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ECOLOGY
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Addendum 3 to Quality Assurance Project Plan

Long-Term Monitoring of Persistent, Bioaccumulative, and Toxic Chemicals Using Age-Dated Lake Sediment Cores

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Addendum 3 to
Quality Assurance Project Plan

Long-Term Monitoring of Persistent, Bioaccumulative, and Toxic Chemicals Using Age-Dated Lake Sediment Cores

by Callie Mathieu and Jakub Bednarek

November 2020

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Signatures are not available on the Internet version.
EAP: Environmental Assessment Program

1.0 Table of Contents

Note: The numbered headings in this document correspond to the headings used in the original QAPP. Only relevant sections are included. This is why some numbered headings are missing, and why, for instance, the text begins at 3.0.

3.0 Background

The Washington State Department of Ecology (Ecology)'s Persistent, Bioaccumulative, and Toxics (PBT) Monitoring Program began a long-term study to assess PBT chemical trends through age-dated lake sediment cores in 2006. Ecology collects a single sediment core from three lakes each year to construct historical deposition profiles of PBTs in the environment. Sediment core samples are analyzed for a rotating PBT contaminant selected annually to fill data gaps in Washington State. New lakes are also chosen each year to achieve a broad spatial coverage of the state and to target waterbodies based on the parameters to be analyzed.

The quality assurance project plan (QAPP) for this study outlined a process of writing annual addenda to document the target PBT analyte and study locations for that sampling year (Mathieu, 2016). This addendum describes the following for 2020:

- The target analyte will be hexabromocyclododecane (HBCD).
- Sediment cores will be collected from Hicks Lake, Leland Lake, and Lake Kapowsin

HBCD was next on the rotating list of parameters for this project and was last sampled in 2013. HBCD is a brominated flame retardant listed as a PBT of concern in the Washington State PBT Rule. Ecology included HBCD on the agency PBT List but has not scheduled it for development of a CAP.

It was selected as a target analyte in 2013 in order to gather environmental data on the occurrence and temporal trends of PBT flame retardants in Washington State. Other flame retardants listed in the PBT Rule were considered, but not chosen for analysis in 2013 for the following reasons:

- Lack of laboratory capabilities (hexabromobiphenyl).
- Evidence against the chemical's stability in sediments (tetrabromobisphenol A).
- Budget constraints (short-chain chlorinated paraffins).
- Existing monitoring (polychlorinated diphenyl ethers).

Ecology had also recently detected HBCD in fish tissue from Washington State lakes and recommended including the contaminant in future monitoring studies (Johnson and Friese, 2012).

3.1 Study area and surroundings

Three waterbodies are selected each year for sediment core collection. The 2020 study locations are Hicks Lake, Lake Kapowsin, and Leland Lake (Figure 1). All three lakes are west of the Cascade mountain range. Sites are chosen close to or within known or potential sources and pathways of the target PBT contaminant, as well as far from sources where atmospheric deposition is the predominant pathway. Typically, we choose two sites near known sources and pathways of the target contaminant, and one site to represent atmospheric deposition. However, since atmospheric deposition is a major pathway for HBCD, two of the 2020 sites were chosen to reflect this as the predominant pathway: Lake Kapowsin and Leland Lake. Hicks Lake was selected to represent sediment HBCD contamination transport to a lake located in a largely residential watershed. The potential primary source in a residential area would be through construction and demolition. See Section 3.1.3 for more information on sources and pathways of HBCD.

The 2020 sampling lakes were also chosen partially based on sampling feasibility amidst the COVID-19 pandemic restrictions. Other considerations for waterbody selection are:

- Spatial distribution to achieve statewide coverage.
- Suitable access to the waterbody for the coring boat.
- Waterbodies where data from other studies are available.
- Watersheds within a range of land-use types.
- Physical features of the lake and watershed, including lake depositional patterns, maximum and mean depths, and elevation.
- Collaboration with other programs and agencies.

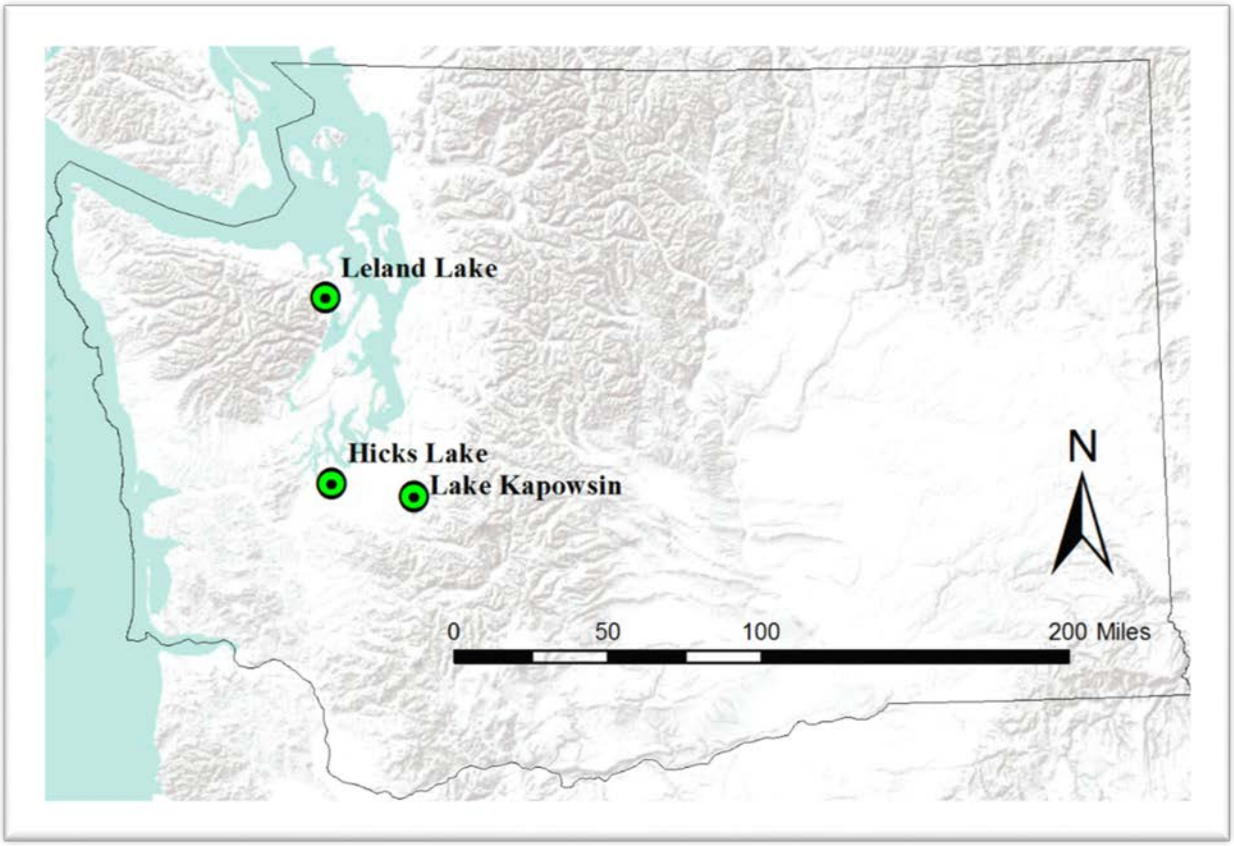

Figure 1. Map of 2020 study locations.

Hicks Lake

Hicks Lake is a 160-acre lake in the city of Lacey, Thurston County. It is the first of four lakes interconnected by Woodland creek, which eventually discharges into Henderson Inlet. The downstream lakes are Pattison Lake, Long Lake, and Lake Lois. The lake drains approximately 1.8 square miles, has 2.4 miles of shoreline and has an average depth of 18 feet. The maximum depth is 35 feet which is our target for sediment core sampling.

The primary land use of Hicks Lake is residential waterfront lots. Land use in the Hicks Lake watershed also includes undeveloped forest and commercial properties. There is a public access point on the west side of the lake via the Washington Fish and Wildlife boat launch and

Wanschers Community Park. The lake is primarily used for recreational boating, fishing, and swimming.

Lake Kapowsin

Lake Kapowsin is a 512-acre lake located in central Pierce County, 25 miles southeast of Tacoma. The lake was formed 500 years ago by mudflows from Mount Rainier that dammed off present day Kapowsin Creek. Its water sources are surface flows, primarily from Ohop Creek in the south. It is drained by Kapowsin Creek in the north (Crandell, 1963).

Land ownership along the shore lands is 50% privately held by a timber company, tribes, municipal government, and others. The remaining 50% is owned by Washington Department of Natural Resources and is managed as an aquatic reserve.

The land use surrounding Lake Kapowsin is primarily regrowth of coniferous forest land. Upland uses include a commercial rock quarry and timber harvest operation, and residential land. The lake is a hotspot for fishing and hunting. Non-motorized boating is favored among recreational users. The long term goals of the Lake Kapowsin aquatic preserve are habitat restoration and stewardship (Davenport et al., 2016).

Leland Lake

Leland Lake is a 107-acre lake in eastern Jefferson County on the Olympic Peninsula. The lake lies along Leland Creek in the Little Quilcene River watershed and eventually drains to Quilcene Bay in Hood Canal. About 90% of the lake's shore lands are undeveloped residential lots. The north east corner lot is owned by Washington Department of Fish and Wildlife and provides recreational access to anglers and users of the county campground. The lake serves public recreational angling, boating, paddling, and swimming.

Table 1 displays physical characteristics of the three study locations. As seen in Table 1, the three lakes cover a range of lake and watershed areas, calculated as a ratio in the WA:LA column.

Table 1. 2020 Sediment Core Study Lakes.

Waterbody	County	Elevation (ft)	Max. Depth (ft)	Mean Depth (ft)	Lake Area (ac)	Watershed Area (ac)	WA:LA
Hicks Lake	Thurston	162'	35'	18'	160	1152	7
Lake Kapowsin	Pierce	600'	29'	14'	590	15616	26
Leland Lake	Jefferson	190'	20'	13'	110	3654	33

WA:LA = watershed area to lake area ratio

3.1.1 Logistical problems

Field collections in 2020 may encounter logistical problems based on COVID-19 restrictions. Field crews must follow Ecology respiration matrix protocols and determine whether travel is allowed to the study location. As of the time of writing this QAPP, Washington State is under various restrictions by counties, called "phases." Ecology has imposed field work restrictions, such as no overnight travel, based on what phase the county the field work to be conducted is in.

At the time of writing all sites were within counties with sufficiently high COVID-19 phases as determined by the state for same-day travel. If any of the sites become unsuitable for sampling, due to COVID-19 restrictions or any other reason, a back-up study location will be sampled instead. Alternate locations for 2020 include Spanaway Lake in Pierce County, which is a sediment core target location for another 2020 Ecology study (Hobbs, in prep.) and Lake Wenatchee in Chelan County.

3.1.2 History of study area

The 2020 sampling locations were selected based on previously determined criteria in the original QAPP and are described in Section 3.1. The lakes have not been sampled previously as part of this project. There are no known HBCD data/historical contamination for the 2020 study locations or their watersheds. If identified, sources of HBCD to the lakes would likely be due to atmospheric deposition and releases from residential and commercial building construction in the watershed.

3.1.3 Parameters of interest

HBCD refers to a technical mixture composed primarily of alpha, beta, and gamma diastereoisomers. Commercial mixtures were comprised of gamma- (75-89%), alpha- (10-13%), and beta- (1-12%) HBCD isomers (Covaci et al., 2006). In sediments, the diastereomeric makeup of samples is generally consistent with commercial mixture percentages (Harrad et al., 2009; Morris et al., 2004). All three diastereoisomers will be analyzed and reported individually for this study.

HBCD exhibits high aquatic toxicity and is a human health concern for reproductive, developmental, and neurological effects, based on animal studies (EPA, 2010). While HBCD has been manufactured since the 1960s, concern over the persistence, bioaccumulation, and toxicity of HBCD grew in the 2000s and it was listed as a persistent organic pollutant under the Stockholm Convention in 2013. Since then, manufacturers in the United States began to phase out their use of HBCD and as of the last few years it is no longer manufactured or imported domestically (EPA, 2019).

HBCD was used primarily as a flame retardant in extruded and expanded polystyrene (EPS and XPS) foam as thermal insulation in building construction. It was also used as a flame retardant for textiles in furniture upholstery and other products, such as latex binders, adhesives, and paints (Environment Canada, 2011). HBCD is an additive flame retardant and is not bound to material. Therefore, it has the potential to enter the environment through the use or disposal of products containing HBCD and may be present in wastewater treatment plant effluent and landfill and incinerator emissions (EPA, 2010). At construction sites, HBCD has the potential to be released to the soil, with subsequent transfer to air or runoff (Environment Canada, 2011).

HBCD is ubiquitous in the environment and has been reported in air, sediment, water, and aquatic biota samples (Covaci et al., 2006). HBCD can be transported long distances through the atmosphere and has been found in many different environmental media throughout the world (Covaci et al., 2006). Sources to the environment generally include diffuse particulate releases to soil during construction and demolition of XPS- or EPS-insulated buildings and through the use or disposal of products containing HBCD (EPA, 2010). Particulates containing HBCD are transferred to air or stormwater runoff and through wastewater treatment plant effluent and landfill emissions (EPA, 2010).

3.1.4 Results of previous studies

Past studies detected HBCD in sediment cores and fish tissues at several lakes in Washington. A 2012 study found that tissues of common carp and largescale suckers had bioaccumulated HBCD at Lake Washington, Lake Spokane, lower Yakima River, and the lower Columbia River (Johnson and Friese, 2012). The concentration range of HBCD was 103 to 1,120 ng/Kg. Only alpha-HBCD was detected in this study.

A more recent study of fish tissues by Mathieu and Wong (2016) confirmed similar results to the 2012 Johnson and Friese study. Alpha-HBCD was detected in largescale suckers at twelve Washington lakes. Concentrations ranged from 116 ng/Kg to 362 ng/Kg (median 243 ng/Kg). Only alpha-HBCDD was detected, confirming studies that show the alpha- isomer as the predominant isomer to accumulate in biota (Mathieu and Wong 2016, Law and Herzke, 2011).

In 2013, HBCD was detected in sediment core samples dated later than 1960 at Lake Cavanaugh, Kitsap Lake, and Lake Sawyer (Mathieu and McCall, 2014). Temporal patterns showed concentrations increasing since the 1960s, when production of HBCD began. The most recent sediment core samples showed concentrations that range from 8.64 ng/g total HBCD at Lake Cavanaugh to 30.0 ng/g total HBCD at Lake Sawyer. Gamma-HBCD was the dominant isomer detected.

Other parameters have been studied at the proposed study locations. Lake Kapowsin is an aquatic preserve and has a comprehensive lake management plan. Physical and biological characteristics are described in the lake management plan associated studies (Davenport et al., 2016). Lake Hicks has an integrated aquatic vegetation management plan. In association with this plan, some lake characterization studies have been conducted (Herrera, 2017). Leland Lake is monitored by Jefferson County Department of Health for toxins related to harmful algal blooms.

3.1.5 Regulatory criteria or standards

No environmental regulatory thresholds exist for HBCD in lake sediments for Washington State. This study does not collect data to determine compliance with regulatory standards or criteria.

4.0 Project Description

4.2 Project objectives

The objective specific to the 2020 sampling year is to collect a single sediment core each from Hicks Lake, Lake Kapowsin, and Leland Lake and analyze nine horizons in each core for alpha-, beta-, and gamma- HBCD isomers.

4.5 Study boundaries

At each study lake, a sediment core will be collected from a discrete sampling point in the deepest part of the lake. Figures 2 through 4 display the target sampling locations for 2020.

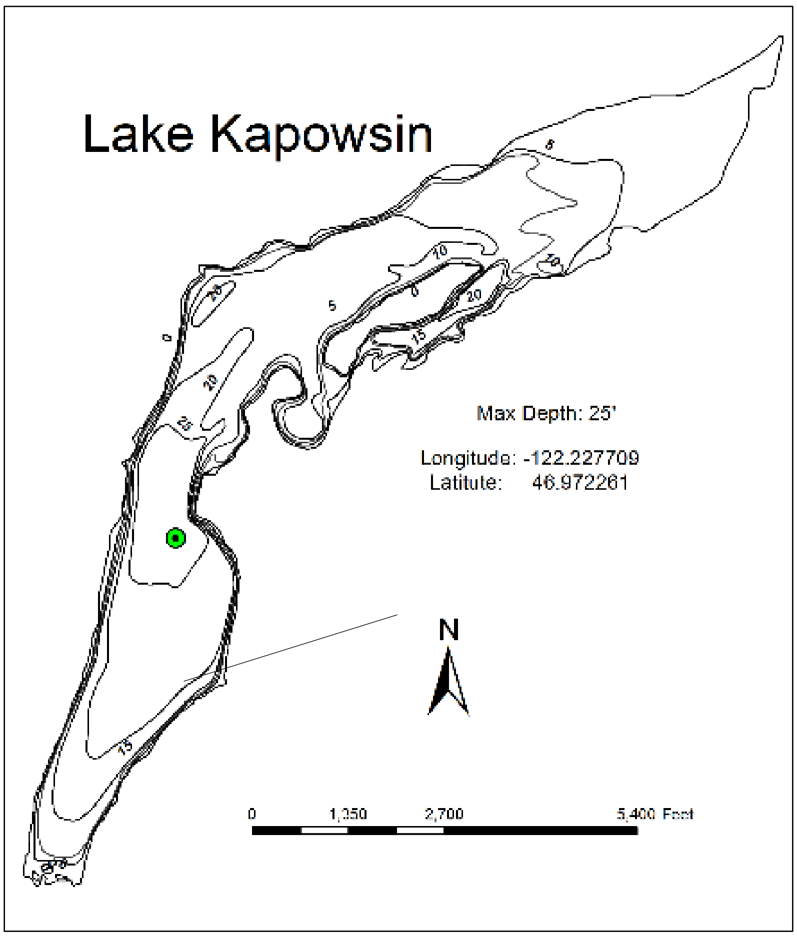

Figure 2. Bathymetric map of Lake Kapowsin with sampling point indicated by green dot.

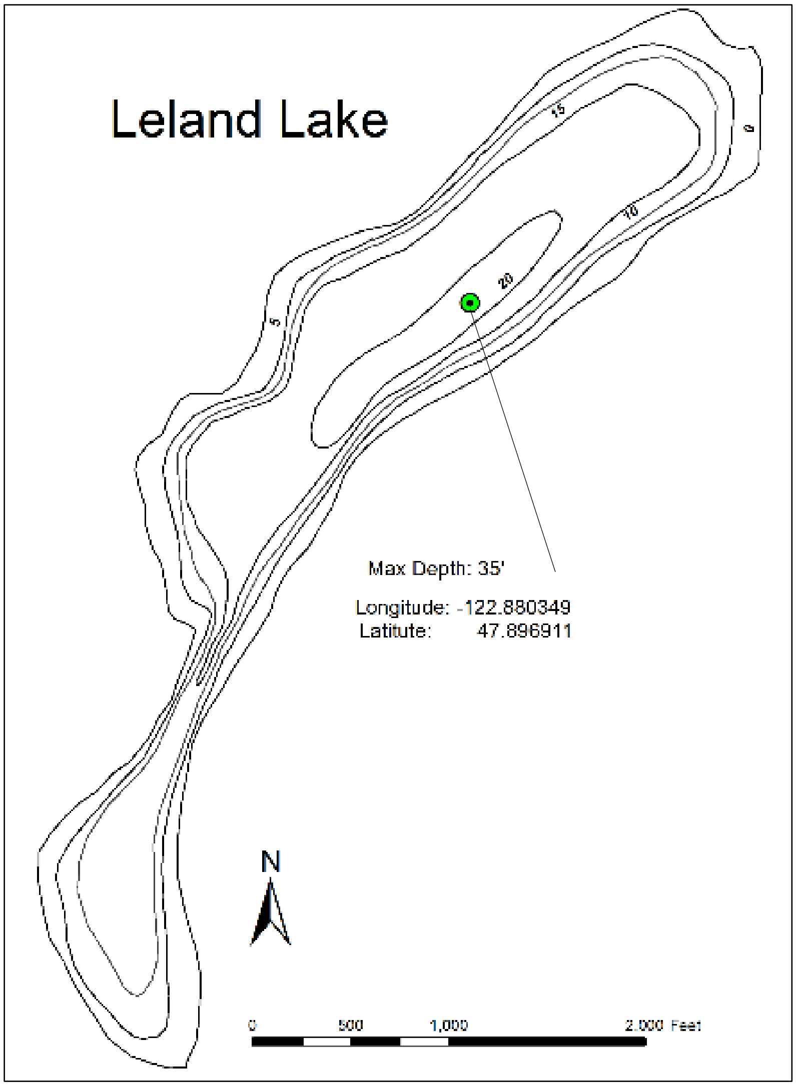

Figure 3. Bathymetric map of Leland Lake with sampling point indicated by green dot.

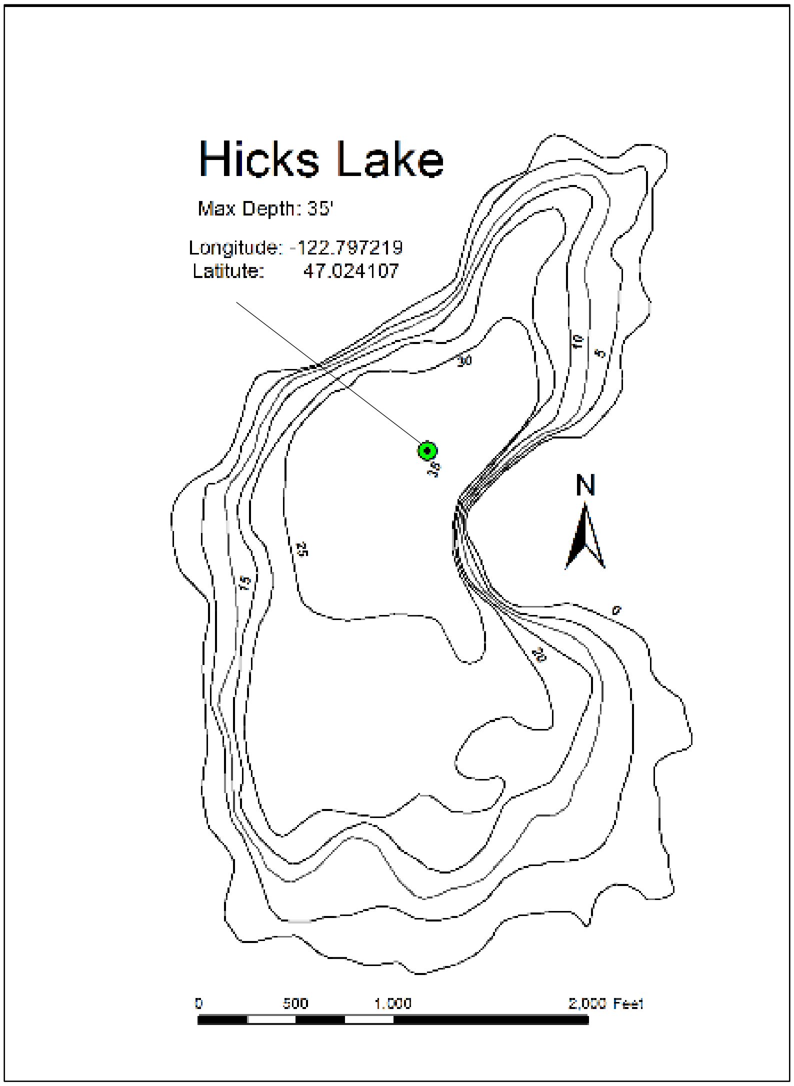

Figure 4. Bathymetric map of Hicks Lake with sampling point indicated by green dot.

WRIAs

- Hicks Lake: 13 - Deschutes
- Lake Kapowsin: 10 - Puyallup/White
- Leland Lake: 17 - Quilcene/Snow

HUC numbers

- Hicks Lake: 17110019 - Puget Sound
- Lake Kapowsin: 17110014 - Puyallup
- Leland Lake: 17110018 - Hood Canal

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Table 2. Organization of project staff and responsibilities.

EAP Staff	Title	Responsibilities
James Medlen Toxic Studies Unit SCS 360-407-6194	Client and Supervisor for the Project Manager	Clarifies scope of the project. Provides internal review of the QAPP addendum and final report. Approves the final QAPP and addendums. Manages budget and staffing needs.
Jessica Archer SCS 360-407-6698	Client and SCS Manager	Provides internal review of the QAPP addendum and final report. Approves the final QAPP addendum.
Callie Mathieu Toxic Studies Unit SCS 360-407-6965	Project Manager /Principal Investigator and co-author	Co-authors the QAPP addendum. Coordinates with MEL and contract laboratory. Oversees field collections. Conducts QA review of data, analyzes and interprets data, and writes the final report.
Jakub Bednarek Toxic Studies Unit SCS 360-407-6765	Field Lead and co-author	Co-authors QAPP addendum, leads field collections, records field information, and sends samples to the laboratory. Enters data into EIM.
Alan Rue Manchester Environmental Laboratory 360-871-8801	Director	Reviews and approves the final QAPP addendum.
Arati Kaza 360-407-6964	Ecology Quality Assurance Officer	Reviews and approves the draft QAPP addendum and the final QAPP addendum.

EAP: Environmental Assessment Program
EIM: Environmental Information Management database
QAPP: Quality Assurance Project Plan
SCS: Statewide Coordination Section

5.4 Project schedule

Table 3. Proposed schedule for completing field and laboratory work, data entry into EIM, and reports.

Field and laboratory work		
Product	Due date	Lead staff
Field work completed	11/2020	Jakub Bednarek
Laboratory analyses completed	03/2021	
Environmental Information System (EIM) database (EIM Study ID SEDCORE20)		
Product	Due date	Lead staff
EIM data loaded	08/2021	Jakub Bednarek
EIM QA	09/2021	Callie Mathieu
EIM complete	10/2021	Jakub Bednarek
Final report		
Product	Due Date	Lead Staff
Draft due to supervisor	08/2021	Callie Mathieu/Jakub Bednarek
Draft due to client/peer reviewer	09/2021	Callie Mathieu/Jakub Bednarek
Final (all reviews done) due to publications coordinator	10/2021	Callie Mathieu/Jakub Bednarek
Final report due on web	12/2021	Callie Mathieu/Jakub Bednarek

5.6 Budget and funding

Laboratory costs estimated in Table 4 will be fully funded by PBT Monitoring funds, which comes from the state toxics control account.

Table 4. Estimated laboratory budget for 2020 sampling.

Parameter	Field Samples (# of samples)	QA Samples[1] (# of samples)	Total Number of Samples	Cost per Sample	MEL Subtotal	Contract Lab Subtotal	MEL Contract Fee
HBCD	27	2	29	$650	---	$18,850	$5,655*
T-Pb	30	4	34	$50	$1,700	---	---
TOC, TN	30	2	32	$50	$1,600	---	---
LOI	30	2	32	$50	$1,600	---	---
^{210}Pb	45	3	48	$150	---	$7,200	$360**
^{226}Ra	9	1	10	$100	---	$1,000	$50**
Grain Size	3	2	5	$120	---	$600	$30**
MEL subtotal					**$4,900**	---	---
Contracting Subtotal					---	**$33,745**	
Lab Grand Total					**$38,645**		

[1]Includes only QA samples that are not free of charge with the analysis (laboratory duplicates).
*MEL contract fee of 30% for managing the contract and providing data validation.
**MEL contract fee of 5% for managing the contract only.

6.0 Quality Objectives

6.2 Measurement quality objectives

Measurement quality objectives (MQOs) for laboratory analyses are shown in Table 5.

Table 5. Measurement quality objectives.

Analyte	LCS (recovery)	Lab Duplicates (RPD)	Method blanks	Matrix Spike (recovery)	Matrix Spike Duplicates (RPD)	Surrogate Standards (recovery)
HBCD	70 - 130%	<40%	< LOQ	---	---	40 - 150%
T-Pb	85 - 115%	---	< LOQ	75 - 125%	< 20%	---
TOC, TN	---	< 20%	---	---	---	---
LOI	---	< 20%	---	---	---	---
^{210}Pb	80 - 120%	< 30%	< LOQ	---	---	---
^{226}Ra	80 - 120%	< 30%	< LOQ			
Grain Size	---	< 25%	---	---	---	---

LCS = laboratory control samples. RPD = relative percent difference. TOC = total organic carbon. TN = total nitrogen. LOI = loss on ignition.

7.0 Study Design

7.1 Study boundaries

7.1.3 Parameters to be determined

In 2020, sediment core samples will be analyzed for three HBCD isomers: alpha, beta, and gamma, as well as the parameters listed in Table 7 for age-dating and interpretation. The HBCD isomers will be analyzed in 9 sediment horizons per core, and horizons will be selected as outlined in the original QAPP. In addition to HBCD, loss on ignition (LOI) and ^{226}Ra will be analyzed in 2020 to help interpret the sediment core profile. Loss on ignition will be analyzed in the same 10 intervals per core as selected for total lead and total organic carbon/total nitrogen (TOC/TN). Three intervals per core will be analyzed for ^{226}Ra to confirm background 210Pb concentrations.

8.0 Field Procedures

8.2 Containers, preservation methods, holding times

Sample containers, minimum sample sizes, preservation methods, and sample holding-time requirements are shown below in Table 6.

Table 6. Containers, sample size, preservation methods, and holding times.

Parameter	Minimum Quantity Required	Container	Field preservation	Preservation after processing	Holding Time
HBCD	100 g ww	4 oz amber glass jar	cool to 4° C	Freeze, -18° C	1 year frozen; 28 days after extraction[1]
T-Pb	25 g ww	4 oz glass jar	cool to 4° C	Freeze, -18° C	1 year frozen
TOC, TN	20 g ww	4 oz glass jar	cool to 4° C	Freeze, -18° C	6 months
LOI	25 g ww	4 oz glass jar	cool to 4° C	Freeze, -18° C	6 months
^{210}Pb	20 g ww	2 oz glass jar	cool to 4° C	none required	n/a
^{226}Ra	360 g ww	8 oz glass jar	cool to 4° C	none required	n/a
Grain Size	150 g ww	8 oz HDPE jar	cool to 4° C	cool to 4° C	6 months

ww = wet weight
[1]Standard method holding times have not been established for HBCD in sediment.

9.0 Laboratory Procedures

9.2 Lab procedures table

Ecology's Manchester Environmental Laboratory (MEL) will conduct all analyses outlined in Table 7 except for HBCD, ^{210}Pb, ^{226}Ra, and grain size, which will be conducted by contract laboratories.

An external laboratory will conduct the HBCD analysis under a contract managed by MEL. The contract laboratory will analyze HBCD using liquid chromatography – tandem mass spectrometry (LC-MS/MS) with isotopic dilution. The contract laboratory will report all three diastereomers separately (alpha, beta, and gamma) and use labeled surrogates for each measured isomer. The extraction and analysis method is laboratory-specific and will be described in the 2020 sampling results report.

Table 7 describes the number of samples, expected range of results, and the laboratory methods.

Table 7. Laboratory procedures.

Parameter	Number of samples	Arrival date	Expected range of results	Reporting limit	Sample Prep method	Analytical method
HBCD	27	11/4/2020	< 0.25 - 20 ng/g	0.25 ng/g	Soxhlet with DCM	LC-MS/MS isotopic dilution
T-Pb	30	11/4/2020	0.1 - 1,000 mg/Kg	0.1 mg/Kg dw	EPA 6020B	ICP-MS
TOC, TN	30	11/4/2020	0.1 – 20% of dw	0.1% of dw	SM5310B (preacidified)	EPA-440
LOI	30	11/4/2020	1.0-20 mg	1.0 mg	SM2540G	ASTM D7348-13
^{210}Pb	45	11/4/2020	< 0.45 - 30 pCi/g	0.45 pCi/g	Alpha spectroscopy	Alpha spectroscopy
^{226}Ra	9	11/4/2020	<0.5 - 2.0 pCi/g	0.5 - 1.0 pCi/g	Gamma spectroscopy	Gamma spectroscopy
Grain Size	3	11/4/2020	---	0.001	PSEP-EPA, 1986	Sieve-pipette

EPA = Environmental Protection Agency; dw = dry weight; LC-MS/MS = liquid chromatography tandem mass spectrometry; ICP-MS = inductively coupled plasma-mass spectrometry; PSEP = Puget Sound Estuary Program; DCM = dichloromethane.

9.3 Sample preparation method

Because there are no standard EPA methods for HBCD in sediments, the laboratories bidding on the contract will need to describe their sample preparation method and past performance with the method. The contract laboratory's sample preparation method should include Soxhlet extraction using dichloromethane.

9.5 Lab(s) accredited for method(s)

Washington State does not currently provide accreditation for HBCD analysis in sediments. Therefore, a waiver will be obtained for this analysis. The contract laboratory will be required to demonstrate that they have successfully performed this analysis in the past and provide client references for the requested analysis.

10.0 Quality Control Procedures

10.1 Table of field and lab quality control required

MEL and contract laboratories will perform the quality control (QC) tests presented in Table 8.

Table 8. Laboratory quality control samples, types, and frequency.

Parameter	LCS	Method blanks	Matrix spikes	Matrix spike duplicates	Laboratory duplicates	Surrogates
HBCD	1/batch	1/batch	---	---	1/batch	each sample
T-Pb	1/batch	1/batch	1/batch	1/batch	---	---
TOC, TN	1/batch	1/batch	---	---	1/batch	---
LOI	1/batch	1/batch	---	---	1/batch	---
^{210}Pb	1/batch	1/batch	---	---	1/batch	---
^{226}Ra	1/batch	1/batch	---	---	1/batch	---
Grain Size	1/batch	1/batch	---	---	1/batch	---

LCS = laboratory control sample. One batch equals 20 samples or fewer.

13.0 Data Verification

13.2 Lab data verification

Laboratory data will be verified for errors, omissions, and compliance with method acceptance criteria according to the original QAPP for this project for all parameters except HBCD. Data verification for HBCD will be conducted along with an EPA Stage 4 data validation described in section 13.3, below.

13.3 Validation requirements, if necessary

MEL will be responsible for carrying out validation of the HBCD data. The HBCD data will undergo an EPA Stage 4 data validation as defined in EPA (2009). If MEL is unable to perform the data validation with current staff, a contract vendor with the appropriate qualification will be selected to complete it. MEL or the contract vendor will provide a case narrative summarizing the findings of the data validation, along with an electronic data deliverable (EDD) with the final result and final result qualifier as provided by the data validator.

14.0 Data Quality (Usability) Assessment

14.2 Data analysis and presentation methods

A summary of the data will be presented in the final report. Contaminant results will be presented as both concentrations and fluxes. Fluxes will be calculated as the contaminant concentration multiplied by the sediment accumulation rate for the sediment core interval ($g/cm^2/yr$). The final report summarizing sediment core results will present the separately reported diastereomers, as well as total HBCD values.

14.3 Treatment of non-detects

Methods for calculating T-HBCD will follow the same logic presented in the original QAPP outlined for polychlorinated biphenyls.

Blank censoring of the HBCD analytical data will follow a "five-times rule." Results will be considered a non-detect if the concentration in the native sample is less than five times the concentration of a positively identified HBCD compound in the associated laboratory method blank.

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