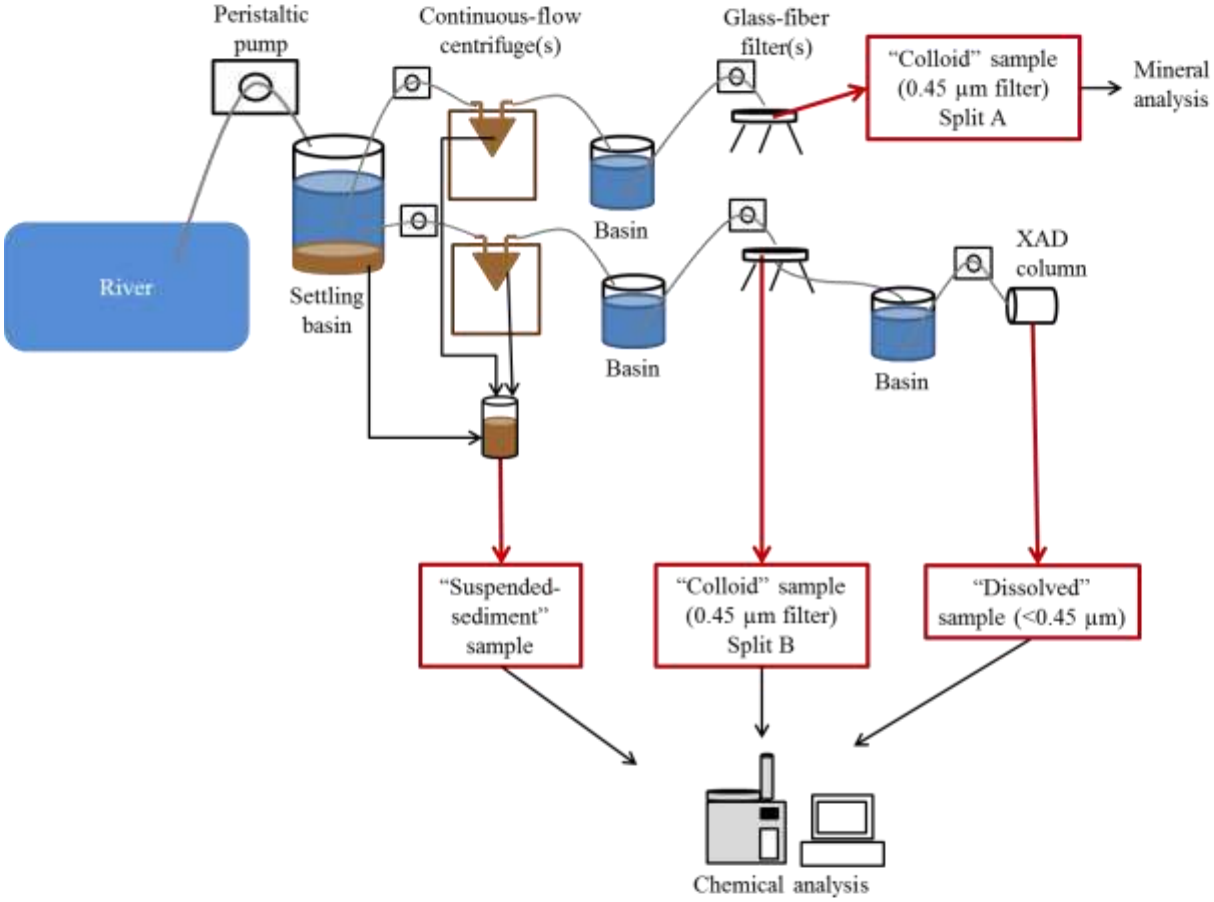
PCB-152	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-153/168	pg/L [a]	5	1	pg/g	0.5	1
PCB-155	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-156/157	pg/L [a]	10	1	pg/g	1	1
PCB-158	pg/L [a]	5	1	pg/g	0.5	1
PCB-159	pg/L [a]	5	1	pg/g	0.5	1
PCB-161	pg/L [a]	5	1	pg/g	0.5	1
PCB-162	pg/L [a]	5	1	pg/g	0.5	1
PCB-164	pg/L [a]	5	1	pg/g	0.5	1
PCB-165	pg/L [a]	5	1	pg/g	0.5	1
PCB-167	pg/L [a]	5	1	pg/g	0.5	1
PCB-169	pg/L [a]	5	1	pg/g	0.5	1
PCB-170	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-171/173	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-172	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-174	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-175	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-176	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-177	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-178	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-179	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-180/193	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-181	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-182	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-183/185	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-184	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-186	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-187	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-188	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-189	pg/L [a]	5	1	pg/g	0.5	1
PCB-190	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-191	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-192	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-194	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-195	pg/L [a]	5	1	pg/g	0.5	1
PCB-196	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-197/200	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-198/199	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-201	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-202	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-203	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-204	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-205	pg/L [a]	5	1	pg/g	0.5	0.1

PCB-206	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-207	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-208	pg/L [a]	5	1	pg/g	0.5	0.1
PCB-209	pg/L [a]	5	1	pg/g	0.5	0.1

[a] PCB units for filter and XAD are pg/sample

Appendix B. Summary of USGS Standard Operating Procedure for the collection and concentration of suspended-sediment samples by continuous-flow centrifugation

The protocol for the collection and processing of suspended sediment by continuous-flow centrifugation in the field is summarized in the following schematic and described below:



1. Equipment

1.1 Field Equipment

- Tubing, Teflon, smooth wall (0.625 OD x 0.062 wall, 0.500 ID), 50-100 ft, methanol-cleaned
- ISCO 6712 Autosampler or similar unscreened, high-flow pump
- ISCO stainless steel strainer
- 2 to 6 12-V batteries, charged
- 1-6 30-gal polyethylene drums and lids (1 lid has a center hole for pump tubing)

- 2-10 30-gal Teflon drum liners, methanol-cleaned
- Continuous-flow centrifuge(s) (CFC Express, Scientific Methods, Inc.)
- AC Power or 2-6 charged 12-V batteries
- 2-6 centrifuge bowls
- Peristaltic pump(s) with C-FLEX tubing, methanol-cleaned
- 1-6 pieces of Teflon tubing (small diameter to fit in C-FLEX tubing, ~5-10 ft) with small glass funnel attached, both methanol-cleaned
- 1-6 "float systems" to keep the glass funnel suspended near the water surface in the drum, consisting of:
    - 1-L Teflon bottle
    - small piece of C-FLEX tubing to funnel tubing around float bottle
      Note: both pieces should be methanol-cleaned on the outside
- 2 pieces of tubing, any kind, to drain centrifuge outflow water
- 100 mL graduated cylinder
- Stopwatch
- 32-oz tall wide-mouth glass jar
- Ice chest
- Squirt bottle, PFA
- Organic-free water
- (Optional) YSI 6290 or similar multi-parameter sonde with 100' cable
- (Optional) Various bottles and jars for additional parameters
- Safety equipment
    - Traffic control plan, as applicable
    - Cones and/or Men Working signs
    - High-visibility clothing
    - PFDs with whistle
    - Throw ropes
- Field folder
    - Field forms
    - Bottle labels
    - Rite-in-rain notebook
    - Rite-in-rain pens, pencils, sharpies
- Gloves of various sizes
- Tech wipes
- Deionized water jug
- Deionized water squirt bottle
- Bags of various sizes
- Flagging tape
- Electrical tape

- Scissors/knife
- Tagline/measuring tape
- Tool bag

1.2 Lab Processing Equipment

- Squirt bottle, PFA
- Organic-free water
- Amber glass jars, for sub-samples
- Glass pipette, 50-mL or 100-mL, methanol-cleaned
- Clay Adams Brand Dynac Centrifuge Model 420102
- 4-8 Teflon or glass centrifuge tubes to fit 101 x 41.4 mm rotor, methanol-cleaned

2. Sample Collection and Processing

2.1 Implement safety plan, which includes:

2.1.1 All field personnel wear PFDs with whistle, current on surface water safety training

2.1.2 Implement Traffic Control Plan using cones and/or signs, with designated traffic signalers as necessary. For the Green River study, there is limited vehicle traffic at both stations.

2.1.3 During all times when equipment is in the water, and especially during high-flow storm events, one person will monitor upstream for large floating debris

2.2 When handling methanol-cleaned parts that will come in contact with the river water, don clean nitrile gloves. This includes both ends of the Teflon ISCO intake tubing, both ends of the ISCO pump tubing, the Teflon drum liners, the float system and small-diameter Teflon tubing, both ends of the C-FLEX tubing, both ports of the centrifuge bowl(s), and the sample jar.

2.3 Thread the pre-cleaned intake tubing through the permanent bank tubing to desired in-river location

2.4 Attach other end of intake tubing to ISCO pump tubing (Note: vertical pumping limit of ISCO is approximately 28 ft)

2.5 Turn on ISCO pump and flush tubing for approximately 3-5 minutes

2.6 Set up drum, drum liner, small-diameter Teflon tubing with glass funnel, float system, C-FLEX tubing in peristaltic pump, centrifuge (with power) with bowl and outflow tubing

2.7 Begin collecting river water in drum (record sample collection start time) according to desired pump program (for example, continuous pumping or cycling pumping)

2.8 Adjust peristaltic pump flow rate to 600 mL/min using stopwatch and graduated cylinder (for example, 100 mL in 10 seconds)

2.9 Continue pumping until sufficient water has been collected for sediment chemical analysis

2.10 Transfer bowl contents to sample jar regularly (every 2-12 hours, depending on river conditions) and store jar quiescently at 4 °C

2.11 Monitor pumping and processing equipment and replace batteries, tubing, etc. as needed

2.12 Collect field parameters and samples for other desired parameters

2.13 When sample collection is done, turn off ISCO pump and record sample collection end time

2.14 Continue pumping water through the centrifuge until there is less than 1 L of water remaining in the drum

2.15 Turn off the peristaltic pump and centrifuge

2.16 Transfer bowl contents and drum contents into the sample jar and store on ice

2.17 Dissemble equipment and load into vehicles for transport to laboratory

3. Laboratory Processing

3.1 Store the sample jar at 4°C overnight to settle

3.2 Pipette the overlying water into Teflon or glass centrifuge tubes

3.3 Spin in floor centrifuge for 20 minutes at 1000 x g or greater

3.4 Transfer any centrifuged sediment in the bottom of tubes to the sample jar

3.5 Homogenize the sample using Teflon spatula and sub-sample into separate sample containers as needed

3.6 Ship samples on ice with paperwork to analytical lab(s)

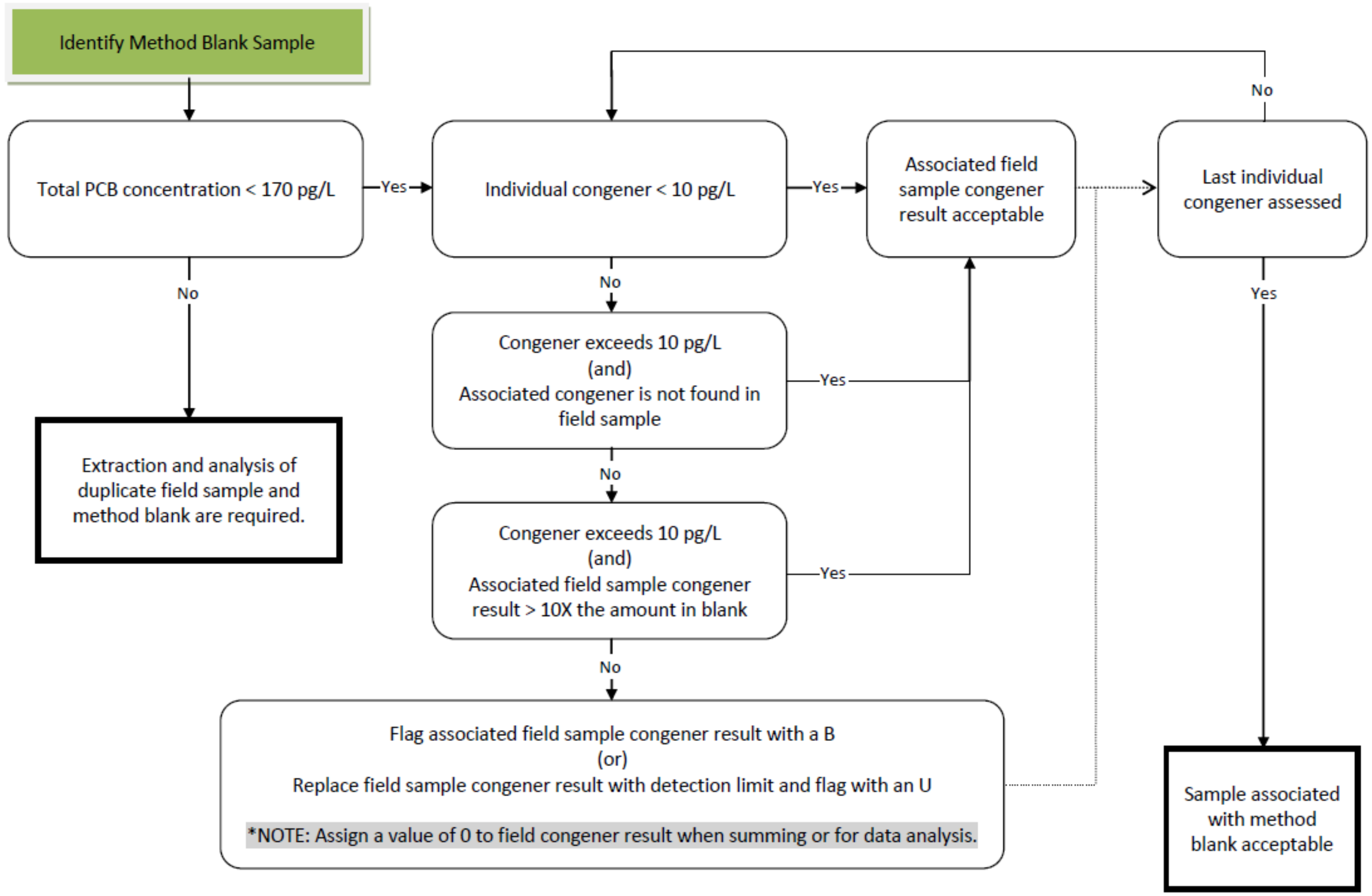
4. Preparation for Subsequent Sampling

- Wash Teflon tubing, drum liner, funnel, float system, and C-FLEX tubing as follows:
    - Wash and scrub using phosphate-free soap and warm tap water
    - Rinse with tap water
    - Soak for at least 30 minutes in 5% hydrochloric acid
    - Rinse with deionized water
    - Rinse with trace-grade methanol.
    - Allow to air-dry, then bag.
- Rinse river water and mud from other field equipment; air-dry
- Charge batteries
- Store cleaned equipment for next sampling event

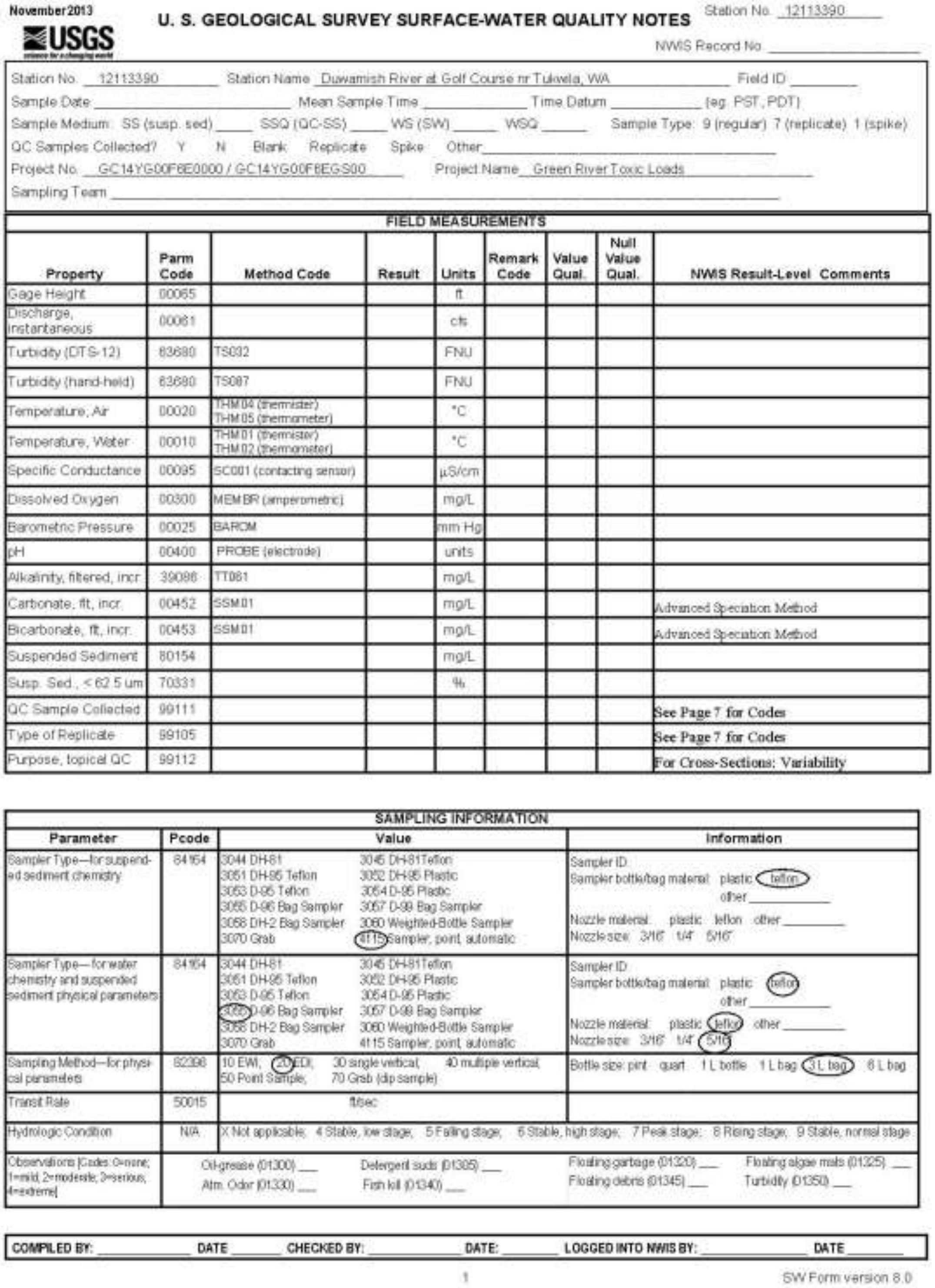
Appendix C. Polychlorinated biphenyl (PCB) Method Blank Contamination Decision Rules

These rules, developed for Spokane River, WA, will be applied to this Green River project, using 5X instead of 10X (second column, 3rd box).

METHOD BLANK CONTAMINATION DECISION RULES



Appendix D. Example USGS field form.



November 2013

USGS

U. S. GEOLOGICAL SURVEY SURFACE-WATER QUALITY NOTES

Station No. 12113390

NWIS Record No. ______________

Station No. 12113390 Station Name Duwamish River at Golf Course nr Tukwila, WA Field ID ________

Sample Date ____________________ Mean Sample Time ____________ Time Datum __________ (eg. PST, PDT)

Sample Medium: SS (susp. sed) _____ SSQ (QC-SS) _____ WS (SW) _____ WSQ _____ Sample Type: 9 (regular) 7 (replicate) 1 (spike)

QC Samples Collected? Y N Blank Replicate Spike Other ______________________________

Project No. GC14YG00F8E0000 / GC14YG00F8EGS00 Project Name Green River Toxic Loads

Sampling Team ____

FIELD MEASUREMENTS

Property	Parm Code	Method Code	Result	Units	Remark Code	Value Qual.	Null Value Qual.	NWIS Result-Level Comments
Gage Height	00065			ft				
Discharge, instantaneous	00061			cfs				
Turbidity (DTS-12)	63680	TS032		FNU				
Turbidity (hand-held)	63680	TS087		FNU				
Temperature, Air	00020	THM04 (thermister) THM05 (thermometer)		°C				
Temperature, Water	00010	THM01 (thermister) THM02 (thermometer)		°C				
Specific Conductance	00095	SC001 (contacting sensor)		µS/cm				
Dissolved Oxygen	00300	MEMBR (amperometric)		mg/L				
Barometric Pressure	00025	BAROM		mm Hg				
pH	00400	PROBE (electrode)		units				
Alkalinity, filtered, incr	39086	TT081		mg/L				
Carbonate, flt, incr	00452	SSM01		mg/L				Advanced Speciation Method
Bicarbonate, flt, incr	00453	SSM01		mg/L				Advanced Speciation Method
Suspended Sediment	80154			mg/L				
Susp. Sed., < 62.5 um	70331			%				
QC Sample Collected	99111							See Page 7 for Codes
Type of Replicate	99105							See Page 7 for Codes
Purpose, topical QC	99112							For Cross-Sections: Variability

SAMPLING INFORMATION

Parameter	Pcode	Value	Information
Sampler Type—for suspended sediment chemistry	84164	3044 DH-81 3045 DH-81Teflon 3051 DH-95 Teflon 3052 DH-95 Plastic 3053 D-95 Teflon 3054 D-95 Plastic 3055 D-96 Bag Sampler 3057 D-99 Bag Sampler 3058 DH-2 Bag Sampler 3060 Weighted-Bottle Sampler 3070 Grab [✓ 4115] Sampler, point, automatic	Sampler ID Sampler bottle/bag material: plastic [✓ teflon] other ________ Nozzle material: plastic teflon other ________ Nozzle size: 3/16" 1/4" 5/16"
Sampler Type—for water chemistry and suspended sediment physical parameters	84164	3044 DH-81 3045 DH-81Teflon 3051 DH-95 Teflon 3052 DH-95 Plastic 3053 D-95 Teflon 3054 D-95 Plastic [✓ 3055] D-96 Bag Sampler 3057 D-99 Bag Sampler 3058 DH-2 Bag Sampler 3060 Weighted-Bottle Sampler 3070 Grab 4115 Sampler, point, automatic	Sampler ID Sampler bottle/bag material: plastic [✓ teflon] other ________ Nozzle material: plastic [✓ teflon] other ________ Nozzle size: 3/16" 1/4" [✓ 5/16"]
Sampling Method—for physical parameters	82398	10 EWI; [✓ 20] EDI; 30 single vertical; 40 multiple vertical; 50 Point Sample; 70 Grab (dip sample)	Bottle size: pint quart 1 L bottle 1 L bag [✓ 3 L bag] 8 L bag
Transit Rate	50015	ft/sec	
Hydrologic Condition	N/A	X Not applicable; 4 Stable, low stage; 5 Falling stage; 6 Stable, high stage; 7 Peak stage; 8 Rising stage; 9 Stable, normal stage	
Observations [Codes: 0=none; 1=mild; 2=moderate; 3=serious; 4=extreme]		Oil-grease (01300) ___ Detergent suds (01305) ___ Atm. Odor (01330) ___ Fish kill (01340) ___	Floating garbage (01320) ___ Floating algae mats (01325) ___ Floating debris (01345) ___ Turbidity (01350) ___

COMPILED BY: __________ **DATE** ______ **CHECKED BY:** __________ **DATE:** ______ **LOGGED INTO NWIS BY:** __________ **DATE** ______

1

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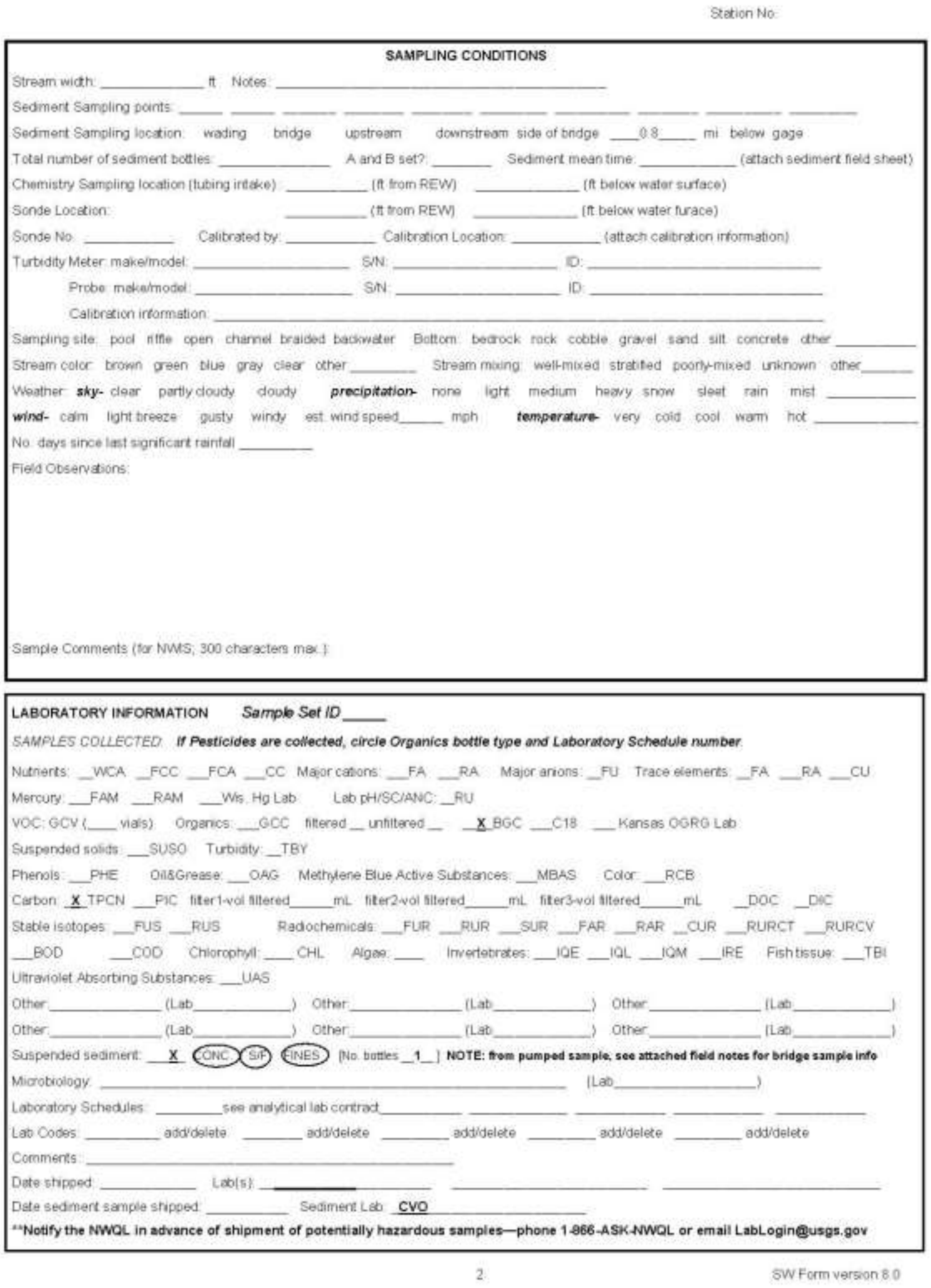


Station No:

SAMPLING CONDITIONS

Stream width: ______________ ft Notes: ____

Sediment Sampling points: ______ ______ _______ ________ ________ _________ __________ ________ __________ _________

Sediment Sampling location: wading bridge upstream downstream side of bridge ____0.8_____ mi below gage

Total number of sediment bottles: ________________ A and B set? ________ Sediment mean time: _____________ (attach sediment field sheet)

Chemistry Sampling location (tubing intake): ___________ (ft from REW) ______________ (ft below water surface)

Sonde Location: ___________ (ft from REW) ______________ (ft below water furace)

Sonde No: ____________ Calibrated by: ____________ Calibration Location: ____________ (attach calibration information)

Turbidity Meter: make/model: _____________________ S/N: ______________________ ID: _______________________________

Probe: make/model: _____________________ S/N: ______________________ ID: _______________________________

Calibration information: ____

Sampling site: pool riffle open channel braided backwater Bottom: bedrock rock cobble gravel sand silt concrete other ___________

Stream color: brown green blue gray clear other _________ Stream mixing: well-mixed stratified poorly-mixed unknown other_______

Weather: ***sky-*** clear partly cloudy cloudy ***precipitation-*** none light medium heavy snow sleet rain mist ____________

wind- calm light breeze gusty windy est. wind speed______ mph ***temperature-*** very cold cool warm hot ______________

No. days since last significant rainfall __________

Field Observations:

Sample Comments (for NWIS; 300 characters max.):

LABORATORY INFORMATION ***Sample Set ID*** _____

SAMPLES COLLECTED: ***If Pesticides are collected, circle Organics bottle type and Laboratory Schedule number.***

Nutrients: __WCA __FCC __FCA __CC Major cations: __FA __RA Major anions: __FU Trace elements: __FA __RA __CU

Mercury: __FAM __RAM __Wis. Hg Lab Lab pH/SC/ANC: __RU

VOC: GCV (____ vials) Organics: __GCC filtered __ unfiltered __ __X_BGC __C18 __ Kansas OGRG Lab

Suspended solids: __SUSO Turbidity: __TBY

Phenols: __PHE Oil&Grease: __OAG Methylene Blue Active Substances: __MBAS Color: __RCB

Carbon: _X_TPCN __PIC filter1-vol filtered______mL filter2-vol filtered______mL filter3-vol filtered______mL __DOC __DIC

Stable isotopes: __FUS __RUS Radiochemicals: __FUR __RUR __SUR __FAR __RAR __CUR __RURCT __RURCV

__BOD __COD Chlorophyll: ____CHL Algae: ____ Invertebrates: __IQE __IQL __IQM __IRE Fish tissue: __TBI

Ultraviolet Absorbing Substances: __UAS

Other:_______________ (Lab______________) Other:_______________ (Lab______________) Other:_______________ (Lab______________)

Other:_______________ (Lab______________) Other:_______________ (Lab______________) Other:_______________ (Lab______________)

Suspended sediment: __X__ [✓ CONC] [✓ S/F] [✓ FINES] [No. bottles __1__] **NOTE: from pumped sample, see attached field notes for bridge sample info**

Microbiology: ____ (Lab__________________)

Laboratory Schedules: _________see analytical lab contract__________ ____________ ____________ ____________ ____________

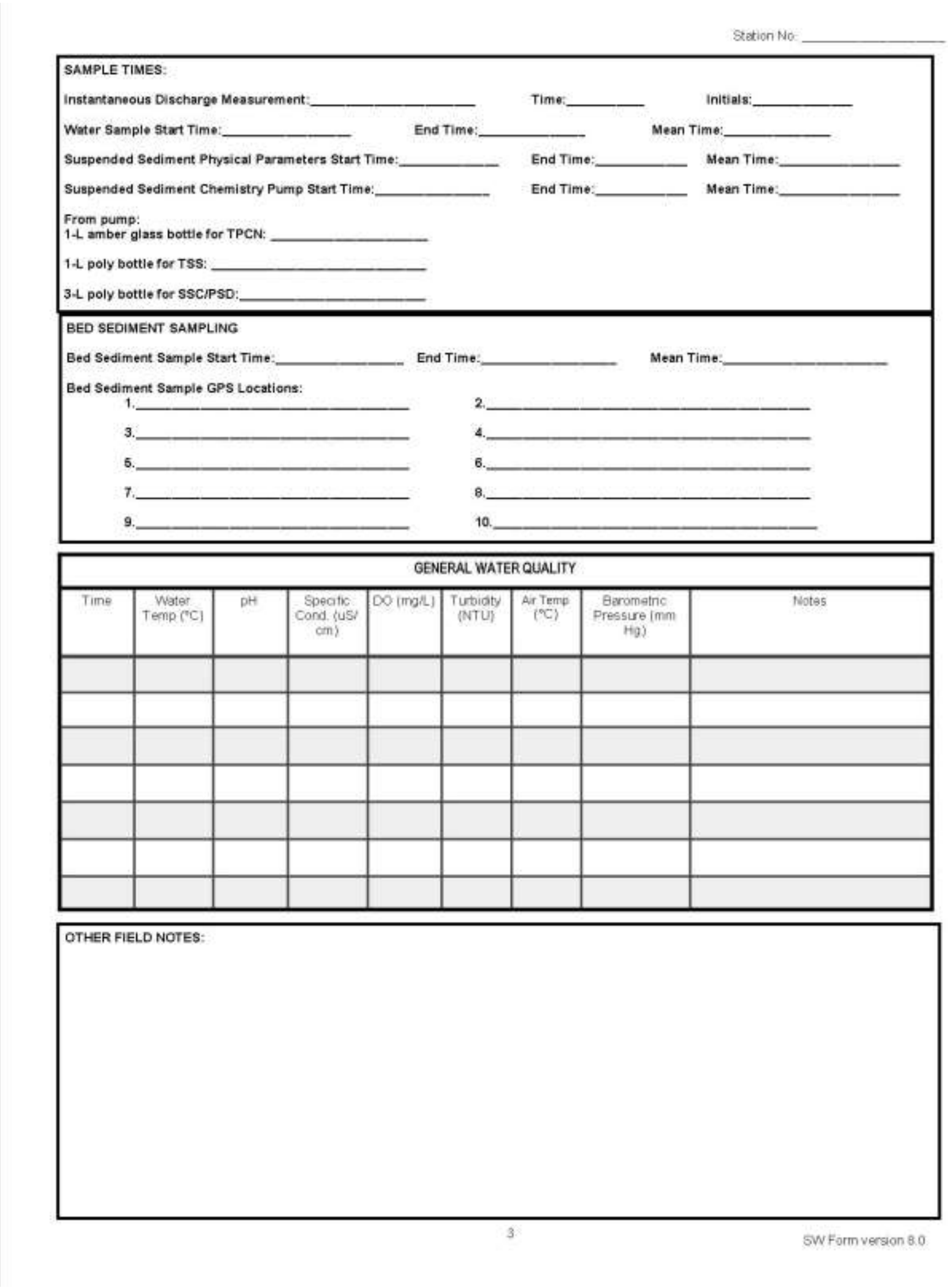
Lab Codes: __________ add/delete _________ add/delete _________ add/delete _________ add/delete _________ add/delete

Comments: ____

Date shipped: ____________ Lab(s): ________________ ______________________ ____________________________

Date sediment sample shipped: ___________ Sediment Lab: **CVO** ____________________

****Notify the NWQL in advance of shipment of potentially hazardous samples—phone 1-866-ASK-NWQL or email LabLogin@usgs.gov**



Station No: ____________

SAMPLE TIMES:

Instantaneous Discharge Measurement:____________ **Time:**________ **Initials:**________

Water Sample Start Time:________ **End Time:**________ **Mean Time:**________

Suspended Sediment Physical Parameters Start Time:________ **End Time:**________ **Mean Time:**________

Suspended Sediment Chemistry Pump Start Time:________ **End Time:**________ **Mean Time:**________

From pump:
1-L amber glass bottle for TPCN: ____________

1-L poly bottle for TSS: ____________

3-L poly bottle for SSC/PSD:____________

BED SEDIMENT SAMPLING

Bed Sediment Sample Start Time:________ **End Time:**________ **Mean Time:**________

Bed Sediment Sample GPS Locations:

1.____________ 2.____________

3.____________ 4.____________

5.____________ 6.____________

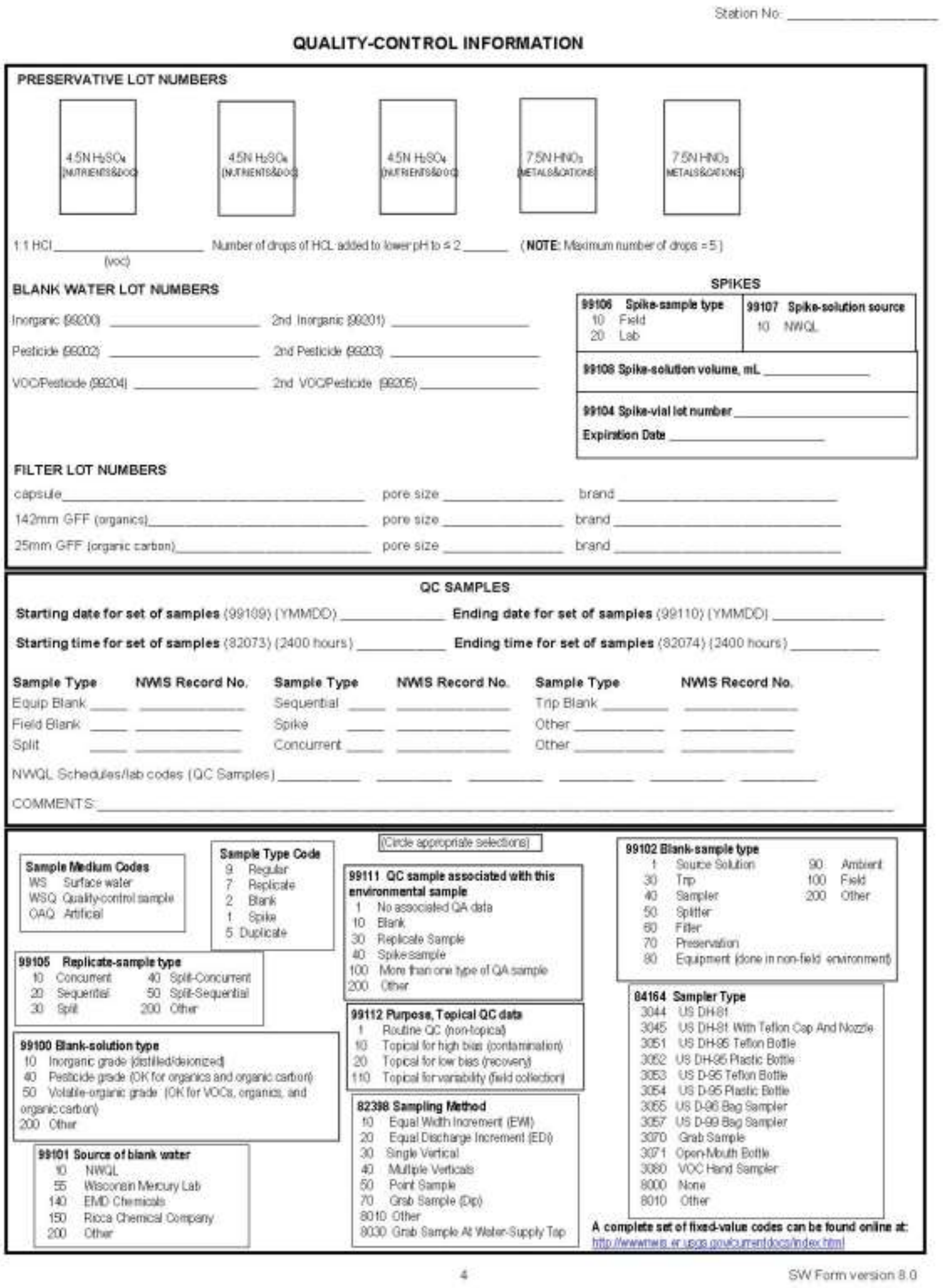
7.____________ 8.____________

9.____________ 10.____________

GENERAL WATER QUALITY

Time	Water Temp (°C)	pH	Specific Cond. (uS/cm)	DO (mg/L)	Turbidity (NTU)	Air Temp (°C)	Barometric Pressure (mm Hg)	Notes

OTHER FIELD NOTES:



Station No: ____________________

QUALITY-CONTROL INFORMATION

PRESERVATIVE LOT NUMBERS

4.5N H_2SO_4 (NUTRIENTS&DOC)	4.5N H_2SO_4 (NUTRIENTS&DOC)	4.5N H_2SO_4 (NUTRIENTS&DOC)	7.5N HNO_3 (METALS&CATIONS)	7.5N HNO_3 (METALS&CATIONS)

1:1 HCl ____________________ (voc) Number of drops of HCL added to lower pH to ≤ 2 ______ (**NOTE**: Maximum number of drops = 5)

BLANK WATER LOT NUMBERS

Inorganic (99200) ____________________ 2nd Inorganic (99201) ____________________

Pesticide (99202) ____________________ 2nd Pesticide (99203) ____________________

VOC/Pesticide (99204) ____________________ 2nd VOC/Pesticide (99205) ____________________

SPIKES

99106 Spike-sample type
10 Field
20 Lab

99107 Spike-solution source
10 NWQL

99108 Spike-solution volume, mL ______________

99104 Spike-vial lot number ____________________

Expiration Date ____________________

FILTER LOT NUMBERS

capsule ____________________ pore size ______________ brand ____________________

142mm GFF (organics) ____________________ pore size ______________ brand ____________________

25mm GFF (organic carbon) ____________________ pore size ______________ brand ____________________

QC SAMPLES

Starting date for set of samples (99109) (YMMDD) ______________ **Ending date for set of samples** (99110) (YMMDD) ______________

Starting time for set of samples (82073) (2400 hours) ____________ **Ending time for set of samples** (82074) (2400 hours) ____________

Sample Type		**NWIS Record No.**	**Sample Type**		**NWIS Record No.**	**Sample Type**		**NWIS Record No.**
Equip Blank	_____	____________	Sequential	_____	____________	Trip Blank	________	____________
Field Blank	_____	____________	Spike	_____	____________	Other	________	____________
Split	_____	____________	Concurrent	_____	____________	Other	________	____________

NWQL Schedules/lab codes (QC Samples) __________ __________ __________ __________ __________ __________

COMMENTS: ____

(Circle appropriate selections)

Sample Medium Codes

WS	Surface water
WSQ	Quality-control sample
OAQ	Artificial

Sample Type Code

9	Regular
7	Replicate
2	Blank
1	Spike
5	Duplicate

99105 Replicate-sample type

10	Concurrent	40	Split-Concurrent
20	Sequential	50	Split-Sequential
30	Split	200	Other

99100 Blank-solution type

10	Inorganic grade (distilled/deionized)
40	Pesticide grade (OK for organics and organic carbon)
50	Volatile-organic grade (OK for VOCs, organics, and organic carbon)
200	Other

99101 Source of blank water

10	NWQL
55	Wisconsin Mercury Lab
140	EMD Chemicals
150	Ricca Chemical Company
200	Other

99111 QC sample associated with this environmental sample

1	No associated QA data
10	Blank
30	Replicate Sample
40	Spike sample
100	More than one type of QA sample
200	Other

99112 Purpose, Topical QC data

1	Routine QC (non-topical)
10	Topical for high bias (contamination)
20	Topical for low bias (recovery)
110	Topical for variability (field collection)

82398 Sampling Method

10	Equal Width Increment (EWI)
20	Equal Discharge Increment (EDI)
30	Single Vertical
40	Multiple Verticals
50	Point Sample
70	Grab Sample (Dip)
8010	Other
8030	Grab Sample At Water-Supply Tap

99102 Blank-sample type

1	Source Solution	90	Ambient
30	Trip	100	Field
40	Sampler	200	Other
50	Splitter		
60	Filter		
70	Preservation		
80	Equipment (done in non-field environment)		

84164 Sampler Type

3044	US DH-81
3045	US DH-81 With Teflon Cap And Nozzle
3051	US DH-95 Teflon Bottle
3052	US DH-95 Plastic Bottle
3053	US D-95 Teflon Bottle
3054	US D-95 Plastic Bottle
3055	US D-96 Bag Sampler
3057	US D-99 Bag Sampler
3070	Grab Sample
3071	Open-Mouth Bottle
3080	VOC Hand Sampler
8000	None
8010	Other

A complete set of fixed-value codes can be found online at: http://wwwnwis.er.usgs.gov/currentdocs/index.html

4

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