2022 Central Basin Marine Sediments: Sampling and Analysis Plan

May 2022

King County

Department of Natural Resources and Parks
Water and Land Resources Division
Science and Technical Support Section
King Street Center, KSC-NR-0704
201 South Jackson Street, Suite 704
Seattle, WA 98104
206-477-4800 TTY Relay: 711
www.kingcounty.gov/EnvironmentalScience

Alternate Formats Available
206-477-4800 TTY Relay: 711

2022 Central Basin Marine Sediments: Sampling and Analysis Plan

Submitted by:

Wendy Eash-Loucks
Marine and Sediment Assessment Group
King County Water and Land Resources Division
Department of Natural Resources and Parks

King County
Department of
Natural Resources and Parks
Water and Land Resources Division

Citation

King County. 2022. 2022 Central Basin Marine Sediments: Sampling and Analysis Plan. Prepared by Wendy Eash-Loucks, Water and Land Resources Division. Seattle, Washington.

Table of Contents

Executive Summary 1
1.0 Introduction 2
2.0 Monitoring Program Goals 3
3.0 Monitoring History 4
4.0 Sampling Design 6
4.1 Data Quality Objectives 6
4.1.1 Precision, Accuracy, and Bias 6
4.1.2 Representativeness 6
4.1.3 Completeness 7
4.1.4 Comparability 7
4.1.5 Sensitivity 7
4.2 Sampling Strategy 7
4.3 Sample Acquisition and Analytical Parameters 9
4.4 Data Analysis 9
5.0 Sampling Procedures 11
5.1 Station Positioning 11
5.2 Sampler Deployment and Retrieval 12
5.3 Sample Acceptability Criteria 12
5.4 Sample Processing—Sediment Chemistry 13
5.5 Sample Processing—Benthic Taxonomy 14
5.6 Sampling Equipment Decontamination 15
5.7 Sample Storage and Delivery 15
5.8 Chain of Custody 15
6.0 Sample and Field Documentation 17
7.0 Project Health and Safety 20
7.1 General Vessel Safety 20
7.2 Grab Sampling 21
7.3 Chemical Hazards 21
8.0 Sediment Chemistry Analytical Methods and Quality Assurance/Quality Control 23
8.1 Quantitation Limits 23

8.2 Conventionals 25
8.3 Metals 26
8.4 Organics 26
8.4.1 BNAs 27
8.4.2 Chlorinated Pesticides and PCBs 29
8.4.3 Tributyltin 30
8.4.4 PBDEs 30
8.5 Sediment Chemistry QA/QC 31
8.5.1 Method Blank Evaluation 32
9.0 Benthic Taxonomy Analytical Methods and Quality Assurance/Quality Control 34
10.0 Data Reporting and Recordkeeping 36
10.1 Sediment Chemistry Data 36
10.1.1 LIMS Data Qualifiers 36
10.2 Benthic Taxonomy Data 37
10.3 Draft and Final Reports and EIM Files 37
10.4 Recordkeeping 37
11.0 Project Organization and Schedule 38
12.0 References 39

Figures

Figure 1. 2022 Central Basin ambient subtidal sediment monitoring station locations. 8
Figure 2. King County Environmental Laboratory chain of custody form. 16
Figure 3. King County Environmental Laboratory standard field sheet. 19

Tables

Table 1. Central Basin ambient sediment monitoring station description, selection rational, and sampling history. 5
Table 2. 2022 Central Basin ambient sediment sampling station locations (NAD83) and depths. 8
Table 3. Sediment sample containers, storage conditions, and analytical hold times. 14
Table 4. Conventionals target analytes, methods, and detection limit goals. 25
Table 5. Trace metals target analytes and detection limit goals (mg/Kg wet weight). ...26
Table 6. BNA target analytes and detection limits (µg/Kg wet weight). 28
Table 7. Chlorinated pesticide and PCB target analytes and sample detection limit goals (µg/Kg wet weight). 29
Table 8. Butyltin target analytes and sample detection limit goals (µg/Kg wet weight). 30
Table 9. PBDE target analytes and sample detection limit goals (µg/Kg wet weight).....30
Table 10. Marine sediment chemistry quality control samples. 31
Table 11. QA1 acceptance criteria for marine sediment chemistry samples. 32

Appendices

Appendix A: Metals and Organics Sediment Quality Criteria
Appendix B: Sediment Chemistry and Benthic Infauna Sampling Supplies
Appendix C: Metals and Organics Performance-Based QC Limit Goals
Appendix D: KCEL Laboratory Information Management System (LIMS) Products

EXECUTIVE SUMMARY

This sampling and analysis plan (SAP) presents project information and sampling and analytical methodologies that will be used to conduct subtidal sediment sampling in 2022 for King County's Marine Ambient Monitoring Program. This work is being performed as part of a long-term sediment monitoring program designed to assess sediment quality in Puget Sound's Central Basin. The data are also used as a comparative aid to confirm that discharges from the County's wastewater outfalls are not adversely affecting the marine environment. This SAP includes a description of the project, sampling and analytical methodologies, and reporting requirements.

The current ambient sediment monitoring program was last modified in 2007. It includes eight stations in Elliott Bay, which are sampled every two years, and six additional stations in the Central Basin of Puget Sound (three mainstem and three additional embayments), which are sampled every five years. This sampling event includes the six stations located in the mainstem and embayments of the Central Basin that were last sampled in 2017, and one new site located in Quartermaster Harbor.

The goals of the 2022 Central Basin sediment sampling event are to collect chemistry and benthic infauna taxonomy data of sufficient quantity and quality to be able to:

- Assess the health of the benthic community at stations from which sediment chemistry data have been collected;
- Evaluate spatial differences in benthic community assemblages as well as compare the benthic community assemblages to regional Puget Sound benthic community data;
- Evaluate the spatial variation of sediment chemical concentrations;
- Evaluate the temporal variation of sediment chemical concentrations by comparing to historical data;
- Compare sediment chemical concentrations to Puget Sound-wide regional values; and
- Evaluate sediment chemical concentrations relative to the current marine sediment quality standards of Chapter 173-204 WAC (Ecology, 2013).

The following parameters will be analyzed in all samples: sediment conventionals (total solids, total organic carbon, particle size distribution, ammonia, and total sulfide), metals, and organics (including base-neutral-acids [BNAs], chlorinated pesticides and PCBs, PBDEs, and tributyltin). Benthic taxonomy will also be assessed at all seven stations.

1.0 INTRODUCTION

This sampling and analysis plan (SAP) presents project information along with sampling and analytical methodologies that will be employed to conduct sediment quality monitoring in 2022 as part of King County's Ambient Marine Monitoring Program. This work is being performed as part of a long-term sediment monitoring program designed to assess sediment quality in the Central Basin. The SAP includes a description of the project, monitoring history, sampling design, sampling and analytical methodologies, quality assurance/quality control, and reporting requirements. The SAP has been prepared in accordance with the Washington State Department of Ecology (Ecology) guidance document *Sediment Cleanup User's Manual* (*SCUM*) (Ecology, 2021).

2.0 MONITORING PROGRAM GOALS

King County's marine ambient monitoring program gathers water, sediment, and biological data in the Central Basin of Puget Sound including associated embayments, such as Elliott Bay and the Duwamish River. These data are used to provide an understanding of water and sediment quality issues, and to assess the general health of the Puget Sound marine environment within King County. The data are also used as a comparative aid to confirm that discharges from the County's wastewater outfalls are not adversely affecting the marine environment.

King County maintains a long-term dataset for water and sediment quality that goes back to the 1960s for some stations and parameters. This dataset can provide insight into natural variations and be used to assess recent conditions. The goal of the marine monitoring program is to identify sources of pollution when possible, provide water and sediment quality information for management decisions, and evaluate status and trends of marine water and sediment quality within King County.

The study goals of the 2022 ambient sediment sampling event are to collect chemistry and benthic infauna taxonomy data of sufficient quantity and quality to be able to:

- Assess the health of the benthic community at stations from which sediment chemistry data have been collected;
- Evaluate spatial differences in benthic community assemblages as well as compare the benthic community assemblages to regional Puget Sound benthic community data;
- Evaluate the spatial variation of sediment chemical concentrations;
- Evaluate the temporal variation of sediment chemical concentrations by comparing to historical data;
- Compare sediment chemical concentrations to Puget Sound-wide regional values; and
- Evaluate sediment chemical concentrations relative to the current marine sediment quality standards of Chapter 173-204 WAC (Ecology, 2013).

3.0 MONITORING HISTORY

King County has collected sediment quality data from subtidal, ambient monitoring stations for over 30 years. These subtidal monitoring stations have been located within the Central Basin of Puget Sound, including Elliott Bay. Sampling locations have traditionally been located to monitor sediment quality at both ambient stations and in areas within the general vicinity of, but away from, the direct impact of pollution sources. These pollution sources include both point-source pollution such as wastewater and storm water outfalls and general non-point sources such as the Duwamish River.

From as early as 1985 through 1996, the previous sediment monitoring program collected samples roughly annually. From 1998 through 2004 samples were collected every other year. During this time, samples were collected from the top two centimeters (cm) of sediment and analyzed for metals and organic chemicals in order to evaluate sediment quality in the most-recently deposited material. Sediment quality has been evaluated by comparing metals and organics concentrations to the published sediment quality chemical criteria of the Washington State Sediment Management Standards (SMS) as well as Puget Sound region-wide sediment data.

The former subtidal sediment monitoring program was temporarily discontinued after 2004 to provide an opportunity for King County staff scientists and a peer review panel to evaluate data generated from the program as well as other data collection efforts within the region. Following this review, King County expanded its marine ambient subtidal sediment monitoring program in 2007 to focus on sediment quality in Elliott Bay, where many sources of pollution exist, and ambient sediment quality in the Central Basin of Puget Sound as well as three additional embayments of interest. The current ambient sediment monitoring program includes eight sites in Elliott Bay, sampled every two years, and six sites in the Central Basin of Puget Sound (three mainstem and three additional embayments) sampled every five years. The six long-term mainstem and embayment sampling stations, with one additional embayment station, will be sampled this year (2022). The stations and their sampling history are shown in Table 1.

Table 1. Central Basin ambient sediment monitoring station description, selection rational, and sampling history. X's indicate years when samples were collected.

			Years Sampled													
Station	**Description**	**Station Selection Rationale**	**1981**	**1985**	**1986**	**1988 - 1993***	**1995**	**1996**	**1998**	**2000**	**2004**	**2007**	**2010**	**2012**	**2017**	**2022**
KSBP01	Central Basin—Point Jefferson	North Central Basin water quality station	x									x		x	x	x
LSML01	Central Basin—West Seattle	Long-term sediment monitoring station		x	x	x	x	x	x	x	x	x		x	x	x
NSEX01	Central Basin—East Passage	South Central Basin water quality station										x		x	x	x
KSRU03	Outer Salmon Bay	Potential impact from locks/vessel traffic										x		x	x	x
LSVV01	Fauntleroy Cove	History of water quality issues/ferry										x		x	x	x
MSVK01	Inner Quartermaster	Important habitat area/high										x	x	x	x	x
MSYK02	Mid Quartermaster	Near established eelgrass bed														x

*Sampled annually

4.0 SAMPLING DESIGN

In order to meet the goals of the marine ambient sediment monitoring program, it is necessary to collect data of known quality and quantity. The following sections outline the plan for sampling and analyzing ambient sediments in 2022 in the Central Basin.

4.1 Data Quality Objectives

The data quality objectives (DQOs) of the 2022 Central Basin ambient sediment sampling event outlined below are to collect data of sufficient *quantity* and *quality* and to meet the project goals outlined in Section 2.

The *quantity* of sediment chemistry data to be collected is based on previous sampling events conducted under King County's marine ambient monitoring program as well as Ecology's marine sediment monitoring program. One sediment chemistry sample will be collected from each of the seven stations, as well as three replicate benthic infauna samples for taxonomy analysis.

Project data will be assessed to determine whether the data collected are of sufficient *quality* to meet the study goals. The data quality issues of precision, accuracy, bias, representativeness, completeness, comparability, and sensitivity are described in the following subsections.

4.1.1 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 determined mean 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 sediment chemistry and benthic taxonomy may be measured by one or more of the following quality control (QC) procedures:

- analysis of various laboratory QC samples such as method blanks, matrix spikes, standard reference materials, and laboratory duplicates or triplicates for sediment chemistry; and
- analysis of triplicate benthic community samples from each station and confirmation of species identifications of the event's reference collection conducted by a secondary taxonomist.

4.1.2 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. Sediment chemistry and benthic taxonomy test samples will be collected from stations with pre-selected coordinates to represent specific site locations. Sediment chemistry samples, if taken concurrently, will be homogenized to minimize

variations in the chemical and physical composition of the sediments. Three replicate benthic taxonomy samples will also be collected to better represent the variation in the community at the site. Following the guidelines described for sampler decontamination, sample acceptability criteria, and sample processing (Section 5) will also help ensure that samples are representative.

4.1.3 Completeness

Completeness is defined as the total number of samples for which acceptable analytical data are generated, compared to the total number of samples submitted for analysis. Adhering to standardized sampling and testing protocols will aid in providing a complete set of data for this study. The goal for completeness is 100%. If 100% completeness is not achieved, the study team will evaluate whether the DQOs can still be achieved or if additional samples may need to be collected and analyzed.

4.1.4 Comparability

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

4.1.5 Sensitivity

Sensitivity is a measure of the capability of analytical methods to meet study goals. For most sediment chemistry analytical methods presented in Section 8, techniques are sensitive enough to allow comparison of sediment chemistry data to current Ecology sediment quality criteria. However, there may be some issues in meeting target detection limits for some trace organic parameters when concentrations are normalized to dry weight and to organic carbon. Refer to Section 8 for more details on how sensitivity is determined and reported for each of the analytical methods to be used for this project.

4.2 Sampling Strategy

Sediment chemistry and benthic taxonomy samples will be collected from the seven stations shown in Figure 1. Station location names, coordinates, and approximate water depths for these stations are shown in Table 2.

Table 2. 2022 Central Basin ambient sediment sampling station locations (NAD83) and depths.

Station	Description	Northing	Easting	Latitude	Longitude	Approx. Depth (m)
KSBP01	Point Jefferson	275439	1248062	47.74396	-122.42817	280
LSML01	West Seattle	209645	1242954	47.56333	-122.44333	250
NSEX01	East Passage	134701	1255331	47.35862	-122.38708	175
KSRU03	Outer Salmon Bay	247607	1253037	47.66796	-122.40564	10
LSVV01	Fauntleroy Cove	195347	1253935	47.52476	-122.39769	12
MSVK01	Inner Quartermaster Harbor	149555	1239632	47.39844	-122.45167	6
MSYK02	Mid. Quartermaster Harbor	143421	1240853	47.38170	-122.44622	3

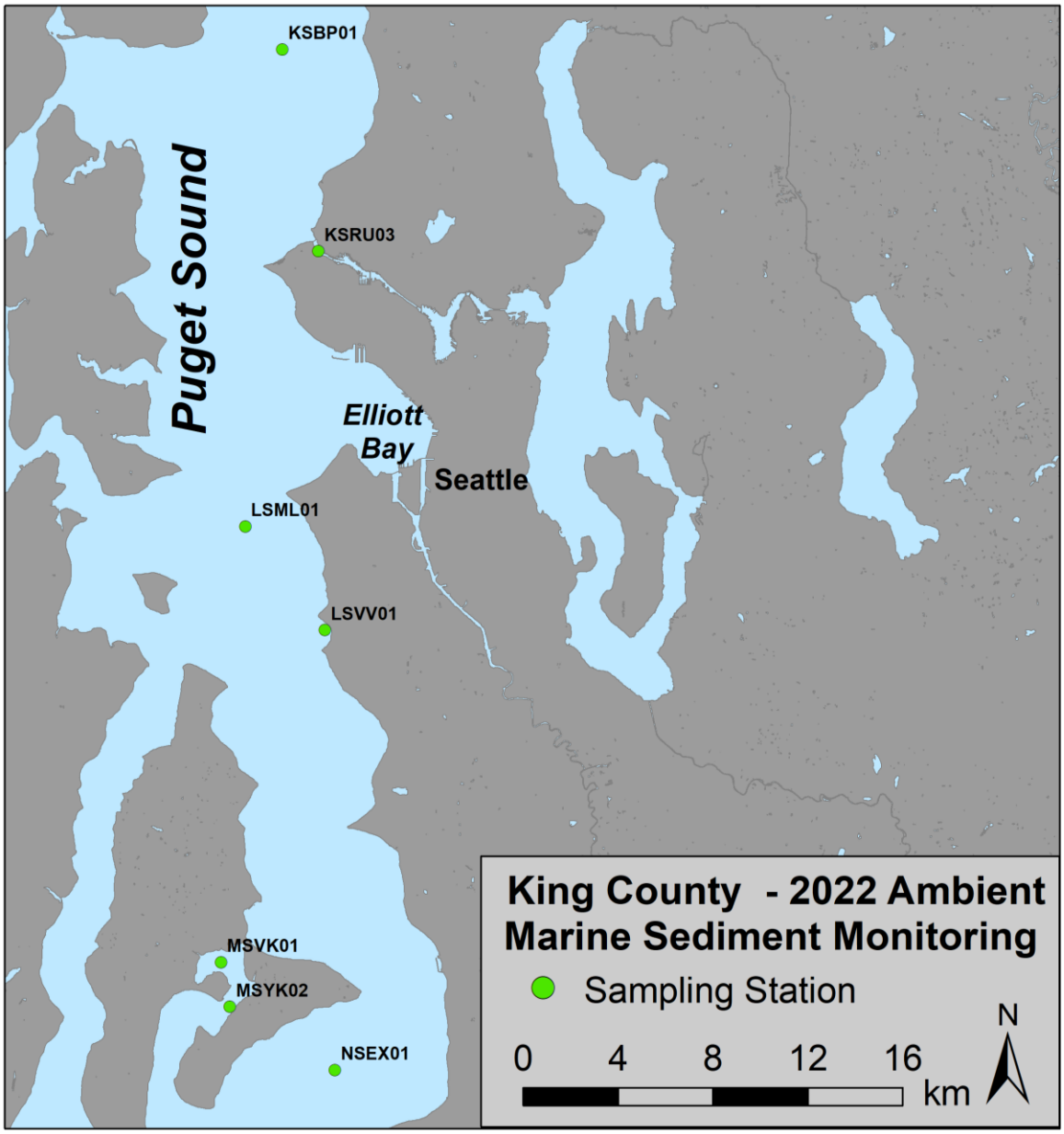

Figure 1. 2022 Central Basin ambient subtidal sediment monitoring station locations.

4.3 Sample Acquisition and Analytical Parameters

Chemistry samples will be collected at the seven stations in June of 2022 from the top 2 cm of sediment to characterize the quality of recently deposited sediment. Sampling procedures are described in Section 5. Sediment will be homogenized and split into laboratory containers in the field. Parameters of interest will include metals and organic compounds, as well as conventional sediment chemistry and physical properties. Analytical parameters have been chosen based on guidance from Ecology's *SCUM* (Ecology, 2021) and for consistency with Ecology's ambient sediment monitoring program, and King County's historical sediment monitoring dataset. Analytical parameters will include:

- conventionals—ammonia, particle size distribution (PSD), total organic carbon (TOC), total solids, and total sulfide;
- metals—aluminum, arsenic, cadmium, chromium, copper, iron, lead, mercury, nickel, silver, tin, and zinc; and
- organics—base/neutral/acid semivolatile organic compounds (BNAs), butyltins, chlorinated pesticides, PBDEs, polychlorinated biphenyls (PCBs), and total 4-nonylphenol (see Section 8.4 for a complete list of organic parameters).

Benthic taxonomy samples will be collected in triplicate, concurrently with chemistry samples and from all seven stations. Each of these sediment samples will consist of the entire contents of a single van Veen grab sampler. The deployment of tandem van Veen grab samplers will allow for the concurrent collection of the chemistry and benthic taxonomy samples.

4.4 Data Analysis

Sediment chemistry data will be evaluated by comparison with SMS sediment chemical criteria from Table A-1 of Chapter 173-204 WAC (Ecology, 2021; also see Appendix A). Sediment data for some organic compounds are typically normalized to organic carbon content for comparison to SMS criteria. Normalization to organic carbon can produce biased results, however, when the organic carbon content of the sample is very low (Ecology, 1992). When the organic carbon content of a sample is near 0.1 or 0.2% (1,000 to 2,000 milligrams/kilogram (mg/Kg) dry weight), even background concentrations of certain organic compounds can exceed sediment quality criteria. If the organic carbon content at any particular station is below 0.5% or above 3.5% dry weight, then dry weight-normalized results for non-ionizable organic compounds will be compared to Apparent Effects Thresholds (AET) criteria (EPA, 1988; Ecology, 2021), rather than SMS criteria. Concentrations of SMS and non-SMS chemicals will be compared to results from previous King County and regional monitoring data.

Benthic taxonomy results will be compared to historical data from the same sites as well as to Puget Sound regional benthic data from areas of similar physical characteristics such as depth, grain size distribution, and organic carbon content. Data from the three replicate samples at each of the seven stations will be used to calculate averaged diversity index

values and species abundance and richness (total abundance and richness as well as abundance and richness of each major taxon).

5.0 SAMPLING PROCEDURES

Sampling for sediment chemistry and benthic infauna will be conducted in June of 2022 to be consistent with previous monitoring dates. All sampling will be performed according to guidelines recommended in *SCUM* and the Puget Sound Estuary Program's (PSEP) Puget Sound Protocols (PSEP, 1987; Ecology, 2021). Checklists of supplies for sediment chemistry and benthic infauna sampling are available in Appendix B.

Sediment samples will be collected using two 0.1 m^2 modified, stainless steel van Veen grab samplers deployed in tandem via hydrowire and hydraulic winch from the King County research vessel *SoundGuardian*. Sediment will be collected from the contents of a minimum of three deployments of the dual grab samplers to obtain the volume needed for required analyses. Chemistry samples will be collected from the top 2 cm of sediment. Sediment chemistry samples will be stored on ice in coolers while in the field, and then transported to the King County Environmental Laboratory (KCEL) at the end of each sampling day.

Benthic infauna samples will be collected from the entire contents of a single grab. Samples will be sieved and preserved in buffered formalin in the field by KCEL staff and transferred to subcontracted personnel for later sorting, identification, and quantification. Established chain of custody (COC) procedures will be followed for this sampling event.

5.1 Station Positioning

Reliable station positioning is crucial to be able to revisit established stations for future sampling events. Inaccuracies in station positioning when conducting sediment grab sampling in deep water can result from the action of currents and wind on the sampling vessel as well as current forces and viscous drag on the sampler and deployment line.

Station positioning for the 2022 ambient sediment sampling event will employ a combination of a differential global positioning system (DGPS), and positioning software to calculate the position of the van Veen grab samplers as they contact the seafloor.

For station positioning, the research vessel will employ a Trimble® DGPS. Prior to the sampling event, the prescribed station coordinates will be entered into the shipboard DGPS laptop computer. During the sampling event, the shipboard navigational system will utilize the differential data transmissions from regional Coast Guard base stations to automatically correct its GPS satellite data. The GPS antenna is boom-mounted above the sampler descent line to achieve a more accurate coordinate fix above the sampling point.

Upon contact of the grab samplers with the bottom, the coordinate data representing the sediment grab impact point will be electronically recorded in real time. Positioning information will include local time and date that a position is recorded, comments, and coordinate data in both latitude/longitude and NAD 83 State Plane formats.

Sample collection is expected to take place within a 6-meter radius of each station's prescribed position and samples will not be collected if the observed coordinates of the grab deployment are outside of this limit. If conditions such as a steep slope or rocky substrate preclude sample collection at a particular station, the station may be relocated after consultation with the study coordinator if relocation will not compromise the project goals. If the study coordinator is not available, the sampling team will use best professional judgement to collect a sample as close as possible, but outside of the 6-meter radius. Any station relocation will be documented and reported.

5.2 Sampler Deployment and Retrieval

Two 0.1 m^2, modified, stainless steel van Veen grab samplers will be deployed in tandem at each sampling station. The grab samplers will be lowered at a controlled speed of approximately 30 meters per minute until they approach the bottom, at which time the speed will be decreased to approximately 10 meters per minute to minimize potential bow wake activity and subsequent bottom disturbance.

After the grabs have tripped upon reaching the bottom, they will be raised slowly to allow gentle and complete closure of the sampler jaws to avoid sample disturbance and loss. Once clear of the bottom, the ascent speed will be increased to approximately 30 meters per minute. Care will be taken to ensure that minimal sample disturbance occurs when swinging the grabs on board. Collection of undisturbed sediment requires that the grab samplers:

- create a minimal bow wake when descending;
- form a leak-proof seal upon closure of the jaws;
- are carefully retrieved to prevent excessive sample disturbance; and
- allow easy access to the sediment within the grab.

5.3 Sample Acceptability Criteria

When the grab samplers have been secured on board, the hinged top flaps will be opened and the samples examined for acceptability. Acceptability criteria will include:

- the grabs are not overfilled to the point where there is evidence of sample loss around the access doors;
- overlying water is present, indicating minimal leakage;
- overlying water is not excessively turbid, indicating minimal sample disturbance; and
- a minimum acceptable sample penetration depth has been achieved.

Samples collected from the top 2 cm of sediment for chemistry analysis will require a minimum grab penetration depth of 4 cm. The full grab is used for benthic infauna samples, and the minimum grab penetration depth is 11 cm in order to include the biologically

active zone (top 10 cm). Sampling coarser sediments at deeper depths can occasionally prove problematic when trying to obtain the required penetration depth. If multiple grab deployments at any station fail to recover the correct amount of sediment, subsequent grab deployments will be accepted with less than 4 cm of sediment for chemistry and 11 cm for benthic infauna, based on the discretion of the lead sampler in consultation with the study coordinator. This situation will be documented on field notes.

Care will be taken to extract sediment for chemistry analysis from the most undisturbed center portion of each grab without collecting sediment that has touched the sides or bottom of the grab. Penetration depth will be determined by measuring the depth of sediment within each grab by sliding a ruler vertically along the inside of the grab's side wall after each successful cast. Penetration depth can also be calculated by measuring the space between the sediment's surface within the grab and the top of the grab, then subtracting this vertical distance from 17 cm, the total inside height of the grab. Overlying water within the grab will be carefully siphoned off of the sediment surface for all acceptable samples.

Samples collected from the 0 to 2 cm depth stratum will require a minimum of three successful deployments of the tandem van Veen grab samplers. Sediment chemistry aliquots will be collected from one or both of the grabs from each successful deployment with benthic taxonomy samples collected from the remaining grabs. The three grab deployments will result in enough sediment to composite material for all chemistry testing as well as three replicate benthic taxonomy samples.

5.4 Sample Processing—Sediment Chemistry

Prior to any subsampling, sediment aliquots will be collected for analysis of total sulfides. The sulfide samples will always be collected from the top 2 cm of sediment in the first pair of grabs, using a stainless steel spoon and placed directly into the appropriate sample containers without homogenization. It is necessary to preserve total sulfide samples as soon as possible. After the four ounce total sulfides container has been filled completely, 5 milliliters (ml) of 2N zinc acetate will be added to the top of the sediment prior to sealing the container.

The remaining top 2 cm of undisturbed sediment from the same grab will be collected and then placed in a stainless steel bowl. Sediment will be collected from the 0 to 2 cm depth stratum using a stainless steel spoon. When sufficient sediment for all analyses has been collected, the sediment in the stainless steel bowls will be thoroughly homogenized. After homogenization, sediment aliquots will be transferred to appropriate laboratory containers. Prior to homogenization, collected sediments will be stored covered with aluminum foil in coolers between grab deployments.

Head space will be left in all chemistry sample containers, with the exception of total sulfides, to allow for further mixing in the laboratory and expansion should the containers be stored frozen. All sample containers will be stored in insulated, ice-filled coolers while in

the field. All sediment chemistry sample containers, storage conditions, and hold times are summarized in Table 3.

Table 3. Sediment sample containers, storage conditions, and analytical hold times.

		Preferred		Acceptable	
Analyte	**Container**	**Storage Conditions**	**Hold Time**	**Storage Conditions**	**Hold Time**
Ammonia	4-oz. CWM PP	refrigerate at 4°C	7 days to analyze	N/A	N/A
Particle Size Distribution	16-oz. CWM PP	refrigerate at 4°C	6 months to analyze	N/A	N/A
Total Organic Carbon	4-oz. CWM PP	freeze at -18°C	6 months to analyze	refrigerate at 4°C	28 days to analyze
Total Solids (collect w/ TOC)	4-oz. CWM PP	freeze at -18°C	6 months to analyze	refrigerate at 4°C	14 days to analyze
Total Sulfide	4-oz. CWM PP No Headspace	refrigerate at 4°C w/ 2N Zn acetate	7 days to analyze	N/A	N/A
Mercury	8-oz. CWM PP	freeze at -18°C	1 year to analyze	N/A	N/A
Other Metals (Collect with Mercury)	8-oz. CWM PP	freeze at -18°C	2 years to analyze	refrigerate at 4°C	6 months to analyze
BNAs	8-oz. glass	freeze at -18°C	1 year to extract 40 days to analyze	refrigerate at 4°C	14 days to extract 40 days to analyze
Tributyltin	8-oz. glass	freeze at -18°C	1 year to extract 40 days to analyze	refrigerate at 4°C	14 days to extract 40 days to analyze
Pesticides/PCBs (collect w/ BNAs)	8-oz. glass	freeze at -18°C	1 year to extract 40 days to analyze	refrigerate at 4°C	14 days to extract 40 days to analyze
PBDEs (collect w/ BNAs)	8-oz. glass	freeze at -18°C	1 year to extract 40 days to analyze	refrigerate at 4°C	14 days to extract 40 days to analyze

CWM PP – Clear, wide-mouth polypropylene, TOC = total organic carbon, BNAs = base/neutral/acid semivolatile organic compounds, PCBs = polychlorinated biphenyls, PBDES = polybrominated diphenyl ethers

5.5 Sample Processing—Benthic Taxonomy

One grab from each successful deployment of the dual van Veen grab samplers will be used to collect sediment for benthic taxonomy analysis. Benthic taxonomy samples will be collected concurrently with sediment chemistry samples. Sediment from acceptable grabs will be flushed with ambient seawater through a 1.0-millimeter sieve to remove all fine material. The remaining sediment will then be transferred into 1-liter plastic containers using a minimum amount of seawater.

A 10% solution of buffered formalin will be added to the sample to preserve all tissues prior to sealing the container. Samples will be labeled both on the inside and outside of the container[1], then recorded on chain of custody forms, and placed in coolers. Benthic samples will remain in formalin solution for at least 24 hours but not longer than two weeks. Benthic samples will be rescreened at the contract laboratory from the 10% buffered formalin solution to 70% ethanol as recommended by PSEP protocols (PSEP, 1987).

5.6 Sampling Equipment Decontamination

The grab samplers will be decontaminated between sampling stations by scrubbing with a brush and ambient sea water, followed by a thorough in situ rinsing. Prior to sampling, stainless steel bowls and spoons will be washed and scrubbed in detergent (as specified in KCEL SOP#234). The buckets and utensils will be rinsed with copious quantities of reverse osmosis water, allowed to dry in air, and covered in aluminum foil until used in the field. A separate stainless steel bowl and spoon (and spatula, if necessary) will be dedicated to each sampling station, precluding the need for decontamination of this equipment in the field.

5.7 Sample Storage and Delivery

All sample containers will be stored in an insulated cooler containing ice immediately after collection to maintain the samples at a temperature of approximately 4° Celsius until delivery to the laboratory. Sample containers from each station will be grouped and placed in plastic bags to facilitate sample receipt and login. At the end of each sampling day, all samples will be transported back to KCEL.

5.8 Chain of Custody

Chain of custody (COC) will commence at the time that each sample is collected. While in the field, all samples will be under direct possession and control of King County field staff. For chain of custody purposes, the research vessel will be considered a "controlled area." Each day, all sample information will be recorded on a COC form (Figure 2). This form will be completed in the field and will accompany all samples during transport and delivery to the laboratory each day. Upon arrival at KCEL, the sample delivery person will relinquish all samples 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 refrigerator until the next day. Samples delivered to a contracted laboratory will be accompanied by a properly completed KCEL COC form and custody seals will be placed on the cooler if samples are delivered by an outside courier. Contracted laboratories will be expected to provide a copy of the completed COC form as part of their analytical data package.

[1] Interior labels are made on waterproof paper using pencil. Exterior labels are made on waterproof label and indelible ink.

LABORATORY WORK ORDER
Chain of Custody

King County Department of Natural Resources
Water and Land Resources Division
Environmental Laboratory
322 West Ewing Street
Seattle, Washington 98119-1507

Project Name:
Project Number:
Laboratory Project Manager:
Telephone Number:

				Analyses															
Sample Number	Locator	Collect Date	Collect Time	BNAS	BUTYLTIN	CHLOROBENZENES	METHYL MERCURY	PCBS	METALS - ICP	MERCURY - CVAA	AMMONIA	OIL & GREASE	PSD	TOC	SOLIDS	SULFIDE		Number of Containers	Comments

6.0 SAMPLE AND FIELD DOCUMENTATION

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

- Field sheets generated by King County's Laboratory Information Management System (LIMS) that will include information such as:
  1. sample ID number
  2. station name
  3. station bottom depth
  4. sediment depth (i.e., sampler penetration depth) for each successful deployment
  5. sediment depth stratum sampled (i.e., 0 to 2 cm)
  6. physical sediment characteristics
  7. date and time of sample collection
  8. condition and height of tide
  9. initials of all sampling personnel
- LIMS-generated container labels will identify each container with a unique sample number, station and site names, collect date, analyses required, and preservation method.
- *SoundGuardian's* logbook will contain records of all shipboard activities, destinations, arrival and departure times, general weather and positioning information, and the names of shipboard personnel.
- *SoundGuardian's* cruise plan will list the prescribed stations to be sampled, along with their respective coordinates and other associated locating information.
- Electronic DGPS coordinate data will be electronically logged for each acceptable grab sample using both latitude/longitude and NAD 83 State Plane formats.
- COC documentation will consist of the Lab's standard COC form, which is used to track release and receipt of each sample from collection to arrival at the lab.

A sample of a typical field sheet used by KCEL is included as Figure 3.

The following field measurements and observations will be recorded on the appropriate field sheet/log for each sample:

- sample (bottom) depth—measured as total depth by vessel's fathometer;
- coordinates for individual grab samples;
- sediment depth (grab penetration depth)—measured by ruler inside the grab;

- sediment sampling range (0 to 2 cm);
- sediment type (a mnemonic code indicating color, gross grain size, odor, and debris);
- tide condition and height;
- collect date, start time, and sampling personnel; and
- general sample information such as sampling irregularities (e.g., inferior sample material due to the presence of rocks, debris, etc.).

Login: P7xxxx **Marine Ambient Subtidal 2022** **FSU TC:** _____
Project: 421250-300 **LPM:**

CHAIN OF CUSTODY

	Relinquished by	***Date***	***Time***
	Received by	***Date***	***Time***
	Sample Numbers		***[All]***

Sample Number	**P7xxxx-1**	**P7xxxx-2**	**P7xxxx-3**
QC Link			
Locator	**KSBP01**	**KSRU03**	**LSML01**
Short Loc Desc	PT JEFF	Salmon Bay	S ALKI PT
Locator Desc	CARKEEK AREA OFFSHORE//POINT JEFFERSON	SALMON BAY / OUTSIDE LOCKS	ALKI AREA OFFSHORE//
Site	AMBIENT OFFSHORE	AMBIENT OFFSHORE	AMBIENT OFFSHORE
Comments	2 grab COMP, 0 - 2 cm	2 grab COMP, 0 - 2 cm	2 grab COMP, 0 - 2 cm
Start Date/Time			
End Date/Time			
Time Span			
Sample Depth			
PERSONNEL			
SAMP METH			
SED DEPTH			
SED SAMP RANGE			
SED TYPE			
TIDE COND			
TIDE HT			
Dept, Matrix, Prod (Cont ID)	3 SF NH3-KCL (17) 3 SF PSD (23) 3 SF TOC-QL; TOTS (17) 3 SF TOTSULFIDE-QL (17) 6 SF CVAA-M-QL; ICP-QL (19) 7 SF BNASMS-NPDES-QL; BNASMS-NPDES-SIM-QL; CLPEST-QL; PBDE-QL; PCB-QL; TRIBUTYLTIN-QL (20)	3 SF NH3-KCL (17) 3 SF PSD (23) 3 SF TOC-QL; TOTS (17) 3 SF TOTSULFIDE-QL (17) 6 SF CVAA-M-QL; ICP-QL (19) 7 SF BNASMS-NPDES-QL; BNASMS-NPDES-SIM-QL; CLPEST-QL; PBDE-QL; PCB-QL; TRIBUTYLTIN-QL (20)	3 SF NH3-KCL (17) 3 SF PSD (23) 3 SF TOC-QL; TOTS (17) 3 SF TOTSULFIDE-QL (17) 6 SF CVAA-M-QL; ICP-QL (19) 7 SF BNASMS-NPDES-QL; BNASMS-NPDES-SIM-QL; CLPEST-QL; PBDE-QL; PCB-QL; TRIBUTYLTIN-QL (20)

Page 1 of 3

Figure 3. King County Environmental Laboratory standard field sheet.

7.0 PROJECT HEALTH AND SAFETY

The following general health and safety guidelines have been provided in lieu of a site-specific Health and Safety Plan. These guidelines will be read and understood by all members of the sampling crew.

- All crew of the research vessel will follow all aspects of the KCEL Vessel Safety Plan.
- Samplers will wear chemical-resistant gloves when coming into contact with sediment.
- No eating or drinking by sampling personnel will be allowed during active sampling operations.
- All sampling operations will be conducted during daylight hours.
- All accidents, "near misses," and symptoms of possible exposure will be reported to a crew member's supervisor within 24 hours of occurrence.
- All crew members will be aware of the potential hazards associated with any chemicals used during the sampling effort.

In addition, KCEL staff will follow current laboratory COVID-19 safety protocols while collecting and analyzing sediment samples.

Several hazards are inherent to marine sediment sampling. General vessel safety, physical hazards unique to sediment grab sampling, and chemical hazards are discussed in Sections 7.1 through 7.3.

7.1 General Vessel Safety

To help prevent accidents and ensure adequate preparation for emergencies that may possibly arise, the following safety equipment will be required on the *SoundGuardian*:

- one personal floatation device (PFD) for each crew member and one throwable PFD;
- an accessible, clearly labeled, fully stocked first-aid/CPR kit;
- an accessible and clearly labeled eye wash (when sampling suspected contaminated sediments);
- one (preferably two) VHF marine radio(s) with weather channel;
- a cellular telephone;
- a horn;
- navigation lights;
- an emergency life raft;
- an anchor and suitable line;
- signal flares; and
- a reach pole or shepherd's hook.

Personal protective equipment will be selected and used that will protect workers involved in sediment sampling from the hazards and potential hazards likely to be encountered. Minimum required personal protective equipment for marine sediment sampling shall include the following:

- hard hat;
- steel-toe rubber boots;
- chemical-resistant gloves (e.g., Nitrile); and
- safety glasses.

Recommended additional personal protective equipment will include rain gear and hearing protection when the *SoundGuardian* is under way.

7.2 Grab Sampling

Sampler deployment and sediment retrieval present physical hazards due, in part, to the heavy weight of the grab sampler, its suspension above the vessel deck, and the risk of accidental or premature closure. Prior to each sampling event, all cabling, shackles, pins, housings, and swivels will be inspected to ensure the integrity of all points along the sampling assembly.

The sampler will always be set while it is resting on a stable surface. Once set, a safety pin will be set in place on the triggering mechanism and remain in place until the sampler is swung outboard of the vessel rail. Special care will be exercised when removing the safety pin to ensure personal safety in the event of a gear or winch failure. Fingers will not be placed through the ring of the pin when it is removed and hands will be kept completely clear of the sampler interior after the pin has been removed. If a sampler is retrieved that has not been tripped, it will be lowered to a stable surface before any worker contact.

During grab retrieval, one crew member will watch for the appearance of the grab sampler and alert the winch operator when the sampler is first visible below the water surface. Attempting to bring a swinging grab sampler on board poses a serious risk of being hit or knocked overboard. The winch operator will minimize swinging before the grab sampler is brought on board for the crew to secure. Hard hats and gloves will always be worn when handling the grab sampler.

The winch drum, blocks, A-frame, and any area between the grab sampler and railings, the deck, and heavy equipment, all represent significant pinching and crushing hazards. Only trained and experienced crew members will operate the winch or A-frame during a sampling event. Other crew members will exercise care to avoid these potentially hazardous areas.

7.3 Chemical Hazards

Contact with marine sediment at some sampling stations may present a health hazard from chemical constituents of the sediment, such as PCBs, mercury, phthalates, and PAHs.

Potential routes of exposure to chemical hazards include inhalation, skin and eye absorption, ingestion, and injection. Crew members will exercise caution to avoid coming into contact with sediment at all stations during sampling operations. Protective equipment will include chemical-resistant gloves, safety glasses or goggles, and protective clothing (e.g., rain gear). Crew members will exercise good personal hygiene after sampling and prior to eating or drinking.

8.0 SEDIMENT CHEMISTRY ANALYTICAL METHODS AND QUALITY ASSURANCE/QUALITY CONTROL

Analytical parameters for sediment chemistry samples are presented in the following subsections. Parameters have been selected based on guidance for conducting sediment characterization in *SCUM* (Ecology, 2021) and will allow comparison with published sediment quality criteria (EPA, 1988; Ecology, 2013, 2021). Analyses will be performed at KCEL and will follow *SCUM* guidelines (Ecology, 2021).

The terms MDL and RDL, used in the following sediment chemistry analysis subsections, refer to method detection limit and reporting detection limit, respectively. The MDL is defined as the minimum concentration of a chemical constituent that can be detected, while the RDL is defined as the minimum concentration of a chemical constituent that can be reliably quantified.

SW-846 methods for metals, organic compounds, and some conventionals analyses have eliminated the concept of the method detection limit discussed above and have replaced it with a lower limit of quantitation (LLOQ) concept. Details on how these quantitation limits are used to report data by KCEL are described in Section 8.1. Methods and the resulting detection and reporting limit goals are described in sections 8.2 through 8.4.

8.1 Quantitation Limits

The sensitivity of a particular analytical method is generally determined/defined by the reference method. The EPA reference methods used for this project belong mostly to the SW-846 compendium of analytical methods (including conventionals analyses TOC and total sulfide) and require that sensitivity be defined as a single limit called the "lower limit of quantitation" (LLOQ). With the exception of the metals Method 6010D and 7471B, the LLOQ can be no lower than the lowest concentration on the calibration curve and must be verified according to the requirements in each reference method. For metals Method 6010D and 7471B, the LLOQ may be lower than the lowest concentration on the calibration curve but must be verified within each analytical run by analyzing a standard at the level of the LLOQ and recovering the standard within 80%-120% of its true value (Appendix C).

For the metals (6010D, 7471B), conventionals, (9030B, 9060A), and chlorinated pesticides/PCBs (8081B (02/07)[2]/8082A (02/07)) methods, sample results may not be reported below the LLOQ. In this case, the LLOQ value for each parameter is used as both the LIMS MDL and LIMS RDL (LIMS refers to the KCEL Laboratory Information Management System).

[2] The month/year of current SW846 method update is provided for each method as appropriate.

Sample results for BNAs (Method 8270E (06/18)), PBDEs (3550C (02/07)/8270E (06/18) NCI), and butyltins (Krone et al., 1989 and 8270E (06/18) SIM) may be reported at concentrations below the LLOQ if the qualitative peak identification requirements of the reference method are met. The LLOQs for BNA, PBDE, and butyltin parameters will be reported in the LIMS RDL field, while the LIMS MDL column will be at most one-half the LLOQ. The LIMS MDL may be lower than one half the LLOQ if the qualitative peak identification requirements of the reference method are fulfilled. Therefore, the LIMS MDL values for these parameters represent the lowest concentration at which a given compound meets the qualitative peak identification criteria specified in the reference method.

Any BNA, PBDE or butyltin parameters reported below the LLOQ (LIMS RDL) will be flagged "<QL, J," because any result reported below the lower limit of quantitation (referred to as QL in LIMS) must be reported as an estimated value. See Section 10.1.1 for definitions of data qualifiers.

For the tests based on non-SW-846 reference methods, sensitivity is defined as the MDL, which is calculated by the 40 CFR Part 136, Appendix B, Revision 2 (2016) procedure. The value determined by this procedure may be increased to account for method variability, and will be reported in the LIMS MDL field. The LIMS RDL is calculated by multiplying the LIMS MDL by a factor between 2 and 10, depending on the parameter, and should be considered analogous to the Practical Quantitation Limit or (PQL).

Every effort will be made to achieve method LLOQs below the appropriate SMS chemical criteria (listed in Appendix A) for each parameter. The ability of the laboratory to attain detection limits that meet organic carbon-normalized chemical criteria, however, will depend upon the total solids and TOC content of each sample. Results for certain non-ionizing organic compounds are normalized to organic carbon for comparison to SMS chemical criteria. However, for sediment with a TOC content less than 0.5% (5,000 mg/Kg DW) or greater than 3.5% (35,000 mg/Kg DW), Ecology recommends comparing dry weight-normalized, non-ionic organic data to the Puget Sound AETs. These values are provided in Table 8-1 of *SCUM* (Ecology, 2021) and are considered to be equivalent to the organic carbon normalized benthic SMS values.

Every effort will be made to meet the MDL/RDL goals listed in the following sections of this SAP. However, the values for the LIMS MDLs and LIMS RDLs reported for individual samples may differ from the target detection limit goals reported below in Sections 8.2 thru 8.4. This may occur as a result of necessary analytical dilutions or reductions of extracted or digested sample amounts based on preliminary examination of the sample (including total solid and TOC values). When samples must be diluted for the analysis of one or more target analytes, LIMS MDLs and LIMS RDLs from the original, undiluted extract will be reported for all other parameters that did not require dilution. If it is determined that it will not be possible to meet the SMS criteria based upon TOC and total solids values, SW-846 method criteria, or KCEL Standard Operating Procedure criteria, the project manager will immediately be consulted.

LLOQs, MDLs, and RDLs are periodically evaluated and may change up or down. Any changes to the instrument LLOQ values will be documented in a Data Anomaly Form as a deviation from the SAP. If for any reason after analysis, a non-detected parameter's LLOQ/MDL was raised and is no longer less than or equal to the sediment quality criteria in Appendix A (e.g., required dilution due to matrix interferences or QC failures), the Project Manager will be contacted prior to data being moved to the LIMS database. This will allow a discussion on the implications of not meeting the limits and any other potential corrective actions that might be taken.

8.2 Conventionals

Conventional sediment parameters will include ammonia, particle size distribution (PSD), total organic carbon (TOC), total solids, and total sulfide. The analytical methods and detection limits for conventional parameters are summarized in Table 4.

Table 4. Conventionals target analytes, methods, and detection limit goals.

Parameter	Method	MDL	RDL	Units
Ammonia	Kerouel & Aminot, 1997	0.05	0.1	mg/Kg wet wt.
PSD (gravel and sand)	ASTM D422	0.1	1.0	percent dry wt.
PSD (silt and clay)	ASTM D422	0.5	1.0	percent dry wt.
Total Solids	SM 2540-G	0.005	0.01	percent wet wt.
Parameter	**Method**	**MDL/RDL/QL***		**Units**
Total Organic Carbon	EPA 9060; PSEP, 1996	500		mg/Kg wet wt.
Total Sulfide	EPA 9030B/SM 4500-S2-D	0.5		mg/Kg wet wt.

*QL refers to the Lower Limit of Quantitation (LLOQ) and is equal to the LIMS MDL and RDL for TOC and Total Sulfide.

Total solids will be analyzed on all samples to allow normalization of all other sediment chemistry data except PSD to dry weight. Total solids analysis will be performed according to the latest edition of Standard Methods (SM) 2540-G (APHA, 2017), which is a gravimetric determination. Results for total solids analyses are reported in units of percent on a wet weight basis.

TOC analysis will be performed on all samples to allow normalization of some organic parameters to organic carbon, if warranted. TOC analysis will be performed according to EPA Method 9060 (11/99), which is high-temperature combustion with infrared spectroscopy. Results for TOC analysis are reported in units of mg/Kg on a wet weight basis and/or percent on a wet weight basis. The MDL/RDL/QL value shown in Table 4 will be adjusted for individual samples, based on the amount of sample analyzed.

PSD analysis will be performed according to ASTM Method D422 (ASTM, 2002), which is a combination of sieve and hydrometer analyses. Results for PSD analysis are presented in units of percent on a dry weight basis, both for phi sizes and for the four broad classifications of clay, silt, sand, and gravel. Results for the clay and silt fractions are also

summed to report a result for "percent fines." The MDL and RDL values shown in Table 4 will be adjusted for individual samples, based on the amount of sample analyzed.

Ammonia and total sulfide, which are indicators of potential sediment toxicity, will be analyzed by Kerouel & Aminot, 1997 and SM 4500-S2-D (EPA 9030B), respectively. Ammonia analysis will involve a potassium chloride extraction followed by fluorometric analysis of the extract. Total sulfide will be analyzed by distillation following acidification and colorimetric analysis of the distillate. The MDL/RDL/QL value shown in Table 4 will be adjusted for individual samples, based on the amount of sample analyzed and any required dilutions.

8.3 Metals

Detection limits for the twelve target metals are summarized in Table 5. These LLOQs/LIMs MDLs/LIMS RDLs are presented on a wet-weight basis and are based on an initial sample weight of 1 (+/- 0.05) gram (g) and a final volume of 50 milliliters (ml) for ICP metals and 100 ml for mercury. Mercury will be analyzed by cold vapor atomic absorption spectroscopy (CVAA), following EPA Method 7471B (SW-846) and other metals will be analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES) with a strong acid digestion, following EPA Method 3050B/6010D (SW8-846).

Table 5. Trace metals target analytes and detection limit goals (mg/Kg wet weight).

Analyte	MDL/RDL/QL*		Analyte	MDL/RDL/QL*
Aluminum**	5.00		Lead	2.50
Arsenic	2.50		Mercury	0.005
Cadmium	0.250		Nickel	0.500
Chromium	0.250		Silver	0.500
Copper	0.500		Tin	1.00
Iron	2.50		Zinc	0.250

*QL refers to the Lower Limit of Quantitation (LLOQ) and is equal to the LIMS MDL and RDL for the metals listed.

**Even though the sampling equipment is wrapped or covered with aluminum foil, any possible contribution of aluminum from the foil is considered insignificant in comparison to the level of aluminum in the sediment.

8.4 Organics

Organic parameters will include base/neutral/acid extractable semivolatile compounds (BNAs), chlorinated pesticides, polychlorinated biphenyls (PCBs), butyltins, and polybrominated diphenyl ethers (PBDEs). The analytical methods are described below and detection limits for the target trace organic compounds are summarized in Tables 6 – 9 below (presented on a wet-weight basis).

Results for certain non-ionizing organic compounds are generally normalized to organic carbon for comparison to SMS marine chemical criteria. However, normalization to organic carbon can produce biased results when the organic carbon content of the sample is very low (Ecology, 1992). When the organic carbon content of a sample is near 0.1 or 0.2%

(1,000 to 2,000 mg/Kg DW), even background concentrations of certain organic compounds can exceed the SQS Sediment Quality Standards (SQS).

For sediment with a TOC content less than 0.5% (5,000 mg/Kg DW) or greater than 3.5%, Ecology recommends comparing dry weight-normalized, non-ionic organic data to the Puget Sound AET chemical criteria (EPA, 1988; Ecology, 2021), which are considered to be equivalent to the SQS criteria. This comparison allows a more appropriate evaluation of sediment quality relative to organic compounds (Ecology, 1992, 2021).

For samples with measured dry weight-normalized TOC concentrations between 0.5% and 3.5%, KCEL will normalize the appropriate trace organics data on an organic carbon basis.

For each reported parameter, every effort will be made to achieve analytical method LLOQs below the appropriate SQS criteria listed in Appendix A Table A-1. However, the ability of the laboratory to attain detection limits which meet organic carbon-normalized chemical criteria will depend upon the total solids and TOC observed for each individual sample.

8.4.1 BNAs

The detection limit goals for the target BNA compounds are summarized in Table 6. These LIMS MDLs and RDLs are presented on a wet-weight basis and are based on a 30 g extraction concentrated to a final volume of 1.0 ml for analysis. The LIMS RDL is equal to the LLOQ, which in LIMS will be referred to as the QL (quantitation limit). The LIMS MDL is at most one-half of the LLOQ, and may be lower if the qualitative peak identification requirements of the method are met. Note that the detection limits can vary if the amount of sample extracted deviates from 30 g, or if dilutions are required due to elevated target analyte concentration(s) and/or interferences from high background levels of non-target organic compounds.

For the BNA extraction, samples are sonicated in 1:1 methylene chloride:acetone (3550C (02/07)) and then undergo a gel permeation chromatography cleanup. BNA analysis will be performed according to EPA method 8270E (06/18), using analysis by gas chromatography/mass spectroscopy (GC/MS).

For the majority of parameters listed in Table 6, the detection limits shown are achieved operating the GC/MS in standard scan mode. However, detection limits for the parameters in Table 6 denoted with a "**" are achieved operating the GC/MS in selected ion monitoring (SIM) scanning mode, with method optimization for early-eluting acid compounds. Because sample analysis in SIM mode involves an additional GC/MS analytical run, SIM mode is reserved for analytes where standard scan mode is not likely to achieve quantitation limits (LLOQs) that are below the appropriate SQS criteria.

Table 6. BNA target analytes and detection limits (µg/Kg wet weight).

Analyte	MDL	RDL/QL*		Analyte	MDL	RDL/QL*
1,2,4-Trichlorobenzene	1.7	3.33		Coprostanol	TBD	TBD
1,2-Dichlorobenzene	1.7	3.33		Dibenzo(a,h)anthracene	3.3	6.67
1,4-Dichlorobenzene	1.7	3.33		Dibenzofuran	3.3	6.67
2,4-Dimethylphenol**	17	33.3		Diethyl Phthalate	17	33.3
2-Methylnaphthalene	3.3	6.67		Dimethyl Phthalate**	6.7	16.7
2-Methylphenol	8.3	16.7		Di-N-Butyl Phthalate	3.3	6.67
3-,4-Methylphenol	17	33.3		Di-N-Octyl Phthalate	8.3	16.7
Acenaphthene	3.3	6.67		Fluoranthene	3.3	6.67
Acenaphthylene	3.3	6.67		Fluorene	3.3	6.67
Anthracene	3.3	6.67		Hexachlorobenzene**	0.33	0.667
Benzo(a)anthracene	3.3	6.67		Hexachlorobutadiene**	0.67	1.67
Benzo(a)pyrene	3.3	6.67		Indeno(1,2,3-Cd)Pyrene	3.3	6.67
Benzo(b,j,k)fluoranthene	5	10		Naphthalene	3.3	6.67
Benzo(g,h,i)perylene	3.3	6.67		N-Nitrosodiphenylamine**	3.3	6.67
Benzoic Acid**	100	167		Pentachlorophenol**	33	66.7
Benzyl Alcohol**	67	133		Phenanthrene	3.3	6.67
Benzyl Butyl Phthalate	3.3	6.67		Phenol	17	33.3
Bis(2-Ethylhexyl) Phthalate	6.67	13.3		Pyrene	3.3	6.67
Chrysene	3.3	6.67		Total 4-Nonylphenol	67	333

TBD = To be determined
*QL refers to the Lower Limit of Quantitation (LLOQ) and is equal to the LIMS RDL for the analytes listed.
** Selected ion monitoring (SIM) scanning mode will be used for these analytes and the detection limits for that method are reflected above.

SIM mode will be used for the following parameters:

- 2,4-Dimethylphenol
- Benzoic Acid
- Benzyl Alcohol
- Dimethyl Phthalate
- Hexachlorobenzene
- Hexachlorobutadiene
- N-Nitrosodiphenylamine
- Pentachlorophenol

Note that even with using SIM mode for benzyl alcohol and 2,4-dimethylphenol, the RDL/QL limits presented in Table 6 are not likely to meet the SMS marine Sediment Quality Standard (SQS) for those two parameters based on current instrumentation and extraction procedure. KCEL Trace Organics will be taking additional steps to reduce the detection

limits for these parameters. The first step will involve bringing a new GC/MS system online which is projected to have increased sensitivity. That instrumentation is expected to be fully-operation by June 1, 2022, but detection limits are not yet known. If detection limits are not improved with the new instrumentation, KCEL Trace Organics will use a separate extraction procedure (Soxhlet extraction, EPA Method 3540) in an attempt to optimize the acidic and phenolic sample recoveries for these analytes to achieve LLOQ values below the SQS criteria.

8.4.2 Chlorinated Pesticides and PCBs

The detection limits for the target chlorinated pesticides and PCB Aroclors® are summarized in Table 7. For each Aroclor, the LIMs MDL and RDL are equal to the LLOQ, which in LIMS is referred to as the QL. These limits are presented on a wet-weight basis and are based on a 30 g extraction, which is concentrated to a final volume of 1.0 ml for analysis. These samples are sonicated in 1:1 hexane:acetone (3550C (02/07)), and then undergo gel permeation chromatography, tetrabutylammonium (TBA), and sulfuric acid cleanups. Note that the detection limits can vary if the amount of sample extracted deviates from 30 g, or if a dilution is required due to elevated target analyte concentration(s) and/or interferences from high background levels of non-target compounds. Chlorinated pesticide/PCB analysis will be performed according to EPA methods 8081B (02/07)/8082A (02/07), gas chromatography/electron capture detector (GC/ECD) with dual column confirmation.

Table 7. Chlorinated pesticide and PCB target analytes and sample detection limit goals (µg/Kg wet weight).

Analyte	MDL/RDL/QL*		Analyte	MDL/RDL/QL*
4,4'-DDD	0.167		Endosulfan II	0.167
4,4'-DDE	0.167		Endosulfan Sulfate	0.167
4,4'-DDT	0.167		Endrin	0.167
Aldrin	0.167		Endrin Aldehyde	0.167
Alpha-BHC	0.167		Gamma-BHC (Lindane)	0.167
Alpha-Chlordane	0.167		Trans-Chlordane	0.167
Beta-BHC	0.167		Heptachlor	0.167
Delta-BHC	0.167		Heptachlor Epoxide	0.167
Dieldrin	0.167		Methoxychlor	0.833
Endosulfan I	0.167		Toxaphene	16.7
Aroclor 1016	0.833		Aroclor 1248	0.833
Aroclor 1221	2.50		Aroclor 1254	0.833
Aroclor 1232	2.50		Aroclor 1260	0.833
Aroclor 1242	0.833			

*QL refers to the Lower Limit of Quantitation (LLOQ) and is equal to the LIMS MDL and RDL for the analytes listed.

8.4.3 Tributyltin

Butyltin analysis will be performed according to Krone et al., 1989 and EPA method 8270E (06/18) SIM, which employs solvent extraction with tumbling and analysis by GC/MS in SIM mode. The detection limit goals for the target butyltin compounds are summarized in Table 8. The MDLs and RDLs are presented on a wet weight basis and are based on a 5 g extraction and a final volume of 1.0 mL for analysis. The MDLs and QLs for specific analytes requiring dilution (e.g., exceedance of analyte calibration range or matrix interferences) will be increased to reflect the dilution.

Table 8. Butyltin target analytes and sample detection limit goals (µg/Kg wet weight).

Parameter	MDL	RDL/QL*
Mono-n-Butyltin (as monobutyltin ion)	5	10
Di-n-Butyltin (as dibutyltin ion)	5	10
Tri-n-Butyltin (as tributyltin ion)	5	10
Tetra-n-Butyltin (as tetrabutyltin)	5	10

*QL refers to the Lower Limit of Quantitation (LLOQ) and is equal to the LIMS RDL.

8.4.4 PBDEs

The detection limits for target PBDE congeners are summarized in Table 9. These MDLs and RDLs are presented on a dry-weight basis (assumed 50% solids content) and are based on a 15 g extraction with gel permeation chromatography (GPC) cleanup and concentration to a final volume of 1 mL for analysis. Note that the detection limits can vary if the sample deviates from 15 g, or if dilution is required due to elevated analyte concentration(s) and/or interferences. PBDE analysis will be performed according to EPA methods 3550C (02/07)/8270E (06/18) NCI, which employs solvent extraction with sonication and analysis by gas chromatography/mass spectroscopy (GC/MS) in NCI (negative chemical ionization) SIM mode.

Table 9. PBDE target analytes and sample detection limit goals (µg/Kg wet weight).

BDE congener	Congener #	MDL	RDL/QL*
2,2',4-TriBDE	17	0.013	0.0267
2,4,4'-TriBDE	28/33	0.013	0.0267
2,2',4,4'-TetraBDE	47	0.13	0.333
2,3',4,4'-TetraBDE	66	0.033	0.0667
2,3',4',6-TetraBDE	71	0.013	0.0267
2,2'3,4,4'-PentaBDE	85	0.013	0.0267
2,2'4,4'5-PentaBDE	99	0.33	0.667
2,2',4,4'6-PentaBDE	100	0.033	0.0667
2,2',3,4,4',5'-HexaBDE	138	0.013	0.0267
2,2',4,4',5,5'-HexaBDE	153	0.013	0.0267
2,2',4,4',5',6-HexaBDE	154	0.013	0.0267

2,2',3,4,4',5',6-HeptaBDE	183	0.017	0.0267
2,3,3',4,4',5,6-HeptaBDE	190	0.013	0.0267
2,2',3,3',4,4',5,5',6,6'-DecaBDE	209	0.17	0.333

*QL refers to the Lower Limit of Quantitation (LLOQ) and is equal to the LIMS RDL.

8.5 Sediment Chemistry QA/QC

The quality control (QC) samples that will be analyzed in association with sediment chemistry samples are summarized in Table 10. The frequency of method blanks, duplicates, triplicates, and matrix spikes is one per QC batch (20 samples maximum). The frequency of SRM (standard reference material) or LCS (laboratory control sample) analysis is one per batch (20 samples maximum). LCS analysis is used in lieu of SRM analysis for selected analytes when an SRM may not be readily available. Surrogates are analyzed with every organic sample.

Table 10. Marine sediment chemistry quality control samples.

Analyte	Method Blank	Spiked Blank	Duplicate/ Triplicate	Matrix Spike (MS)	MS Duplicate	SRM/LCS	Surrogates
Ammonia	Yes	Yes	Triplicate	Yes	No	Yes	No
PSD	No	No	Triplicate	No	No	No	No
TOC	Yes	Yes	Triplicate	Yes	No	Yes	No
Total Solids	Yes	No	Triplicate	No	No	No	No
Total Sulfide	Yes	Yes	Triplicate	Yes	No	No	No
Mercury	Yes	Yes	Duplicate	Yes	Yes	Yes	No
Other Metals	Yes	Yes	Duplicate	Yes	No	Yes	No
BNAs	Yes	Yes	Duplicate	Yes	Yes	Yes	Yes
Butyltins	Yes	Yes	Duplicate	Yes	Yes	Yes	Yes
Chl. Pesticides	Yes	Yes	Duplicate	Yes	Yes	Yes	Yes
PCBs	Yes	Yes	Duplicate	Yes	Yes	No	Yes
PBDEs	Yes	Yes	Duplicate	Yes	Yes	Yes	Yes

PSD = particle size distribution; TOC = total organic carbon; BNAs = base/neutral/acid compounds; PCBs = polycyclic aromatic hydrocarbons; PBDEs = polybrominated diphenyl ethers; SRM = standard reference material; LCS = laboratory control sample

Quality assurance (QA1) marine sediment chemistry acceptance criteria (Ecology, 2021) are shown in Table 11.

Some trace metal and organic analyses have empirically derived laboratory limits for various QC samples. Specific laboratory-derived acceptance limits for trace metal and organic analyses are included as Appendix C (Tables C1–C13).

QC results that exceed the acceptance limits will be evaluated to determine appropriate corrective actions. Samples will typically be reanalyzed if the unacceptable QC results indicate a systematic problem with the overall analysis. Unacceptable QC results caused by a particular sample or matrix will not require reanalysis unless an allowed method modification would improve the results.

Analytical results that do not meet QA1 acceptance criteria will be qualified and flagged according to Ecology guidance (Ecology, 2021).

Table 11. QA1 acceptance criteria for marine sediment chemistry samples.

Analyte	Method Blank	Spiked Blank	Duplicate/ Triplicate	Matrix Spike	SRM/LCS	Surrogates
Ammonia	< MDL	80–120%	RSD ≤ 20%	75 - 125%	80–120%	N/A
PSD	N/A	N/A	RSD ≤ 20%	N/A	N/A	N/A
TOC	< 0.5 QL	80 - 120%	RSD ≤ 20%	70-130%	80 - 120%	N/A
Total Solids	< MDL	N/A	RSD ≤ 20%	N/A	N/A	N/A
Total Sulfide	< 0.5 QL	80 - 120%	RSD ≤ 20%	65 - 135%	N/A	N/A
Metals (incl. Mercury)	< 0.5 QL	85–115%	RPD ≤ 20%	75 - 125%	Varies by Analyte	N/A
BNAs	< 0.5 QL	Varies by Analyte	RPD ≤ 35%	Varies by Analyte	Varies by Analyte	Varies by Analyte
Butyltins	< MDL	Varies by Analyte	RPD ≤ 35%	Varies by Analyte	Varies by Analyte	Varies by Analyte
Chl. Pesticides	< 0.5 QL	Varies by Analyte	RPD ≤ 35%	Varies by Analyte	Varies by Analyte	Varies by Analyte
PCBs	< 0.5 QL	Varies by Analyte	RPD ≤ 35%	Varies by Analyte	Varies by Analyte	Varies by Analyte
PBDEs	< 0.5 QL	50- 150%	RPD ≤ 35%	Varies by Analyte	Varies by Analyte	Varies by Analyte

< MDL - Method Blank result should be less than the method detection limit; N/A = Not Applicable; < 0.5QL – Method Blank result should be less than one-half of the QL (Lower Limit of Quantitation, LLOQ), or lower in the case of BNA analyses; PSD = particle size distribution; TOC = total organic carbon; BNAs = base/neutral/acid compounds; PCBs = polycyclic aromatic hydrocarbons; PBDEs = polybrominated diphenyl ethers; SRM = standard reference material; LCS = laboratory control sample; RPD – Relative Percent Difference; RSD – Relative Standard Deviation
QC results for matrix spike, SRM/LCS, and surrogates are in percent recovery of analyte.

8.5.1 Method Blank Evaluation

Evaluation of method blank results is based on the requirements for each reference method. Therefore, the value entered for the LIMS MDL for the method blank may differ from the LIMS MDL for the associated samples.

For metals and conventionals methods (6010D, 7471B, 9030B, and 9060A), and chlorinated pesticides/PCBs (Method 8081B (02/07)/8082A (02/07)), method blank results must be reported to one half the LLOQ. In this case, the LIMS MDL value for the method blank is one half the value reported for the LIMS RDL (LLOQ or QL).

For BNAs (Method 8270E (06/18)) the method blanks must be evaluated to at least one half the LLOQ and lower if the LIMS MDL is less than one half of the LLOQ.

For ammonia, total solids, total sulfide and PSD, the method blanks are evaluated to the same wet-weight LIMS MDL value reported for the undiluted samples.

Any method blank result that is at or above the associated LIMS MDL value will be qualified with a B flag on the QC report. Method blank values that are below the associated LLOQ will also be qualified with a J flag and a <QL qualifier to indicate they are estimated values detected below the QL.

If the associated method blank had a reportable value, any associated sample result measured at or above the LIMS MDL value will be qualified with a either a B or B3 flag based on the ratio of the sample result to the associated method blank result on a wet-weight basis according to the following scheme:

- If the associated method blank is ≥ the LIMS MDL value and the sample result is ≥ MDL but ≤ 5 times the blank result, on a wet-weight basis, a "B" flag is applied.
- If the associated method blank is ≥ the LIMS MDL value and the sample result is > 5 and ≤ 10 times the blank result, on a wet-weight basis, a "B3" flag is applied.
- If the associated method blank is ≥ the LIMS MDL value and the sample result is > 10 times the blank result, on a wet-weight basis, no flag will be applied (the level of sample contamination associated with the method blank is considered negligible relative to the sample result).

9.0 BENTHIC TAXONOMY ANALYTICAL METHODS AND QUALITY ASSURANCE/QUALITY CONTROL

Analytical methods for benthic taxonomy analysis are presented in the following subsections along with quality assurance/quality control (QA/QC) measures that will be employed to ensure data are of sufficient quality to meet the project DQOs.

9.1 Benthic Taxonomy Analytical Methods

Contract laboratory staff will be expected to use standard and accepted techniques to sort all organisms from each replicate sample (PSEP, 1987). Small fractions of sample material will be placed in a petri dish under a 10X dissecting microscope. The petri dish will be scanned systematically and all animals and associated fragments will be removed using forceps. This sorting process will be conducted at least twice to ensure the removal of all animals. These organisms will then be separated out and placed in 70% ethanol according to one of the following major taxonomic groups: Annelida, Crustacea, Mollusca, and miscellaneous taxa (Echinodermata, Nemertea, Sipuncula, etc.).

All organisms will be counted and identified to the lowest practical taxonomic level, which will usually be species. A reference collection, maintained by the contractor with verified specimen identifications, will be used to ensure consistent identifications. If animal fragments are present, only anterior portions will be counted. Identifications will be performed by regional taxonomic experts using stereo dissecting and high-power compound microscopes. Biomass measurements will also be performed in accordance with standard PSEP procedures.

9.2 Benthic Taxonomy QA/QC

The contractor for benthic taxonomic analysis will follow recommended PSEP guidelines for QA/QC (PSEP, 1987). Benthic taxonomy QC will involve:

- Independent verification by a qualified regional expert of 5% of the samples from each major taxonomic group.
- A reference collection will be created which will contain representative specimen from each identification. Synoptic re-identification will be performed on the reference collection by a taxonomist who did not make the original identification. In addition to the synoptic identification confirmation, 5% of the project samples will be recounted and identified,
- 20% of the project samples will be resorted for sort QC; the sort QC needs to meet >95% efficiency
- When the two taxonomists have identified the sample (one primary identification and one QC identification) the results can be compared for similarity. The similarity

index between the original and QC sample results must be within the acceptance limits of the contract laboratory.

10.0 DATA REPORTING AND RECORDKEEPING

This section provides information on how monitoring data will be reported and how project records will be maintained.

10.1 Sediment Chemistry Data

All sediment chemistry data will be stored in the KCEL LIMS database and information needed for a QA1 report (Ecology, 2021) provided. The final QA1-level information to be reported will include:

- a narrative discussing data quality in relation to study objectives and data criteria;
- all associated QC data (LIMS QC reports and worklists);
- copies of field sheets and COC forms; and
- a comprehensive report containing all analytical and field data (including data qualifier flags summarized in 10.1.1).

10.1.1 LIMS Data Qualifiers

The following is a comprehensive list of LIMS laboratory data qualifier flags and the typical conditions under which they are applied.

- **B** or **B3** are applied to sample results when the parameter is detected at a concentration at or above the value for the LIMS MDL in the method blank and if the ratio of the sample to blank result, on a wet-weight basis, meets these rules:
    - A "**B**" flag is applied if the associated blank is ≥ the LIMS MDL value and the sample result is ≥ MDL but ≤ 5 times the blank result.
    - A "**B3**" flag is applied if the associated blank is ≥ the LIMS MDL value and the sample result is > 5 and ≤ 10 times the blank result.
- **J** is applied to a parameter result when the reported value is an estimated value. All values less than the LIMS RDL value will receive a J qualifier for SW-846 methods.
- **JG** is applied to a parameter result when the reported value is an estimated value with a probable low bias.
- **JL** is applied to a parameter result when the reported value is an estimated value with a probable high bias.
- **H** indicates that a holding time criterion was not met prior to completion of analysis.
- **SH** indicates that a sample handling criterion was not met in some manner prior to analysis. The sample may have been compromised during the sampling procedure or may not comply with storage conditions or preservation requirements.
- **R** is applied to a parameter result that has been rejected and deemed unusable for all purposes

- **<MDL** is applied to sample data when a target analyte is not detected. This qualifier will no longer pertain to SW-846 methods. For this SAP, this qualifier will only be used for total solids, ammonia, and PSD data.
- **<RDL** is applied to sample results when a target analyte is detected at a concentration greater than or equal to the associated LIMS MDL, but less than the associated LIMS RDL value. This qualifier will no longer pertain to SW-846 methods and has been replaced by the qualifier <QL. For this SAP, this qualifier will only be used for total solids, ammonia, and PSD data.
- **<QL** replaces the <MDL and <RDL qualifiers for SW-846 methods. It is applied when a target analyte is detected at a concentration less than the associated LLOQ (LIMS RDL value) or when a parameter is not detected.

10.2 Benthic Taxonomy Data

The mean abundance and richness of each major taxon at the benthic taxonomy stations (three replicates per station) will be reported. Various diversity indices will also be calculated for each station, including Shannon-Wiener, Pielou's Evenness, and Swartz's Dominance indices.

10.3 Draft and Final Reports and EIM Files

A draft monitoring report will be prepared that will include a presentation and interpretation of the sediment chemistry and benthic taxonomy results. The report will compare sediment chemistry results to published sediment quality chemical criteria. Chemistry and benthic taxonomy data will also be compared to regional Puget Sound values in order to provide an evaluation of sediment quality within the study area. The report narrative will include summary tables of sediment chemistry and benthic taxonomy results. Complete data results and laboratory QC results will be included as report appendices. The chemistry data will also be reported in the regional Environmental Information Management System (EIMS) format for delivery to Ecology.

10.4 Recordkeeping

All field and sampling records, custody documents, raw lab data, and summaries and narratives will be archived according to KCEL policy, for a minimum of 10 years from the date samples were collected. Interpretative reports and memoranda, along with all chemistry data and benthic taxonomy data, and their respective data analysis project narratives and reports will be stored in project files for a minimum of 10 years from the date samples were collected. Appendix D includes the LIMS product names that will be used to specify the analyses to be completed.

11.0 PROJECT ORGANIZATION AND SCHEDULE

The major tasks involved for the 2022 ambient subtidal sampling event and the King County personnel who will assume responsibility for those tasks are listed below.

- **Wendy Eash-Loucks** King County Marine and Sediment Assessment Group—206-477-4683 wendy.eash-loucks@kingcounty.gov Project management, preparation of SAP, data validation and analysis, and preparation of final study report.
- **Colin Elliott** King County Environmental Laboratory—206-477-4064 n-celliott@kingcounty.gov Review of SAP, coordination of laboratory QA/QC, data validation, and data reporting.
- **Bob Kruger** King County Environmental Laboratory—206-477-7147 bob.kruger@kingcounty.gov Coordination of all field sampling activities.
- **Erin McCabe** King County Environmental Laboratory—206-477-7205 erin.mccabe@kingcounty.gov Coordination of all laboratory analytical activities, data validation, and data reporting.
- **Jay Word** EcoAnalysts—206-779-9500 jword@ecoanalysts.com Coordination of benthic taxonomy sample transfer and analysis.

Sampling for the 2022 marine ambient subtidal sediment sampling event is anticipated to require up to three days of field time (weather dependent) and will be completed in June 2022. Analytical results for sediment chemistry will be available by September 2022. Validated chemistry data packages and electronic data files will be ready for release by October 31, 2022. Benthic taxonomy data and data file(s) are expected to be available by the end of October 2022.

12.0 REFERENCES

APHA. 2017. Standard Methods for the Examination of Water and Wastewater, 23rd Edition. American Public Health Association. Washington, D.C.

ASTM. 2002. Standard Test Method for Particle-Size Analysis of Soils D422-63(2002). American Society for Testing and Materials. West Conshohocken, Pennsylvania.

Ecology. 1992. Technical Information Memorandum, Organic Carbon Normalization of Sediment Data. Prepared by Teresa Michelsen for the Washington State Department of Ecology Sediment Management Unit. Olympia, Washington.

Ecology. 2013. Sediment Management Standards, Chapter 173-204 WAC. Washington State Department of Ecology. Olympia, Washington.

Ecology. 2021. Sediment Cleanup User's Manual: Guidance for Implementing the Cleanup Provisions of the Sediment Management Standards, Chapter 173-204 WAC. Washington State Department of Ecology. Olympia, Washington.

EPA. 1988. Sediment Quality Values Refinement: 1988 Update and Evaluation of Puget Sound AET. Prepared for the U.S. Environmental Protection Agency Region 10 by PTI Environmental Services. Seattle, Washington.

EPA. 2015. Test Methods for Evaluating Solid Waste. Laboratory Manual – Physical/Chemical Methods, SW-846, 5th Edition, Update V. United State Environmental Protection Agency, Office of Solid Waste and Emergency Response. Washington, D.C.

Kerouel, R. and A. Aminot. 1997. Fluorometric determination of ammonia in sea and estuarine waters by direct segmented flow analysis. Mar. Chem. 57:265-275.

Krone, C.A., D.W. Brown, D.G. Burrows, R.G. Bogar, S.L. Chan, U., and U. Varanasi. 1989. A method for analysis of butyltin species and the measurement of butyltins in sediment and English sole livers from Puget Sound. *Mar. Environ. Res.* 27:1-18.

PSEP. 1987. Recommended Protocols for Sampling and Analyzing Subtidal Benthic Macroinvertebrate Assemblages in Puget Sound. Prepared for the Puget Sound Estuary Program (U.S. Environmental Protection Agency Region 10) by Tetra Tech, Inc. Seattle, Washington.

Appendix A: Metals and Organics Sediment Quality Criteria

Table A-1. Washington State Department of Ecology marine sediment chemical criteria (Ecology *SCUM*, 2021 Appendix A Table A-1).

	SMS Marine Sediment[a]		Marine Sediment AETs[b]	
Analyte	**SQS/SCO**	**CSL/SIZmax**	**SQS/SCO**	**CSL/SIZmax**
Metals	**mg/kg dw**		**mg/kg dw**	
Arsenic	57	93	57	93
Cadmium	5.1	6.7	5.1	6.7
Chromium	260	270	260	270
Copper	390	390	390	390
Lead	450	530	450	530
Mercury	0.41	0.59	0.41	0.59
Silver	6.1	6.1	6.1	6.1
Zinc	410	960	410	960
Organic Chemicals	**µg/kg dw (except *)**		**µg/kg dw**	
2,4-Dimethylphenol	29	29	29	29
2-Methylphenol	63	63	63	63
4-Methylphenol	670	670	670	670
Benzoic acid	650	650	650	650
Benzyl alcohol	57	73	57	73
Dibenzofuran	15*	58*	540	540
Phenol	420	1200	420	1200
N-nitrosodiphenylamine	11*	11*	28	40
Phthalates	**mg/kg OC**		**µg/kg dw**	
Bis(2-Ethylhexyl)phthalate	47	78	1300	1900
Butylbenzyl phthalate	4.9	64	63	900
Diethyl phthalate	61	110	200	>1200
Dimethyl phthalate	53	53	71	160
Di-n-butyl phthalate	220	1700	1400	1400
Di-n-octyl phthalate	58	4500	6200	6200
PCBs	**mg/kg OC**		**µg/kg dw**	
Total Aroclors	12	65	130	1000
Polycyclic Aromatic Hydrocarbons	**mg/kg OC**		**µg/kg dw**	
Total LPAH	370	780	5200	5200
Naphthalene	99	170	2100	2100
Acenaphthylene	66	66	1300	1300
Acenaphthene	16	57	500	500
Fluorene	23	79	540	540
Phenanthrene	100	480	1500	1500
Anthracene	220	1200	960	960

	SMS Marine Sediment[a]		Marine Sediment AETs[b]	
Analyte	**SQS/SCO**	**CSL/SIZmax**	**SQS/SCO**	**CSL/SIZmax**
2-Methylnaphthalene	38	64	670	670
Total HPAH	960	5300	12000	17000
Fluoranthene	160	1200	1700	2500
Pyrene	1000	1400	2600	3300
Benz[a]anthracene	110	270	1300	1600
Chrysene	110	460	1400	2800
Total benzofluoranthenes	230	450	3200	3600
Benzo[a]pyrene	99	210	1600	1600
Indeno[1,2,3-c,d]pyrene	34	88	600	690
Dibenzo[a,h]anthracene	12	33	230	230
Benzo[g,h,i]perylene	31	78	670	720
Chlorinated Organics	**mg/kg OC**		**µg/kg dw**	
1,2,4-Trichlorobenzene	0.81	1.8	31	51
1,2-Dichlorobenzene	2.3	2.3	35	50
1,4-Dichlorobenzene	3.1	9	110	110
Hexachlorobenzene	0.38	2.3	22	70
Hexachlorobutadiene	3.9	6.2	11	120
Pentachlorophenol	360**	690**	360	690

[a] Marine SMS values are dry weight normalized for metals and polar organics and normalized to total organic carbon for nonpolar organics.

[b] Dry weight normalized AETs are recommended when total organic carbon is outside the recommended range of 0.5 – 3.5% for organic carbon normalization.

* mg/kg OC

** Pentachlorophenol is in µg/kg dry weight

This page intentionally left blank.

Appendix B: Sediment Chemistry and Benthic Infauna Sampling Supplies

Table B-1. Central Basin sediment chemistry sampling supply checklist.

Check Off		Equipment	QTY.	Used for	Comments
Setup	Loaded				
		GENERAL GEAR:			
		Dunnage (plywood)		Deck protection	
		SS VanVeen grabs	1		
		Sed. table	1		
		Dust pan/Shovel		To scrape off sed. table	
		Sed. depth ruler		To measure grab penetration	
		SAPs copy/COC forms			One COC form per sampling day
		Waypoints loaded to PC			
		Field sheets/Labels			Pre-label all jars
		Field observation form			
		Space pens, clip board			
		Ice chests, ice ea. day			
		SAMPLE MIXING/SPLITTING:			
		Zinc Acetate- 2N		Plus Transfer Pipets	-
		SS bowls	1/sta		-
		SS spatulas/spoons	1/sta		-
		Grab drain tubing			
		Sed. Type key			
		Nitrile gloves			
		Aluminum foil			On *SoundGuardian*
		Brushes		for washing grabs	
		Chairs			
		Stainless Taxonomy Sieves			
		Tax tools			
		Tax Containers			
		Tax preservation material		10% Buffered Formalin	
		SAMPLE CONTAINERS*:*			
		4 oz. (125 ml) wide-mo. PP	1/sta	TOTS/TOC	3/4 full only
		4 oz. (125 ml) wide-mo. PP	1/sta	NH3	3/4 full only
		4 oz. (125 ml) wide-mo. PP	1/sta	TOTSULFIDE	No Headspace- Zn Acetate
		16 oz. (500 ml) wide-mo. PP	1/sta	PSD	Collect 2nd jar for QC every 20 samples
		8 oz. (250 ml) wide-mo. Glass	2/sta	Organics	**Less than** 3/4 full only
		4 oz. (125 ml) wide-mo. PP	1/sta	individual elements ICP + Hg	3/4 full only
		4 oz. (125 ml) wide-mo. Glass	1/sta	Extra for PCB or dioxin/furan congener	**Less than** 3/4 full only

Note: TC must debrief previous crew & brief new crew each morning.

Table B-1. Central Basin sediment benthic infauna sampling supply checklist.

Check Off		Equipment	QTY.	Used for	Comments
Setup	Loaded				
		GENERAL GEAR:			
		Dunnage (plywood)		Deck protection	
		SS VanVeen grabs	1		
		Sed. table	1		
		Dust pan/Shovel		To scrape off sed. table	
		Sed. depth ruler		To measure grab penetration	
		SAPs copy/COC forms			One COC form per sampling day
		Waypoints loaded to PC			
		Field sheets/Labels			Bring at 2+ extra blank sheets of labels in case multiple jars are needed per sample
		Field observation form			
		Space pens, pencils, clip board			Pencils needed writing on benthic taxonomy labels
		Ice chests, ice ea. day			
		SAMPLE MIXING/SPLITTING:			
		Hoses	4	For rinsing samples	-
		Brushes		For washing sieves	-
		Spoons	2	Transferring sample	Different size spoons
		Squeeze bottle	2	For rinsing samples from hand sieve	
		Large stainless taxonomy sieves			
		Small hand sieve			1 mm or smaller
		Forceps	2+		Small pointy ones work best
		Concentrated buffered formalin		To be diluted on deck	
		Large container	1	Diluting formalin with sea water	2+ gallons
		SAMPLE CONTAINERS:			
		Benthic infauna containers of various sizes	36+		Central Basin sediments tend don't have a lot of debris, but may contain large echinoderms, bring a variety of sizes.
					Double check enough containers remain on subsequent sampling days

Note: TC must debrief previous crew & brief new crew each morning.

Appendix C: Metals and Organics Performance-Based QC Limit Goals

Table C-1. Laboratory QC limits for soil metals – LCS recoveries (ERA SOIL)

Parameter	Lower Limit (%)	Upper Limit (%)
Aluminum	48	152
Arsenic	80	120
Cadmium	80	120
Chromium	80	120
Copper	80	120
Iron	63	138
Lead	80	120
Mercury	72	128
Nickel	80	120
Silver	80	120
Tin	77	123
Zinc	80	120

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-2. Laboratory QC limits for sediment metals – SRM recoveries (PACS-3)

Parameter	Lower Limit (%)	Upper Limit (%)
Aluminum	80	120
Arsenic	80	120
Cadmium	80	120
Chromium	80	120
Copper	80	120
Iron	80	120
Lead	80	120
Mercury	80	120
Nickel	80	120
Tin	80	120
Zinc	80	120

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.
* Silver is not certified in PACS-3

Table C-3. Laboratory QC limits for sediment BNAs – matrix spike recoveries.

Parameter	Lower Limit (%)	Upper Limit (%)		Parameter	Lower Limit (%)	Upper Limit (%)
1,2,4-Trichlorobenzene	22	95		Coprostanol	20	180
1,2-Dichlorobenzene	20	110		Di-N-Butyl Phthalate	64	150
1,4-Dichlorobenzene	20	105		Di-N-Octyl Phthalate	43	150
2,4-Dimethylphenol	27	126		Dibenzo(a,h)anthracene	39	150
2-Methylnaphthalene	22	109		Dibenzofuran	49	135
2-Methylphenol	21	126		Diethyl Phthalate	71	130
3-,4-Methylphenol	24	129		Dimethyl Phthalate	66	128
Acenaphthene	37	129		Fluoranthene	53	144
Acenaphthylene	44	134		Fluorene	52	150
Anthracene	37	150		Hexachlorobenzene	51	149
Benzo(a)anthracene	52	149		Hexachlorobutadiene	20	133
Benzo(a)pyrene	62	136		Indeno(1,2,3-Cd)Pyrene	41	150
Benzo(b,j,k)fluoranthene	48	135		N-Nitrosodiphenylamine	20	112
Benzo(g,h,i)perylene	27	150		Naphthalene	58	140
Benzoic Acid	20	150		Pentachlorophenol	35	134
Benzyl Alcohol	28	111		Phenanthrene	51	136
Benzyl Butyl Phthalate	27	150		Phenol	21	142
Bis(2-Ethylhexyl)Phthalate	54	150		Pyrene	59	143
Chrysene	47	141		Total 4-nonylphenol	40	160

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-4. Laboratory QC limits for sediment BNAs – blank spike recoveries.

Parameter	Lower Limit (%)	Upper Limit (%)		Parameter	Lower Limit (%)	Upper Limit (%)
1,2,4-Trichlorobenzene	39	94		Coprostanol	10	159
1,2-Dichlorobenzene	44	105		Di-N-Butyl Phthalate	71	142
1,4-Dichlorobenzene	40	103		Di-N-Octyl Phthalate	43	153
2,4-Dimethylphenol	20	121		Dibenzo(a,h)anthracene	41	150
2-Methylnaphthalene	20	128		Dibenzofuran	52	133
2-Methylphenol	20	123		Diethyl Phthalate	75	131
3-,4-Methylphenol	22	119		Dimethyl Phthalate	70	129
Acenaphthene	43	126		Fluoranthene	56	143
Acenaphthylene	45	132		Fluorene	57	150
Anthracene	48	149		Hexachlorobenzene	53	150
Benzo(a)anthracene	51	150		Hexachlorobutadiene	20	135
Benzo(a)pyrene	61	140		Indeno(1,2,3-Cd)Pyrene	42	150
Benzo(b,j,k)fluoranthene	45	143		N-Nitrosodiphenylamine	57	136
Benzo(g,h,i)perylene	28	150		Naphthalene	28	109
Benzoic Acid	20	92		Pentachlorophenol	25	135
Benzyl Alcohol	26	111		Phenanthrene	47	141
Benzyl Butyl Phthalate	36	150		Phenol	26	136
Bis(2-Ethylhexyl)Phthalate	61	150		Pyrene	60	144
Chrysene	45	150		Total 4-nonylphenol	40	160

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-5. Laboratory QC limits for sediment BNAs – surrogate recoveries.

Parameter	Lower Limit (%)	Upper Limit (%)
2,4,6-Tribromophenol	45	150
2-Fluorophenol	20	136
d5-Phenol	20	142
d5-Nitrobenzene	22	126
d4-2-Chlorophenol	20	127
2-Fluorobiphenyl	22	135
d14-Terphenyl	25	150

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-6. Laboratory QC limits for sediment BNAs – SRM recoveries.

Parameter	Lower Limit (%)	Upper Limit (%)
Benzo(a)anthracene	45	122
Benzo(a)pyrene	60	116
Benzo(b,j,k)fluoranthene	50	122
Benzo(g,h,i)perylene	32	133
Chrysene	68	144
Dibenzo(a,h)anthracene	51	200
Fluoranthene	51	127
Indeno(1,2,3-Cd)Pyrene	36	127
Phenanthrene	48	112
Pyrene	55	119

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-7. Laboratory QC limits for sediment chlorinated pesticides and PCBs – matrix spike recoveries.

Parameter	Lower Limit (%)	Upper Limit (%)
4,4'-DDD	64	112
4,4'-DDE	68	115
4,4'-DDT	55	123
Aldrin	52	117
Alpha-BHC	57	109
Alpha-Chlordane	66	111
Beta-BHC	64	110
Delta-BHC	63	118
Dieldrin	65	116
Endosulfan I	20	125
Endosulfan II	30	117
Endosulfan Sulfate	44	120
Endrin	65	141
Endrin Aldehyde	20	100
Gamma-BHC (Lindane)	58	115
Trans-Chlordane	56	125
Heptachlor	62	117
Heptachlor Epoxide	64	108
Methoxychlor	61	125
Aroclor 1242	51	100
Aroclor 1260	35	108

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-8. Laboratory QC limits for sediment chlorinated pesticides and PCBs – blank spike recoveries.

Parameter	Lower Limit (%)	Upper Limit (%)
4,4'-DDD	68	118
4,4'-DDE	67	111
4,4'-DDT	71	117
Aldrin	33	100
Alpha-BHC	22	100
Alpha-Chlordane	60	112
Beta-BHC	49	107
Delta-BHC	62	105
Dieldrin	64	113
Endosulfan I	30	103
Endosulfan II	43	111
Endosulfan Sulfate	56	114
Endrin	65	115
Endrin Aldehyde	22	100
Gamma-BHC (Lindane)	33	101
Trans-Chlordane	55	113
Heptachlor	34	100
Heptachlor Epoxide	52	110
Methoxychlor	66	119
Aroclor 1242	38	100
Aroclor 1260	70	109

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-9. Laboratory QC limits for sediment chlorinated pesticides and PCBs – SRM and surrogate recoveries.

Parameter	Lower Limit (%)	Upper Limit (%)
Alpha-Chlordane	69	136
Aroclor 1260	46	140
Decachlorobiphenyl	51	115
2,4,5,6-Tetrachloro-m-xylene	47	122

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-10. Laboratory QC limits for sediment tributyltin.

Tri-n-butyltin (as tributyltin)	Lower Limit (%)	Upper Limit (%)
Matrix Spike Recovery	20	150
Blank Spike Recovery	20	150
SRM & Surrogate Recoveries	20	150

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-11. Laboratory QC limits for sediment PBDEs - PBDE spike blank recoveries.

Parameter	Lower Limit (%)	Upper Limit (%)		Parameter	Lower Limit (%)	Upper Limit (%)
PBDE-17	42	122		PBDE-100	38	122
PBDE-28	30	136		PBDE-138	32	144
PBDE-47	47	139		PBDE-153	39	130
PBDE-66	50	155		PBDE-154	41	119
PBDE-71	41	128		PBDE-183	45	118
PBDE-85	40	128		PBDE-190	10	143
PBDE-99	41	136		PBDE-209	10	108

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-12. Laboratory QC limits for sediment PBDEs - PBDE matrix spike recoveries

Parameter	Lower Limit (%)	Upper Limit (%)		Parameter	Lower Limit (%)	Upper Limit (%)
PBDE-17	49	131		PBDE-100	42	134
PBDE-28	51	137		PBDE-138	40	161
PBDE-47	48	140		PBDE-153	54	120
PBDE-66	72	132		PBDE-154	44	134
PBDE-71	53	130		PBDE-183	52	125
PBDE-85	55	132		PBDE-190	40	129
PBDE-99	51	131		PBDE-209	10	111

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

Table C-13. Laboratory QC limits for sediment PBDEs – PBDE LCS recoveries

Parameter	Lower Limit (%)	Upper Limit (%)		Parameter	Lower Limit (%)	Upper Limit (%)
PBDE-17	60	200		PBDE-138	44	170
PBDE-28	48	153		PBDE-153	37	168
PBDE-47	57	186		PBDE-154	54	160
PBDE-85	40	176		PBDE-183	45	171
PBDE-99	43	179		PBDE-209	90	200
PBDE-100	53	148				

QC Limits are laboratory performance based limits and may change as recovery windows are evaluated and updated annually.

This page intentionally left blank.

Appendix D: KCEL Laboratory Information Management System (LIMS) Products

Table D-1. King County Environmental Laboratory's Laboratory Information Management System (LIMS) products and list types.

Parameter	LIMS Product
Ammonia	NH3-KCL
PSD	PSD
TOC	TOC-QL
Total Solids	TOTS
Total Sulfide	TOTSULFIDE-QL
Mercury by CVAA	HG-CVAA-M-QL
ICP Metals	NPDES ICP-QL, AL-ICP-QL, FE-ICP-QL, SN-ICP-QL
BNAs	BNASMS-NPDES-QL, BNASMS-NPDES-SIM–QL
PCBs	PCB-QL
PBDEs	PBDE-QL
Butyltins	TRIBUTYLTIN-QL
CL Pesticides	CLPEST-QL

CVAA – Cold vapor atomic absorption spectroscopy.
ICP – Inductively coupled plasma optic emission spectroscopy.