Upper Green River Basin Water Quality Survey

Sampling and Analysis Plan

August 2013

Final

Department of Natural Resources and Parks
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Sampling and Analysis Plan

Prepared for:

King County Department of Natural Resources and Parks
Wastewater Treatment Division

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ACRONYMS

AXYS	AXYS Analytical Services Ltd.
COC	chain of custody
DOC	dissolved organic carbon
DQOs	data quality objectives
Ecology	Washington Department of Ecology
EPA	U.S. Environmental Protection Agency
FSU	Field Science Unit
KCEL	King County Environmental Laboratory
LCS	laboratory control sample
LDW	Lower Duwamish Waterway
LIMS	Laboratory Information Management System
LMCL	lowest method calibration limits
LPAH	low molecular weight PAHs
MDL	method detection limit
ML	minimum level
OPR	ongoing precision and recovery
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyls
RDL	reporting detection limit
RPD	relative percent difference
PQL	practical quantitation limit
QA/QC	quality assurance/quality control
QC	quality control
SAP	sampling and analysis plan
SCWG	Source Control Work Group
SDL	specific detection limit
SRM	standard reference material
TOC	total organic carbon
TSS	total suspended solids

1.0. INTRODUCTION

This sampling and analysis plan (SAP) presents project information and sampling and analytical methodologies for the Upper Green River Basin Water Quality Survey. These methods will be employed to collect whole surface water samples and flow measurements to better understand the relative concentrations of polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and arsenic in the upper Green River compared to water samples collected in the middle and lower Green River during 2011 and 2012 (King County, in progress).

1.1 Project Background

The Duwamish River originates at the confluence of the Green and Black Rivers near Tukwila, Washington, and flows northwest for approximately 19 km (12 mi), splits at the southern end of Harbor Island to form the East and West Waterways, and then discharges into Elliott Bay in Puget Sound, Seattle, Washington. The Lower Duwamish Waterway (LDW) is approximately 5 miles long and consists of the downstream portion of the Duwamish River, excluding the East and West Waterways.

King County is a member of the Source Control Work Group (SCWG) for the Lower Duwamish Superfund site; other members include lead agency Washington Department of Ecology (Ecology), US Environmental Protection Agency (EPA), City of Seattle and the Port of Seattle. The SCWG works to understand potential chemical sources within the LDW Superfund site and to control and reduce sources that can contaminate waterway sediments. King County Wastewater Treatment Division seeks to better understand the potential sources of contaminants of concern into combined sewer overflow basins which discharge to the LDW and also contaminant inputs to the LDW from upstream sources.

The LDW Remedial Investigation (Windward 2010) indicates that more than 99% of the new sediment deposited in the LDW each year originates upstream in the Green/Duwamish River basin. Because of this, future LDW surface sediment quality is closely tied to the quality of incoming sediment from the Green/Duwamish River. A number of studies and sampling programs have evaluated chemical concentrations in both surface water and suspended solids within the Green/Duwamish River system (Herrera Environmental Consultants 2005; Herrera Environmental Consultants 2007; Gries and Sloan 2009; Windward 2010). King County recently completed a study evaluating the relative contributions of PCBs, PAHs and arsenic from the major tributaries in the Green River basin to the Green/Duwamish River and ultimately to the LDW (King County, in progress). Water quality in the Duwamish River is closely tied to water quality conditions in the Green River Basin, which is the major source of water to the Duwamish River.

The primary purpose of the sampling and analysis effort described here is to improve the understanding of contaminant concentrations in the Upper Green River Basin. King County is interested in measuring concentrations of key contaminants in areas of the watershed where chemical sources are limited. There is also an interest in gaining a better understanding of migrating salmon as a source of PCBs. To address these questions, surface water samples from the upper Green River Basin, above the Howard Hansen Dam, where access by anadromous salmon is restricted and contaminant sources are limited (largely atmospheric or geologic in the case of arsenic), will be collected and analyzed.

This survey will focus on analysis of PCBs, PAHs, and arsenic because the LDW remedial investigation has identified these chemicals as human health contaminants of concern (COC) within the LDW and residual risks are predicted to be present after cleanup. Dioxins/furans were also identified as human health COCs; however, these compounds were not included in this survey as they are not expected to be present at detectable levels in surface water samples. This survey will generally follow the methods outlined in the Green River Study Sampling and Analysis Plan (King County 2011). However, the sampling methods will differ due to logistical constraints at the upper watershed sampling locations.

1.2 Scope of Work

This sampling effort will involve collection and analysis of whole surface water samples for analysis of PCBs, PAHs and arsenic from two locations in the Upper Green River Basin above the Howard Hansen Dam. Due to the limited number of samples that will be collected, this effort is considered a survey. As such, the data will not provide sufficient information to estimate contaminant loading or capture seasonal or annual variability in chemical concentrations. Samples will be collected from both locations under both dry season baseflow conditions (3 event) and wet season storm event conditions (3 events). The two sampling locations, which were selected based on accessibility and lack of known use by anadromous fish, are located on the mainstem of the Upper Green River (approximately 20 miles upstream of the reservoir)[1] and on Sunday Creek, a tributary to the Upper Green River (Figure 1). Samples will be collected by compositing 6 grab samples collected at approximately 20 minute intervals over the course of two hours. All samples will be analyzed for PCB congeners, PAHs, and arsenic, in addition to total organic carbon (TOC), dissolved organic carbon (DOC) and total suspended solids (TSS).

1.3 Survey Schedule

Field reconnaissance to evaluate feasible sampling locations was conducted in the spring of 2013. Three dry season baseflow samples will be collected in September 2013. Three wet season storm event samples will be collected between October 2013 and March 2014. Chemical analysis will be complete in the second quarter of 2014, followed by data validation.

1.4 Project Staff

The following staff members are responsible for project execution:

Jeff Stern, LDW Project Manager.....206-263-6447

Wastewater Treatment Division Manager and Technical lead for all Lower Duwamish River studies.

[1] Note the drainage basin upstream of this location on the main stem of the Green River is much smaller than the main stem Green River locations sampled in previous water sampling efforts at Flaming Geyser State Park and Foster Links Golf Course (King County 2011).

Deb Lester, Green River Study Project Manager.....206-296-8325

Responsible for basin study project execution and adherence to SAP and schedule.

Debra Williston, Water and Land Resources Division Technical Lead 206-263-6540

Technical Support for all Lower Duwamish River studies including study project.

Jeff Droker, Field Science Unit Field Lead 206-684-2309

Responsible for sample collection.

Fritz Grothkopp, KC Environmental Lab Project Manager.....206-684-2327

Manages sample analysis, sample shipment, and data delivery.

Scott Mickelson, Data Validation Lead 206-296-8247

Responsible for all data validation.

2.0. STUDY DESIGN

The goal of this effort is to collect surface water samples and flow information that represent dry season baseflow and wet season storm event conditions at two locations in the Upper Green River Basin. All samples will be analyzed for PCB congeners, PAHs, arsenic; TSS, TOC and DOC. Resulting data will allow King County to better understand upper watershed contributions of these contaminants to the lower watershed. In addition, the survey will generate data from locations within the Green Watershed where there are limited contaminant sources; the potential sources include atmospheric (PCBs and PAHs), geologic (arsenic). The rain line runs within the drainage basin of Sunday Creek; therefore, creosote treated wood may also be a potential source of PAHs.

2.1 Data Quality Objectives

The data quality objectives (DQOs) for this effort are to collect data of known and sufficient quality to meet survey goals. Validation of survey data will assess whether the data collected are of sufficient quality to meet the survey goals. The data quality issues of precision, accuracy, bias, representativeness, completeness, comparability, and sensitivity are described in the following sections, along with data validation. Data validation is discussed in Section 5.0.

2.1.1 Precision, Accuracy, and Bias

Precision is the agreement of a set of results among themselves and is a measure of the ability to reproduce a result. Accuracy is an estimate of the difference between the true value and the measured value. The accuracy of a result is affected by both systematic and random errors. Bias is a measure of the difference, due to a systematic factor, between an analytical result and the true value of an analyte. Precision, accuracy, and bias for analytical chemistry may be measured by one or more of the following quality control (QC) samples:

- Analysis of various laboratory QC samples such as method blanks, spiked blanks, matrix spikes, laboratory control samples and laboratory duplicates or triplicates; and
- Collection and analysis of field replicate samples.

Precision of replicates is expected to be within the limits specified in Section 4. If precision is considered too low for project needs, these data will be used to guide future sampling efforts.

Accuracy is assessed through matrix spikes and spike duplicates along with the ongoing precision and recovery sample control charts. Additionally, the isotopic dilution method chosen for this study is the most rigorous method for PCB congener analysis. This method uses isotopically-labeled congeners, to track the recovery performance of the range of congener homologs. Thus, each congener concentration is theoretically adjusted for the extraction efficiency and analytical performance of that specific sample.

2.1.2 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at the sampling point, or an environmental condition. Surface water samples will be collected from stream or river locations to represent water quality during defined dry and wet season conditions. The samples are intended to generate data of sufficient quality to provide survey level water quality data for PCBs, PAHs and arsenic from the upper Green River Basin.

Samples will be collected in such a manner as to minimize potential contamination and other types of degradation in the chemical and physical composition of the water. This can be achieved by following guidelines for sampler decontamination, sample acceptability criteria, sample processing, observing proper hold-times, preservation, storage and preparation of samples.

2.1.3 Completeness

Completeness is defined as the total number of samples analyzed for which acceptable analytical data are generated, compared to the total number of samples submitted for analysis. Sampling with adherence to standardized sampling and testing protocols will aid in providing a complete set of data for this survey. The goal for completeness is 90%. The samples from each event should produce greater than 90% acceptable data under the QC conditions described elsewhere in this SAP.

2.1.4 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. This goal is achieved through the use of standard techniques to collect and analyze representative samples, along with standardized data validation and reporting procedures. By following the guidance described in this SAP, the goal of comparability between this and future sampling events will be achieved.

2.1.5 Sensitivity

Sensitivity is a measure of the capability of analytical methods to meet the survey goal. The analytical method detection limits (MDLs) presented in Section 5 are sensitive enough to detect PCB congeners, low level PAHs and arsenic at concentrations sufficient to increase the understanding of the contribution of these chemicals to the middle and lower Green River Basin.

2.2 Sampling and Analytical Strategy

The sampling strategy is designed to provide survey level water quality data within the upper Green River Basin to allow for a preliminary evaluation of the contribution of these contaminants from the Upper Green River Basin to the middle and lower Green River. The sampling effort will also provide contaminant information in areas of the Green Basin where contaminant sources are very limited. The survey is designed to begin to address the following questions:

1) What are the concentrations of PCBs, PAHs and arsenic during dry season baseflow and wet season storm event conditions in the upper Green River Basin where contaminant sources are very limited?
2) What are initial estimates of the relative contributions of PCBs, PAHs and arsenic from the Upper Green River Basin to the middle and lower Green River?

To begin to answer these questions, composite grab samples will be collected from one location on the Upper Green River Mainstem and one tributary. Due to access challenges and associated logistical constraints it is not feasible to collect time or flow weighted composite samples at these locations with an autosampler. As such, grab samples will be collected approximately every 20 to 30 minutes over a two hour period (total of 6 grabs per composite) at each location.

Baseflow Sample Collection

Three composite samples will be collected from each of the two locations during dry season baseflow (July – September) conditions during September 2013 with an antecedent dry period of at least 3 days. Flow will be manually measured using a Swoffer flow meter during the sampling event at each location, assuming conditions are safe for the field team to obtain measurements along a cross-section of the stream.

Storm Event Sample Collection

Collection of wet season (October – April) storm event samples from the two locations will be triggered by a rain event where 0.25 to 0.50" of precipitation is predicted. Three sets of storm event samples will be collected from each location. Precipitation will tracked using data from the NOAA weather station at Lester, WA[2]. The intent is to capture wash-off events with the potential to transport target chemicals downstream. Flow will be manually measured using a Swoffer flow meter during the sampling event at each location, assuming conditions are safe for the field team to obtain measurements along a cross-section of the stream. As sampling progresses, the sampling protocols may need to be revaluated; any changes will be documented in the data report.

2.3 Sampling Station Locations and Sample Identification

Sample locations will be identified using a unique locator name. The locator name, the date of collection and the unique sample identification number generated by King County Environmental Laboratory (KCEL) will identify individual samples collected at each location. The two sampling locations are shown in Figure 1. The corresponding locator numbers and sample coordinates are shown in Table 1.

[2] http://www.wrh.noaa.gov/mesowest/getobext.php?wfo=sew&sid=LSFW1&num=48&raw=0&dbn=m&banner=off

Table 1. Upper Green River and Tributary Sampling Locations and Locator Names.

Locator	Locator Description	Northing[a]	Easting[a]
UG319	**Upper Green Mainstem –approximately 20 miles** upstream of reservoir	456907.1	21538.15
SC319	**Sunday Creek** – at 5200 Road bridge	453400.3	24363.11

[a] State plane coordinates in North American Datum 1983 (NAD983) Washington State Plane North (4601)

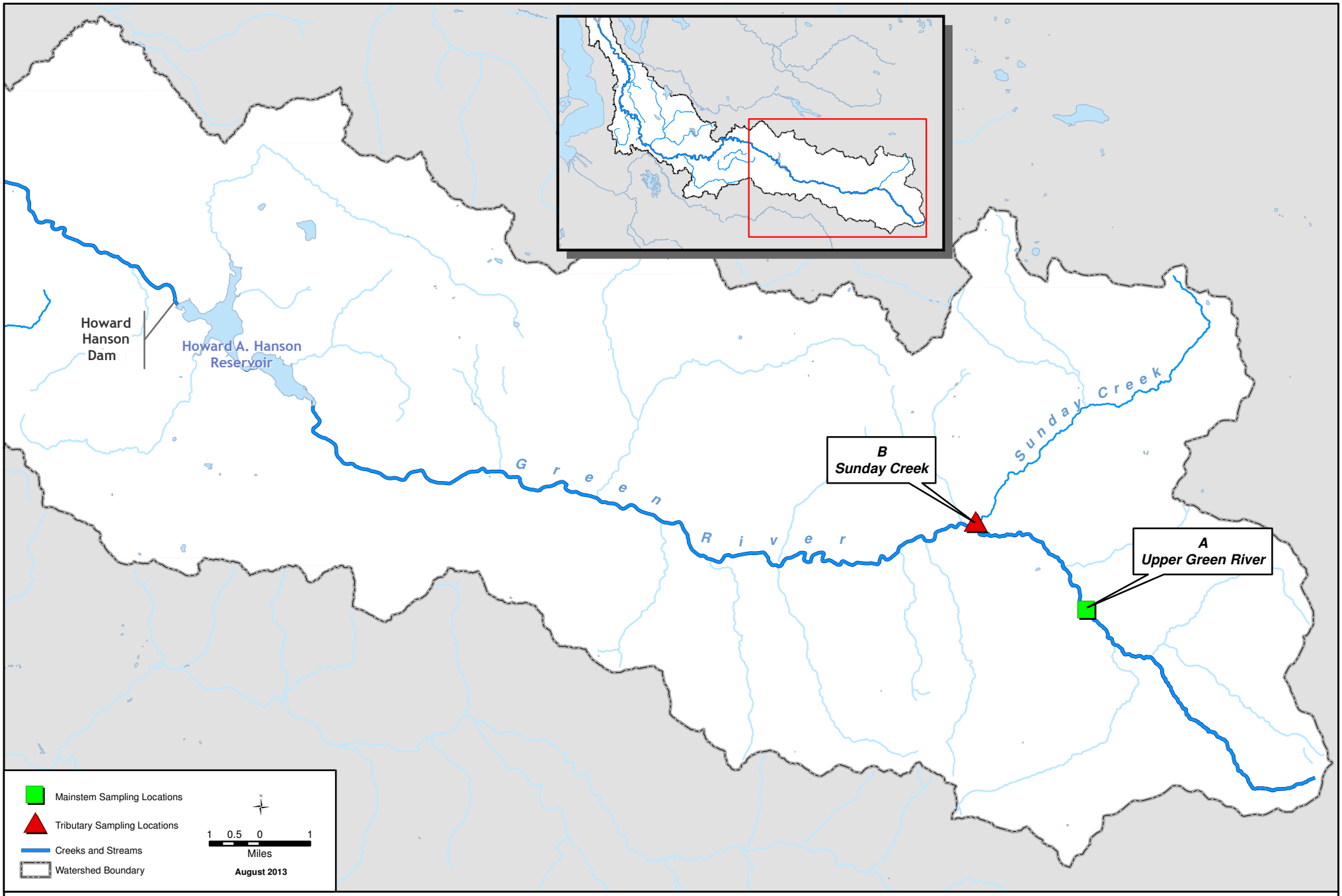
2.3.1 Sample Acquisition and Analytical Parameters

King County Field Science Unit (FSU) staff will primarily conduct sampling; however, other King County Water and Land Resources staff may provide assistance as needed. Sampling techniques are discussed in Section 3. Each sample will be analyzed for 209 PCB congeners, low level PAHs and arsenic along with DOC, TOC, and TSS. Table 2 summarizes the number of samples to be collected at each location including estimated number of sample replicates. The specific PAHs are listed in Section 4. PCB congener analysis will be conducted by AXYS Analytical in Sidney, British Columbia. All other chemical analyses and conventional analyses will be conducted by the KCEL, an Ecology Certified Laboratory.

Table 2. Number of Samples and Replicates per Sampling Locations

Sample Locations	Dry Baseflow	Wet Season/Storm	Field Replicates[a]
Upper Green River-Mainstream	3	3	2
Sunday Creek	3	3	2
Total Number of Samples	**6**	**6**	**4**

[a] One field replicate will be collected during collection of base flow samples at each location and one field replicate will be collected at each location during collection of storm events at each location.
Note: one field blank will be collected at KCEL.



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Wastewater Treatment Division

File Name: \\dnrp1\projects\wtd\projects\greenduwamish\projects\2013WtareReport\sampling_upstream.mxd
Shaun O'Neil 20130809

Figure 1.

Surface Water Sampling Locations

3.0. SAMPLING PROCEDURES

This section describes field procedures for sample collection, including sampling equipment decontamination and use, and procedures for recording field measurements and conditions. Requirements for sample containers and preservation, and sample custody procedures are also described.

3.1 Sample Collection

All grab samples will be collected using a site dedicated 1-liter stainless steel beaker with handle; two grabs of similar volume will be collected at 20 to 30 minute intervals over a two hour period. Field personnel will wade into the stream/river and submerge the 1-liter stainless beaker approximately 6-inches below the water surface upstream of their position to collect the grab sample. The individual grab samples will then be transferred to a glass (or Teflon®) carboy in the field creating a composite sample for each sampling event at each location. A target volume of 14 liters of water will be collected over the course of each sampling event. A Swoffer flow meter will be used to measure the flow at the sampling location.

Following the collection of each grab sample, FSU staff will place the sample in the carboy. The carboy will be stationed in a cooler with ice throughout the sampling event. At the conclusion of sampling, the carboy will be sealed with a Teflon® lined lid and transported immediately to KCEL. The composite grab samples will then be split out into the appropriate laboratory sample containers. This will be done by continuously agitating the sample in the carboy while transferring sample aliquots to the appropriate laboratory containers using a Teflon® siphon tube. Each sample container, except for the dissolved arsenic bottle, will be filled to the appropriate level from the carboy. This procedure will ensure a representative sample from the carboy in each laboratory sample container. The dissolved arsenic sample will be filtered directly from the carboy using a Teflon® siphon tube and drawing sample from the carboy through a 0.45 micron capsule filter using a peristaltic pump. Because the sample aliquot for dissolved arsenic cannot be filtered within 15 minutes of collection, appropriate hold-time violation flags will be added to the data.

3.2 Sampling Equipment

In addition to the samplers discussed in Section 3.1, the field equipment listed below will be available for field staff.

1) Sampling supplies:
   a) Cooler with ice
   b) Nitrile gloves
   c) 1-liter Stainless steel beakers
   d) Glass (or Teflon®) carboys
   e) Swoffer flow meter
2) Safety equipment:
   a) Hard hat

b) Personal Floatation Device
c) Waders
d) Field vest (reflective)
e) Documentation supplies:
f) Field notebook
g) Sample labels
h) Chain-of-custody forms
i) Camera

When visiting the sampling station, field personnel will record the following information on field forms that are maintained in a waterproof field notebook.

- Date
- Time of sample collection or visit
- Name(s) of sampling personnel
- Description of sampling location
- Weather conditions
- Number and type of samples collected
- Flow measurements
- Log of photographs taken, if any taken
- Deviations from sampling procedures
- Unusual conditions (e.g., water color or turbidity, presence of oil sheen, odors, and land disturbances)

3.3 Equipment Decontamination

Prior to first sampling event, the glass carboys and stainless steel beakers will be prepared by (1) Detergent 8 laboratory detergent followed with a hot water rinse; (3) a deionized water (ASTM I or II) rinse; and acetone rinse. The beaker will also be soaked in or rinsed with a 5% sulfuric acid solution rinse prior to deionized water rinse. Following this preparation, the carboy and beakers will be dedicated to each site until the sampling for this survey is completed. Following sample collection, all re-usable equipment will be decontaminated. Glass (or Teflon®) carboys will be cleaned in the following manner: (1) Detergent 8 laboratory detergent followed with a hot water rinse; (2) soaked in or rinsed with a 5% sulfuric acid solution rinse; (3) a deionized water (ASTM I or II) rinse; The stainless steel beakers, used to collect the grab samples will be cleaned in the following manner: (1) Detergent 8 laboratory detergent followed with a hot water rinse; and (2) a deionized water (ASTM I or II) rinse. Proofed clean PCB sampling containers will be supplied by AXYS Analytical. One equipment blank will be analyzed to check for possible cross contamination between sampling events. Proper personal protective equipment (new powder-free gloves) will be worn during sampling activities and during decontamination processes.

3.4 Sample Delivery and Storage

All samples will be kept in ice-filled coolers until delivery to the KCEL, on the day of collection. Additional sample preservation, where required, will be performed upon receipt of the samples at the KCEL. Samples will be split from the carboy container into the appropriate analytical containers and preserved according to method specifications at the KCEL.

Containers for PCB congener analysis will be delivered to AXYS Analytical within 1 to 3 months of sample collection. Samples will be held at KCEL at the appropriate temperature until delivery date. Samples will be maintained in coolers with ice and/or ice packs during the delivery process. Samples will either be driven to AXYS Analytical or shipped via overnight express delivery service. Table 3 shows sample handling and storage requirements.

Table 3. Sample Container, Preservation, Storage, and Hold Time Requirements

Analyte	Container	Preservation	Storage	Hold Time
Arsenic (Total & Dissolved)	500 mL Acid washed HDPE	ultra-pure HNO_3 to pH<2	n/a	180 days[1]
PAHs	2 x 1L amber glass	None	refrigerate at 4°C	7/40[2]
Dissolved Organic Carbon	125 mL amber wide mouth HDPE	0.45 µm filtration, then H_3PO_4 to pH<2 within 1 day	refrigerate at <6°C	28 days
Total Organic Carbon	125 mL amber narrow-mouth glass	H_3PO_4 to pH<2 within 1 day	refrigerate at <6°C	28 days
Total Suspended Solids	1-L clear wide mouth HDPE	None	refrigerate at <6°C	7 days
PCB Congeners	2 x 1-L amber glass	None	refrigerate at 4°C in the dark	1 year

[1] Within 15 minutes of collection, dissolved metals samples must be filtered (.45 µm).
[2] 7 days from sampling to extraction, 40 days from extraction to analysis

3.5 Chain of Custody

Chain of custody (COC) will commence at the time of sample collection. All samples will be under direct possession and control of FSU staff or locked in a controlled area. All sample information will be recorded on a COC form. This form will be completed in the field and will accompany all samples during transport and delivery to the laboratory. Upon arrival at the KCEL, the samples will be split into the appropriate containers then relinquished to the sample login person. The date and time of sample delivery will be recorded and both parties will then sign off in the appropriate sections on the COC form at this time. Once completed, original COC forms will be archived in the project file.

Samples delivered after regular business hours will be stored in a secure refrigerator until the next day. Samples delivered to AXYS Analytical will be accompanied by a properly-completed KCEL COC form and custody seals will be placed on the shipping cooler. AXYS Analytical will be expected to provide a copy of the completed COC form as part of their analytical data package.

3.6 Sample Documentation

Sampling information and sample metadata will be documented using the methods noted below.

- Field sheets generated by KCELs Information Management System (LIMS) will be used at all stations and will include the following information:
    1) Sample ID number
    2) Location name
    3) Flow measurements pre and post sample collection
    4) Number of grab samples per composite and sample interval between grab samples
    5) Date and time of sample collection (start and end times of the compositing period)
    6) Initials of all sampling personnel
- LIMS-generated container labels will identify each container with a unique sample number, station and site name, collect date, analyses required, and preservation method.
- The field sheet will contain records of collection times, general weather and the names of field crew staff.
- COC documentation will consist of KCEL's standard COC form, which is used to track release and receipt of each sample from collection to arrival at the lab.

3.7 Field Replicates and Field Blanks

Two field replicates will be collected from each station over the course of the survey for a total of 4 replicates. One set of replicates will be collected during baseflow sample collection and a second set will be collected during storm event sample collection. Field replicates will be analyzed for all parameters and will provide a measure of variability at sampling locations.

Collection and analysis of one field blank at KCEL will be required for the survey. The analysis of the field blank will be used to evaluate levels of contamination that might be associated with the sampling equipment and introduce bias into the sample result. An aliquot of a clean reference matrix (reverse osmosis water) will be processed through the sampling equipment as a blank and analyzed for PCB congeners, PAHs, DOC, TOC and arsenic. The following conditions apply to collection of the field blank sample:

- The field blank sample must be collected with the sampling equipment to be used to collect the samples.
- The field blank sample will be collected before the sampling begins.

Field blank shall be preserved, stored, and analyzed in the same manner as environmental samples. Field blank results for PCB congeners should be consistent with the blank criteria in sections 4.1. For PAHs and arsenic, field blank results should be less than the method detection limit (<MDL).

4.0. ANALYTICAL METHODS AND DETECTION LIMITS

Analytical methods are presented in this section, along with analyte-specific detection limit goals. For the PAHs, arsenic and selected conventional analytes, the terms MDL and RDL, used in the following subsections, refer to method detection limit and reporting detection limit, respectively. The KCEL reports both the LIMS reporting detection limit (LIMS RDL) and the LIMS method detection limit (LIMS MDL) for each sample and parameter, where applicable.

EPA's Office of Wastewater generally defines the PQL (practical quantitation limit) as the minimum concentration of a chemical constituent that can be reliably quantified, while the MDL is defined as the minimum concentration of a chemical constituent that can be detected. The KCEL LIMS RDL is analogous to the PQL for all analyses. It is verified either by including it on the calibration curve or by running a low level standard near the PQL value during the analytical run.

For arsenic and conventionals analyses, LIMS MDLs are typically two to five times higher than the statistically derived MDLs that are calculated using the 40 CFR Part 136, Appendix B procedure (Federal Register, Appendix B. 2007). In the case of some conventionals tests, MDLs are evaluated by the procedure listed in appendix of 40 CFR Part 136[3]. The detection limits derived from this approach are also typically two to five times the statistically derived MDLs that are calculated by the 40 CFR Part 136, Appendix B procedure. In the case of organic mass spectral analyses (i.e., for PAHs), a standard analyzed near the MDL concentration during calibration must produce a valid mass spectra and this standard is used to define the MDL.

Actual KCEL MDLs and RDLs may differ from the target detection limit goals as a result of necessary analytical dilutions or a reduction of extracted sample amounts based upon available sample volumes. Every effort will be made to meet the MDL/RDL goals listed in the SAP.

For PCB high resolution isotopic dilution based methods, the MDL and RDL terms are less applicable because limits of quantitation are derived from calibration capabilities and ubiquitous, but typically low level equipment and laboratory blank contamination. Additional reporting limit terms used particularly for PCB congener analyses are sample specific detection limits and lowest method calibration limits. Sample specific detection limit (SDL) is determined by converting the area equivalent to 2.5 times the estimated chromatographic noise height to a concentration. SDLs are determined individually for every congener, of each sample analysis run and accounts for any effect of matrix on the detection system and for recovery achieved through the analytical work-up. Lowest method calibration limits (LMCL) are based on calibration points from standard solutions. They are prorated by sample size and are supported by statistically-derived method reporting limit values.

[3] Appendix D: DQ FAC Single Laboratory Procedure v2.4 of the *Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs Final Report 12/28/0.7*

The PCB congener data will be reported to LMCLs and flagged down to the SDL value. In many cases the SDL may be below the LMCL. Method 1668A defines a Minimum Level (ML) value for each congener. The ML value is used to evaluate levels in the method blank. The ML is based on the lowest method calibration limit (LMCL) and any laboratory performing the method should be able to achieve at least that level. AXYS Analytical uses an additional calibration point that is lower than the calibration points specified in the method; as such they are able to quantify congeners below the ML specified in the method.

Details regarding the frequency of required QC samples are provided in the individual analytical sections shown below. In general for all methods, this frequency is 1 in 20 samples or 1 per batch whichever is more frequent. Below are general descriptions of types of laboratory QC samples:

- Analysis of method blanks is used to evaluate the levels of contamination that might be associated with the processing and analysis of samples in the laboratory and introduce bias into the sample result. Method blank results for all target analytes (other than PCB congeners) should be "less than the MDL."
- A laboratory duplicate is a second aliquot of a sample, processed concurrently and in an identical manner with the original sample. The laboratory duplicate is processed through the entire analytical procedure along with the original sample in the same quality control batch. Laboratory duplicate results are used to assess the precision of the analytical method and the relative percent difference (RPD) between the results should be within method-specified or performance-based quality control limits. In the case of PAHs a matrix spike duplicate may be used in lieu of a laboratory duplicate due to the large number of non-detects frequently encountered in these analyses.
- A spike blank is a spiked aliquot of clean reference matrix used for the method blank. The spiked aliquot is processed through the entire analytical procedure. Analysis of the spike blank is used as an indicator of method accuracy. It may be conducted in lieu of a laboratory control sample (LCS/SRM). A spike blank duplicate should be analyzed whenever there is insufficient sample volume to include a sample duplicate or matrix spike duplicate in the batch.
- The ongoing precision and recovery (OPR) samples must show acceptable recoveries, according to the respective methods for data to be reported without qualification. The OPR sample is typically called a Lab Control Sample (LCS) or Spiked Blank in LIMS.

4.1 PCB Congeners

PCB congener analysis will follow EPA Method 1668A Revision C (EPA 2010a), which is a high-resolution gas chromatography/high-resolution mass spectroscopy (HRGC/HRMS) method using an isotope dilution internal standard quantification. This method provides reliable analyte identification and very low detection limits. An extensive suite of labeled surrogate standards (Table 4) is added before samples are extracted. Data are "recovery-corrected" for losses in extraction and clean-up, and analytes are quantified against their labeled analogues.

AXYS Analytical will perform this analysis according to their SOP MLA-010 Analytical Method for the Determination of 209 PCB Congeners by EPA Method 1668, which is a proprietary document. A one-liter sample will be extracted followed by standard method clean-up, which includes layered Acid/Base Silica, Florisil, and Alumina. Analysis is performed with

an SPB Octyl column and a secondary DB1 column is used to resolve the co-eluting congeners PCB156 and PCB157. Method 1668A requires that if a sample contains more than 1% total solids, the solids and liquid will be extracted and analyzed separately.

Table 4. Labeled Surrogates and Recovery Standards Used for EPA Method 1668A PCB Congener Analysis

^{13}C-labeled PCB Congener Surrogate Standards				
1	37	123	155	202
3	54	118	167	205
4	81	114	156/157	208
15	77	105	169	206
19	104	126	188	209
^{13}C-labeled Cleanup Standards				
28	111	178		
^{13}C-labeled Internal (Recovery) Standards				
9	52	101	138	194

Table 5 lists the 209 PCB congeners and their respective target SDL and LMCL values. The reporting limits for individual samples may differ from those in Table 5 since they are determined by signal to noise ratios and changes to final volumes. Typical sample detection limits are shown. Note that several of the congeners co-elute and a single SDL or LMCL value is provided for the congeners in aggregate.

Table 5. PCB Congener water detection limit goals in pg/L and lower calibration limits by1668A, AXYS Analytical method MLA 010.

PCB Congener	Typical Detection Limit/MDL	LMCL based on Low Cal./RDL
CL1-PCB-1	1.0	4.0
CL1-PCB-2	1.0	4.0
CL1-PCB-3	1.0	4.0
CL2-PCB-4	2.0	4.0
CL2-PCB-5	2.0	4.0
CL2-PCB-6	2.0	4.0
CL2-PCB-7	2.0	4.0
CL2-PCB-8	2.0	4.0
CL2-PCB-9	2.0	4.0
CL2-PCB-10	2.0	4.0
CL2-PCB-11	2.0	4.0
CL2-PCB-12/13	2.0	8.0
CL2-PCB-14	2.0	4.0
CL2-PCB-15	2.0	4.0
CL3-PCB-16	1.0	4.0
CL3-PCB-17	1.0	4.0
CL3-PCB-19	1.0	4.0
CL3-PCB-21/33	1.0	8.0
CL3-PCB-22	1.0	4.0
CL3-PCB-23	1.0	4.0
CL3-PCB-24	1.0	4.0
CL3-PCB-25	1.0	4.0
CL3-PCB-26/29	1.0	8.0
CL3-PCB-27	1.0	4.0
CL3-PCB-28/20	1.0	8.0
CL3-PCB-30/18	1.0	8.0
CL3-PCB-31	1.0	4.0
CL3-PCB-32	1.0	4.0
CL3-PCB-34	1.0	4.0
CL3-PCB-35	1.0	4.0
CL3-PCB-36	1.0	4.0
CL3-PCB-37	1.0	4.0
CL3-PCB-38	1.0	4.0
CL3-PCB-39	1.0	4.0
CL4-PCB-41/40/71	1.0	12.0
CL4-PCB-42	1.0	4.0
CL4-PCB-43	1.0	4.0

PCB Congener	Typical Detection Limit/MDL	LMCL based on Low Cal./RDL
CL4-PCB-44/47/65	1.0	12.0
CL4-PCB-45/51	1.0	8.0
CL4-PCB-46	1.0	4.0
CL4-PCB-48	1.0	4.0
CL4-PCB-50/53	1.0	8.0
CL4-PCB-52	1.0	4.0
CL4-PCB-54	1.0	4.0
CL4-PCB-55	1.0	4.0
CL4-PCB-56	1.0	4.0
CL4-PCB-57	1.0	4.0
CL4-PCB-58	1.0	4.0
CL4-PCB-59/62/75	1.0	12.0
CL4-PCB-60	1.0	4.0
CL4-PCB-61/70/74/76	1.0	16.0
CL4-PCB-63	1.0	4.0
CL4-PCB-64	1.0	4.0
CL4-PCB-66	1.0	4.0
CL4-PCB-67	1.0	4.0
CL4-PCB-68	1.0	4.0
CL4-PCB-69/49	1.0	8.0
CL4-PCB-72	1.0	4.0
CL4-PCB-73	1.0	4.0
CL4-PCB-77	1.0	4.0
CL4-PCB-78	1.0	4.0
CL4-PCB-79	1.0	4.0
CL4-PCB-80	1.0	4.0
CL4-PCB-81	1.0	4.0
CL5-PCB-82	1.0	4.0
CL5-PCB-83/99	1.0	8.0
CL5-PCB-84	1.0	4.0
CL5-PCB-88/91	1.0	8.0
CL5-PCB-89	1.0	4.0
CL5-PCB-92	1.0	4.0
CL5-PCB-94	1.0	4.0
CL5-PCB-95/100/93/102/98	1.0	20.0
CL5-PCB-96	1.0	4.0
CL5-PCB-103	1.0	4.0
CL5-PCB-104	1.0	4.0
CL5-PCB-105	1.0	4.0

PCB Congener	Typical Detection Limit/MDL	LMCL based on Low Cal./RDL
CL5-PCB-106	1.0	4.0
CL5-PCB-107/124	1.0	8.0
CL5-PCB-108/119/86/97/125/87	1.0	24.0
CL5-PCB-109	1.0	4.0
CL5-PCB-110/115	1.0	8.0
CL5-PCB-111	1.0	4.0
CL5-PCB-112	1.0	4.0
CL5-PCB-113/90/101	1.0	12.0
CL5-PCB-114	1.0	4.0
CL5-PCB-117/116/85	1.0	12.0
CL5-PCB-118	1.0	4.0
CL5-PCB-120	1.0	4.0
CL5-PCB-121	1.0	4.0
CL5-PCB-122	1.0	4.0
CL5-PCB-123	1.0	4.0
CL5-PCB-126	1.0	4.0
CL5-PCB-127	1.0	4.0
CL6-PCB-128/166	1.0	8.0
CL6-PCB-130	1.0	4.0
CL6-PCB-131	1.0	4.0
CL6-PCB-132	1.0	4.0
CL6-PCB-133	1.0	4.0
CL6-PCB-134/143	1.0	8.0
CL6-PCB-136	1.0	4.0
CL6-PCB-137	1.0	4.0
CL6-PCB-138/163/129/160	1.0	16.0
CL6-PCB-139/140	1.0	8.0
CL6-PCB-141	1.0	4.0
CL6-PCB-142	1.0	4.0
CL6-PCB-144	1.0	4.0
CL6-PCB-145	1.0	4.0
CL6-PCB-146	1.0	4.0
CL6-PCB-147/149	1.0	8.0
CL6-PCB-148	1.0	4.0
CL6-PCB-150	1.0	4.0
CL6-PCB-151/135/154	1.0	12.0
CL6-PCB-152	1.0	4.0
CL6-PCB-153/168	1.0	8.0
CL6-PCB-155	1.0	4.0

PCB Congener	Typical Detection Limit/MDL	LMCL based on Low Cal./RDL
CL6-PCB-156/157	1.0	8.0
CL6-PCB-158	1.0	4.0
CL6-PCB-159	1.0	4.0
CL6-PCB-161	1.0	4.0
CL6-PCB-162	1.0	4.0
CL6-PCB-164	1.0	4.0
CL6-PCB-165	1.0	4.0
CL6-PCB-167	1.0	4.0
CL6-PCB-169	1.0	4.0
CL7-PCB-170	1.0	4.0
CL7-PCB-171/173	1.0	8.0
CL7-PCB-172	1.0	4.0
CL7-PCB-174	1.0	4.0
CL7-PCB-175	1.0	4.0
CL7-PCB-176	1.0	4.0
CL7-PCB-177	1.0	4.0
CL7-PCB-178	1.0	4.0
CL7-PCB-179	1.0	4.0
CL7-PCB-180/193	1.0	8.0
CL7-PCB-181	1.0	4.0
CL7-PCB-182	1.0	4.0
CL7-PCB-183/185	1.0	8.0
CL7-PCB-184	1.0	4.0
CL7-PCB-186	1.0	4.0
CL7-PCB-187	1.0	4.0
CL7-PCB-188	1.0	4.0
CL7-PCB-189	1.0	4.0
CL7-PCB-190	1.0	4.0
CL7-PCB-191	1.0	4.0
CL7-PCB-192	1.0	4.0
CL8-PCB-194	1.0	4.0
CL8-PCB-195	1.0	4.0
CL8-PCB-196	1.0	4.0
CL8-PCB-197/200	1.0	8.0
CL8-PCB-198/199	1.0	8.0
CL8-PCB-201	1.0	4.0
CL8-PCB-202	1.0	4.0
CL8-PCB-203	1.0	4.0
CL8-PCB-204	1.0	4.0

PCB Congener	Typical Detection Limit/MDL	LMCL based on Low Cal./RDL
CL8-PCB-205	1.0	4.0
CL9-PCB-206	1.0	4.0
CL9-PCB-207	1.0	4.0
CL9-PCB-208	1.0	4.0
CL10-PCB-209	1.0	4.0

SDL = sample detection limit
LMCL = lower method calibration limit
pg/L = picograms per liter

Quality control samples include method blank, OPR sample, and surrogate spikes. Method blanks and OPR, which are the same as spike blanks, are each included with each batch of samples. Surrogate spikes are labeled compounds that are included with each sample. The sample results are corrected for the recoveries associated with these surrogate spikes as part of the isotope dilution method. In addition, a laboratory duplicate will be conducted with each batch of samples. Note that a matrix spike and matrix spike duplicate are not required, nor meaningful under Method 1668A. Method 1668A has specific requirements for method blanks that must be met before sample data can be reported (see section 9.5.2 of Method 1668A). The OPR samples must show acceptable recoveries, according to Method 1668A, in order to samples to be analyzed and data to be reported. A summary of the quality control samples are shown in Table 6.

Table 6. PCBs QA/QC Frequency and Acceptance Criteria

Frequency	Method Blank	Lab Duplicate (RPD)	OPR (% Recovery)	Surrogate Spikes
	1 per batch*	1 per batch*	1 per batch*	Each sample
PCB Congeners	<LMCL[a]	RPD <50%	laboratory QC limits [b]	laboratory QC limits [b]

batch = 20 samples or less prepared as a set
[a]EPA Method 1668A blank criteria (see Table 2 of the published method) is to be below the Minimum Levels: 2, 10, 50 pg/congener depending on the congener with the sum of all congeners below 300 pg/sample. Higher levels are acceptable when sample concentrations exceed 10x the blank levels.
[b]The laboratory's performance-based control limits that are in effect at the time of analysis will be used as quality control limits.
LMCL = Lowest Method Calibration Limit
RPD = Relative Percent Difference
OPR = ongoing precision and recovery

4.2 Polycyclic Aromatic Hydrocarbons (PAHs)

Samples will be analyzed for the PAHs included in Table 7 below. The samples will be prepared by liquid-liquid extraction as detailed in method EPA method 3520C, KCEL SOP 701. Samples will be analyzed according to EPA Method 8270D; Gas Chromatography/Mass Spectrometry with Selected Ion Monitoring and Large Volume Injection method (GC/MS-SIM LVI). An SOP is currently being developed for this project. MDL and RDL goals are based upon extraction of one-liter of sample concentrated to 1.0 ml final volume. Depending upon the matrix, additional cleanups may be performed to ensure adequate instrument performance.

Every effort will be made to meet the target MDL and RDL goals. Due to the challenges of reporting as many detectable compounds as possible, there may need to be a change to the sample volumes, concentration factors or employ additional cleanups if the analytical protocols in the SOP do not yield enough detectable analytes to meet the project DQOs. Prior to implementing a method change, the project manager will be consulted and the method change will undergo a project level review.

In addition to reporting individual PAH results, KCEL will report total high molecular weight PAHs (HPAHs) and total low molecular weight PAHs (LPAHs) as the sum of detected HPAHs or LPAHs, respectively[4]. If no PAHs are detected within the LPAH or HPAH class, the reported MDL/RDL for these totals will be the highest MDL/RDL reported for the individual PAHs in that class. When individual PAHs in HPAH or LPAH are detected, the reported MDL/RDL for these totals will be the lowest MDL/RDL from the respective LPAH or HPAH class.

Table 7. PAH Target Compounds and Detection Limit Goals in µg/L

Analyte	MDL	RDL	Analyte	MDL	RDL
2-Methylnaphthalene	0.00130	0.00600	Chrysene	0.00050	0.00100
Acenaphthene	0.00070	0.00300	Dibenzo(a,h)anthracene	0.00070	0.00200
Acenaphthylene	0.00050	0.00100	Fluoranthene	0.00033	0.00200
Anthracene	0.00050	0.00100	Fluorene	0.00050	0.00200
Benzo(a)anthracene	0.00050	0.00100	Indeno(1,2,3-cd)Pyrene	0.00070	0.00200
Benzo(a)pyrene	0.00100	0.00200	Naphthalene	0.00130	0.01000
Benzo(b,j,k)fluoranthene	0.00100	0.00200	Phenanthrene	0.00040	0.00400
Benzo(g,h,i)perylene	0.00060	0.00200	Pyrene	0.00050	0.00200

NOTE: The MDL/RDL limits are calculated on a 1 liter extraction to a final volume of 1 ml. MDL/RDL limits will vary depending on amount extracted and final volume.

In addition to the surrogates and internal standards, which assess sample accuracy and bias, a method blank, spike blank, matrix spike and matrix spike duplicate or laboratory duplicate will be analyzed with each set of 20 samples, or one per QC batch. Matrix spike and matrix spike duplicate samples will only be prepared when sufficient water volumes are available. The spike blank, matrix spike, laboratory control sample and surrogate recovery limits will be based on laboratory QC limits; these are empirically derived performance-based laboratory control limits. These limits may be updated once per calendar year and the limits in effect at the time of analysis will be used. Current QA/QC frequency and acceptance criteria for PAH analysis are shown in Table 8.

[4] When PAHs are detected, the reported MDL/RDL for the total LPAH or total HPAH parameter will be lowest MDL/RDL of the individual LPAHs or HPAHs, respectively.

Table 8. PAH QA/QC Frequency and Acceptance Criteria

Frequency	Method Blank	Spike Blank (% Recovery)[b]	Matrix Spike (% Recovery)[b]	Matrix Spike Duplicate or Lab Duplicate (RPD)
	1 per Extraction batch[a]	1 per Extraction batch[a]	1 per QC batch	1 per QC batch
2-Methylnaphthalene	<MDL	21-136	28-97	40
Acenaphthene	<MDL	45-114	38-90	40
Acenaphthylene	<MDL	56-124	48-107	40
Anthracene	<MDL	47-107	49-112	40
Benzo(a)anthracene	<MDL	86-111	83-114	40
Benzo(a)pyrene	<MDL	40-135	27-150	40
Benzo(b,j,k)fluoranthene	<MDL	71-131	43-146	40
Benzo(g,h,i)perylene	<MDL	63-126	26-140	40
Chrysene	<MDL	77-111	68-115	40
Dibenzo(a,h)anthracene	<MDL	61-139	24-150	40
Fluoranthene	<MDL	73-116	65-125	40
Fluorene	<MDL	54-122	42-113	40
Indeno(1,2,3-cd)Pyrene	<MDL	58-137	20-150	40
Naphthalene	<MDL	32-110	20-90	40
Phenanthrene	<MDL	57-104	51-98	40
Pyrene	<MDL	66-143	38-150	40

Surrogate / Frequency	Surrogate (% Recovery)[b]
	Added to all samples and QC
2-Fluorobiphenyl	23-124
D14-Terphenyl	63-150

[a] QC Extraction batch = 20 samples or less prepared within a 12 hour shift
[b] The laboratory's performance-based control limits that are in effect at the time of analysis will be used as quality control limits. These are empirically derived lab performance-based control limits and may be updated once per calendar year and the limits in effect at the time of analysis will be used as QC limits for all ongoing precision and accuracy QC samples and surrogates. Changes to QC Limits due to annual updates should be noted in a SAP addendum or in the data report.
< MDL = Method Blank result should be less than the *method detection limit*.
RPD = Relative Percent Difference

4.3 Arsenic

Arsenic samples will be analyzed and reported by EPA Method 200.8 (Inductively Coupled Plasma-Mass Spectrometry [ICP-MS]), KCEL SOP 624. Total and dissolved arsenic samples will be preserved to a pH less than 2 with ultrapure nitric acid for ICP-MS analysis. The following detection limit goals are targets for arsenic (Table 9). MDL and RDL values for actual samples will be reported to 2 and 3 significant figures, respectively.

Table 9. Arsenic Target Detection Limit Goals (µg/L)

Analyte	MDL	RDL
Arsenic	0.10	0.500

Sample accuracy and bias will be evaluated by a laboratory method blank, lab duplicate, spike blank and matrix spike sample and will be analyzed with each set of 20 samples, or one per batch. QA/QC frequency and acceptance criteria for arsenic analysis are as shown in Table 10. Matrix spikes and lab duplicates may not be analyzed if sufficient sample volume is not available.

Table 10. Arsenic QA/QC Frequency and Acceptance Criteria

Frequency	Method Blank	Spike Blank (% Recovery)	Lab Duplicate (RPD)	Matrix Spike (% Recovery)
	1 per batch	1 per batch	1 per batch	1 per batch
Arsenic by ICP-MS	< MDL	85 – 115%	≤ 20%	75 - 125%

Note: batch = 20 samples or less
< MDL = Method Blank result should be less than the *method detection limit*.
RPD = Relative Percent Difference

4.4 Conventionals

All conventional analyses will follow Standard Methods (SM) protocols (American Public Health Association [APHA] 1998). Table 11 presents the analytical methods, detection limits and units for conventional analyses.

Table 11. Conventionals Analytical Methods and Detection Limit Goals in mg/L

Analyte	Method	KCEL SOP	MDL	RDL
Dissolved Organic Carbon	SM5310-B	336	0.5	1.0
Total Organic Carbon	SM5310-B	336	0.5	1.0
Total Suspended Solids	SM2540-D	309	0.5	1.0

Detection limits will vary slightly from sample to sample, depending on the exact amount of sample volume used for analysis. Table 12 describes the minimum QC required for the conventionals analysis. Conventional QC samples will be analyzed at the frequency of one per QC batch of 20 or less samples.

Table 12. Conventionals QA/QC Frequency and Acceptance Criteria

Analyte / Frequency	Method Blank	Lab Duplicate (RPD)	Spike Blank (% Recovery)	Matrix Spike (% Recovery)	LCS (% Recovery)
	1 per batch*	1 per batch*	1 per batch*	1 per batch*	1 per batch*
Dissolved Organic Carbon	<MDL	20%	80-120%	75-125%	85-115%
Total Organic Carbon	<MDL	20%	80-120%	75-125%	85-115%
Total Suspended Solids	<MDL	25%	N/A	N/A	80-120%

* batch = 20 samples or less prepared as a set
< MDL = less than the Method Detection Limit.
RPD = Relative Percent Difference
LCS = Lab Control Sample
N/A = Not Applicable

5.0. DATA VALIDATION, REPORTING AND RECORD KEEPING

This section presents the data validation, reporting and record keeping for the samples collected under this SAP.

5.1 Data Validation

Chemical data generated during this survey will be validated according to accepted Environmental Protection Agency (EPA) guidelines (EPA 2008 and 2010b), where applicable. KCEL will develop EPA Stage 2a data packages allowing for this level of validation. This level of validation includes reviews of holding times, method blanks, and QA/QC samples. For analyses performed by KCEL, the validator will also review data anomaly forms (DAFs) generated by the laboratory. These forms include an issues related to calibrations, instrument performance, and internal standard summaries. PCB data will undergo a Level III data validation. All necessary data needed for independent review of PCB congener data will be provided by AXYS Analytical. All other chemical analysis and associated conventional water quality data will be validated against requirements of the reference methods as well as the requirements of this SAP. Data validation will be performed by the King County Water and Land Resources Division staff for all data generated by KCEL. Data validation for PCB congener data will be conducted by an outside party for this survey. Data validation memoranda will be produced and maintained along with the analytical data as part of the project records.

5.2 Reporting

All analytical data collected for this survey and any supporting information will be documented in a data report for data. Data validation memoranda will be included in the data report, as will copies of COC forms. All analytical data will be submitted for loading into Ecology's Environmental Information Management (EIM) database.

5.3 Record Keeping

All hard-copy field sampling records, custody documents, raw lab data, and laboratory summaries and narratives generated by KCEL will be archived according to KCEL policy for LDW Superfund records. These records will include both hard copy and electronic data. Conventional, Trace Metals and Trace Organics analytical data produced by the KCEL will be maintained on its LIMS database in perpetuity. AXYS Analytical will provide electronic data deliverables and associated quality control results to King County. While KCEL will maintain a copy of deliverables from AXYS Analytical, copies of full data packages pertaining to King County samples analyzed by AXYS Analytical will be maintained by AXYS Analytical for 10 years from the analysis date.

6.0. REFERENCES

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