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Addendum 5 to Quality Assurance Project Plan

Depositional History of Mercury in Selected Washington Lakes Determined from Sediment Cores

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Publication Information

Addendum

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This addendum is an addition to an original Quality Assurance Project Plan. It is not a correction (errata) to the original plan.

Data for this project will be available on Ecology's Environmental Information Management (EIM) website at www.ecy.wa.gov/eim/index.htm. Search Study ID: SEDCORE15.

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Author and Contact Information

Callie Mathieu
Environmental Assessment Program
Washington State Department of Ecology
Olympia, Washington 98504-7710

For more information contact: Communications Consultant, phone 360-407-6834.

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June 2015

Approved by:

Signature:	Date: June 2015
Holly Davies, Client, Waste 2 Resources Program	
Signature:	Date: June 2015
Carol Kraege, Client's Unit Supervisor, Waste 2 Resources Program	
Signature:	Date: June 2015
John Roland, Client, Toxics Cleanup Program, Eastern Regional Office	
Signature:	Date: June 2015
Callie Mathieu, Lead Author / Project Manager/Principal Investigator, EAP	
Signature:	Date: June 2015
Dale Norton, Author's Unit Supervisor, EAP	
Signature:	Date: June 2015
Will Kendra, Author's Section Manager, EAP	
Signature:	Date: June 2015
Joel Bird, Director, Manchester Environmental Laboratory	
Signature:	Date: June 2015
Bill Kammin, Ecology Quality Assurance Officer	

Signatures are not available on the Internet version.
EAP: Environmental Assessment Program
EIM: Environmental Information Management database

3.0 Background

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. A single sediment core is collected from three lakes per year to construct historical deposition profiles of PBTs in the environment. New lakes are chosen each year to achieve a broad spatial coverage of the state and to target waterbodies based on the parameters to be analyzed. Ecology selects lakes in an attempt to cover a range of potential contaminant sources.

A previous Quality Assurance Project Plan (QAPP) Addendum (Mathieu, 2012) for this study outlined a schedule to rotate target PBT chemicals into the analyte list to provide depositional and temporal data on a wider range of PBTs. This information helps policy makers prioritize PBTs for development of chemical action plans (CAPs) and provides data to evaluate existing CAP reduction strategies.

This addendum describes the 2015 sampling locations and the following changes in target analytes:

- Brominated flame retardants will be the focus of the target analyte list in 2015.
- Chlorinated paraffins will be taken off the target analyte list in 2015.

Sections not included in this addendum remain unchanged from the original QAPP (Coots, 2006).

3.1 Study area and surroundings

Lakes selected for 2015 sampling are described in Table 1 and displayed in Figure 1. Field staff will collect sediment cores from the following lakes in 2015: Lake Meridian, Pierre Lake, and Lake Whatcom.

Lake Meridian lies within the city limits of Kent. The small watershed is highly developed with primarily residential properties, and the lake shoreline is densely populated. Lake Meridian receives an average of 45" of precipitation a year. Inflow is intermittent, coming in at the northwest end. Water flows out at the easternmost point of the lake through a stream channel that drains to Soos Creek. Basin geology consists of predominantly fine-grained Vashon till deposits.

Pierre Lake is located 20 miles north of Kettle Falls in the Kettle River basin. The lake has a large watershed relative to the lake surface area, comprising undeveloped public and private forestland, along with a small percentage of agricultural land. The area receives an annual average of 26" of precipitation. Pierre Creek feeds into the lake from the northeast, and water flows out of the lake to the south via Toulou Creek. The valley geology is made up of glacial drift, with silt and sandy loam soils (Bortleson et al., 1976).

The northwest end of Lake Whatcom is located within the city of Bellingham. Lake Whatcom is a large natural lake made up of three distinct basins (referred to as Basin 1, 2, and 3). Land use in the watershed for the entire lake comprises primarily forestland, with smaller portions of residential and shrub/grassland areas. Commercial, industrial, and agricultural areas make up 1% of the watershed (Cadmus and CDM, 2007) and are concentrated in the northwest region surrounding Basin 1. Annual precipitation to the lake is 44". The lake is fed through many perennial streams, including Anderson Creek which receives diverted flow (via Mirror Lake) from the Middle Fork Nooksack River during sufficient flow periods. Vashon recessional outwash deposits (predominantly coarse-grained) and bedrock deposits make up the geology of the Lake Whatcom area.

Table 1. 2015 Sediment Core Study Lakes.

Waterbody	County	Elevation (ft)	Max Depth (ft)	Mean Depth (ft)	Lake Area (ac)	Watershed Area (ac)	WA:LA
Lake Meridian	King	370	90	41	150	742	5
Pierre Lake	Stevens	2,005	75	28	110	17,152	156
Lake Whatcom*	Whatcom	315	330	150	5,000	35,776	7

*Includes the entire lake (Basins 1, 2, and 3). Max depth of Basin 1 = 95'.
WA:LA = watershed area to lake area ratio

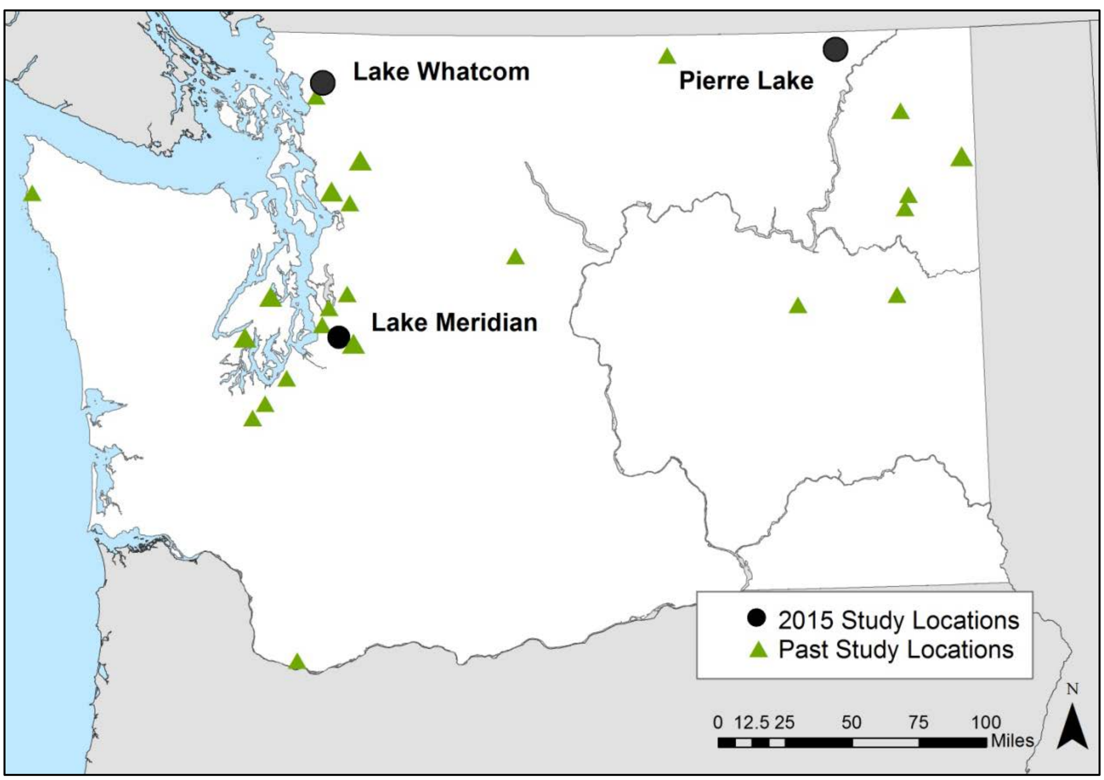

Figure 1. 2015 Sediment Core Study Locations.

3.1.1 Logistical problems

No logistical problems were found during reconnaissance of the study lakes.

3.1.2 History of study area

The 2015 sampling locations were chosen based on criteria outlined in the QAPP, such as to achieve broad spatial coverage of the state and cover a range of land use and contamination potential. Lake Meridian was chosen to help characterize brominated flame retardant contamination in an urban/residential lake with a highly developed watershed. Lake Whatcom was selected as mixed land-use lake, where watershed land types are a combination of forested and residential. Pierre Lake was chosen to represent an undeveloped lake with the primary source of brominated flame retardants being atmospheric deposition.

The three lakes cover a range of physical and topographic characteristics likely to affect contaminant deposition profiles, such as watershed area to lake surface area ratios and elevation. In addition, historical brominated flame retardant data is available for fish tissue collected at all three of the study lakes. This information is briefly described in Section 3.1.4.

3.1.3 Parameters of Interest

Brominated flame retardants are a broad class of chemicals used in consumer products, such as furniture and electronics, to prevent or slow the spread of fire. Additive flame retardants are not chemically bound to the material in the product and leach out of products over time, accumulating in indoor dust. Ecology developed a CAP for polybrominated diphenyl ether (PBDE) flame retardants in 2006 after growing concern that the chemicals were dramatically increasing in people and in the environment (Ecology et al., 2006). Chemical manufacturers voluntarily stopped production of two commercial formulations of PBDEs (penta- and octa-) in the mid-2000s, and phased out most uses of deca-BDE in 2012.

PBDEs are listed on Ecology's PBT List as persistent, bioaccumulative, and toxic chemicals. Due to their persistent nature, PBDEs are found in air, water, soil, sediments, and biota throughout the world (Covaci et al., 2011). PBDEs increase in concentration up trophic systems, and are ubiquitous in humans in the United States (Sjodin et al., 2008). Animal studies show prenatal exposure to PBDEs can impact neurodevelopment, affecting behavior and learning after birth into adulthood, as well as affecting the thyroid and liver (Ecology et al., 2006).

As commercial uses of PBDEs were phased out, manufacturers started using alternative flame retardants as replacements to meet flammability standards. Many of the replacement chemicals for PBDEs are also brominated, and little is known about their toxicity and fate in the environment. Modeling studies suggest that some of the alternative brominated flame retardants have similar hazard profiles to PBDEs and may persist in the environment (EPA, 2014a; Kuramochi et al., 2014). In addition to PBDEs, this study will analyze the following alternative brominated flame retardants: pentabromoethylbenzene (PBEB), hexabromobenzene (HBBz), 1,2-Bis(2,4,6-tribromophenoxy)ethane (BTBPE), and decabromodiphenylethane (DBDPE) (Table 2).

PBEB was produced in the United States until 1986 and in France until 2002 (Vorkamp and Riget, 2014). PBEB is currently listed as a low production volume chemical in the European Union, but is defined as not currently produced on the OSPAR List (Oslo Paris Convention) (de Wit et al., 2011). HBBz has been used since the 1970s (Arp et al., 2011) and is still produced in Japan and China (Vorkamp and Riget, 2014). BTBPE and DBDPE are currently used as replacements for PBDEs (octa and deca formulations, respectively) (Vorkamp and Riget, 2014). No information is available on current production volumes of these alternative brominated flame retardants.

Table 2. Target Brominated Flame Retardant Analyte List for 2015 Sediment Cores.

Parameter Suite	Chemicals Analyzed	Acronym
Brominated Flame Retardants (BFRs)	polybrominated diphenyl ethers*	PBDEs
	pentabromoethylbenzene	PBEB
	hexabromobenzene	HBBz
	1,2-Bis(2,4,6-tribromophenoxy)ethane	BTBPE
	decabromodiphenylethane	DBDPE

*congeners: '-7, -8/11, -10, -12/13, -15, -17/25, -28/33, -30, -32, -35, -37, -47, -49, -51, -66, -71, -75, -77, -79, -85, -99, -100, -105, -116, -119/120, -126, -128, -138/166, -140, -153, -154, -155, -181, -183, -190, -203, -206, -207, -208, -209

Ecology's Toxic Cleanup Program (TCP) Eastern Regional Office (ERO) has requested analysis of additional metals in the Pierre Lake core to aid in evaluating trends related to historic metallurgical smelter emissions. The assessment of area-wide deposition at various upland lakes situated along the upper Columbia River Valley is an ongoing priority for Ecology, as reported previously (Johnson et al., 2011a and Johnson et al., 2013). Arsenic, cadmium, copper, chromium, antimony, titanium, and zinc will be analyzed in the Pierre Lake core with funding from TCP ERO.

3.1.4 Results of previous studies

Sediment cores collected from high elevation lakes in two Washington State national parks were analyzed for PBDEs by Landers et al. (2008). PBDEs were generally below detection limits in the four lakes sampled, with the exception of Golden Lake in Mt. Rainier National Park. Focus-corrected fluxes of total detected PBDEs ranged from 18 - 4490 ng/m^2/yr among the four lakes. No monotonic patterns were apparent for PBDE concentrations or fluxes in any of the cores.

No Ecology studies have measured PBDEs or alternative brominated flame retardants in freshwater sediment cores in Washington State. However, previous Ecology studies have analyzed PBDEs in fish tissue and surface sediments from the 2015 study lakes. Table 3 displays total PBDE concentrations reported by previous Ecology studies at Lake Meridian, Pierre Lake, and Lake Whatcom.

Fish were collected from Pierre and Whatcom Lakes in the fall of 2014 for analysis of PBDEs, PBEB, HBBz, DBDPE, and BTBPE (Mathieu et al., 2014). These results are expected to be published in fall 2015.

Table 3. Total PBDEs (ng/kg) Reported in Fish Tissue and Sediment by Previous Studies.

Waterbody	Collection year	Matrix	Sample type	T-PBDEs (ng/kg)	Reference
Meridian	2006	fish tissue	kokanee	33,600 J	1
	2006		largemouth bass	19,130 J	1
Pierre	2009	fish tissue	smallmouth bass	659	2
	2010		largemouth bass	873	3
	2010	sediment	0-10 cm	425	4
Whatcom	2001	fish tissue	cutthroat trout	2,480	5
	2005		brown bullhead	1,241	6
	2005		peamouth	1,910	6
	2005		cutthroat trout	13,280	6
	2005		yellow perch	170	6
	2005		smallmouth bass	5,390	6

[1]Seiders et al., 2008
[2]EIM data accessed 4/2/15, Study ID = WSTMP09
[3]Johnson et al., 2011a
[4]Johnson et al., 2011b
[5]Seiders, 2003
[6]Johnson et al., 2006

Ecology collected a sediment core from each of Lake Whatcom's three basins in 2002 for analysis of mercury (Norton, 2004). Sediment core profiles from Lake Whatcom showed increasing levels of mercury beginning around 1900 and consistently rising until the peak in the mid-1990s. Mercury sources appeared to decline through the top of the cores (early 2000s). The study was part of a cooperative effort with USGS to evaluate sources of mercury in Whatcom County (Paulson, 2004). Figure 2 displays the profile of mercury concentrations in the sediment core collected from Basin 1 (Norton, 2004).

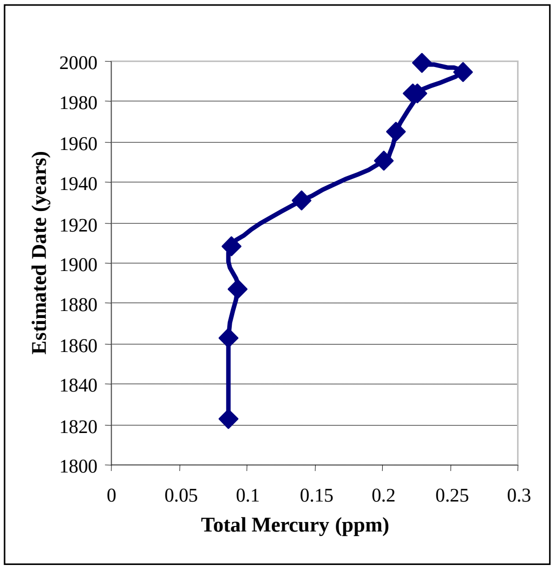

Figure 2. Mercury Concentration Profile (ppm) in Lake Whatcom Basin 1 Sediment Core (from Norton, 2004).

3.1.5 Regulatory criteria or standards

No regulatory criteria or standards exist in Washington State for brominated flame retardants in freshwater sediment.

4.0 Project Description

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. Figure 3 displays target sampling locations. Although Basin 1 in Lake Whatcom is the shallowest of the three basins, the deepest part of Basin 1 was selected for sampling to capture sources of brominated flame retardants coming from the mixed land use of the watershed. Land surrounding Basin 1 is more urbanized and developed compared to Basins 2 and 3. Water exits the lake through Basin 1.

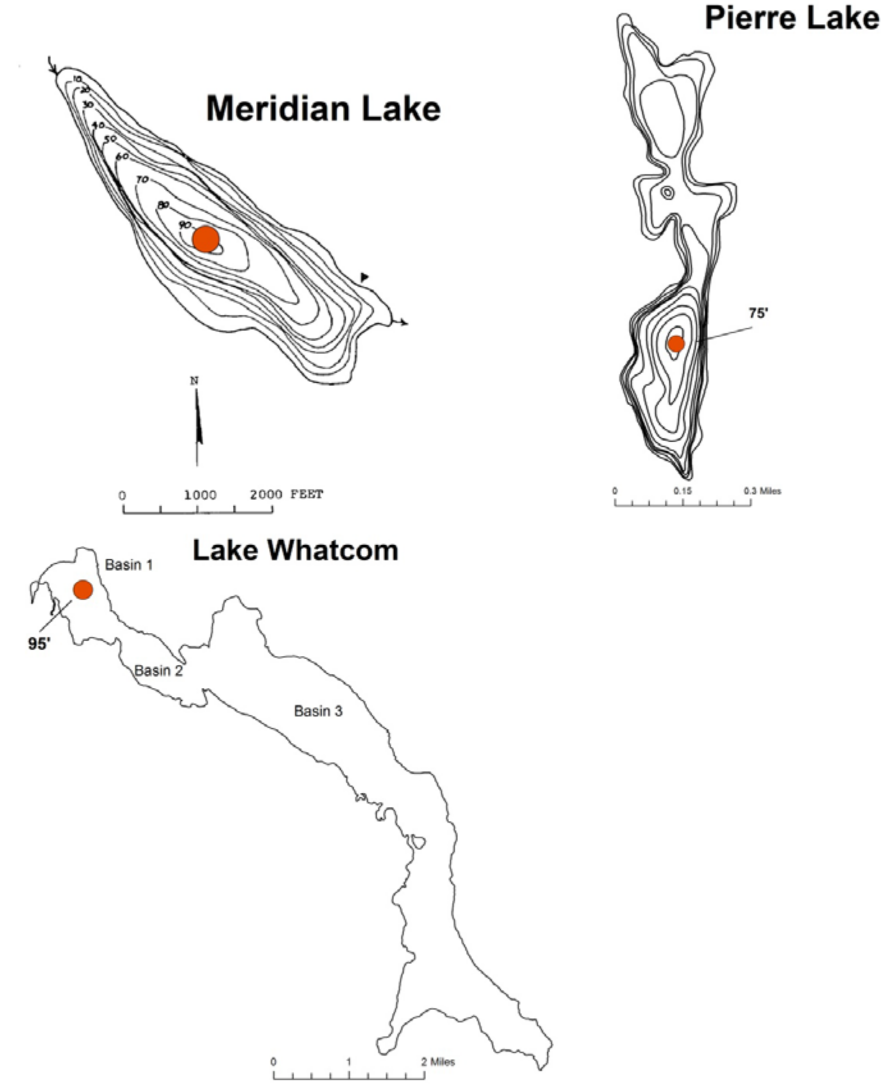

Figure 3. Target Sampling Locations for 2015 Sediment Core Collection (orange circles).
Meridian Lake bathymetry from: Ecology, 1976.

WRIAs

- Lake Meridian: 9
- Pierre Lake: 60
- Lake Whatcom: 1

HUC numbers

- Lake Meridian: 17110013
- Pierre Lake: 17020002
- Lake Whatcom: 17110004

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Organization of Project Staff and Responsibilities is documented in the QAPP for 2014 sampling (Mathieu, 2014). No changes have been made to staff or responsibilities.

5.6 Budget and funding

Table 4 presents the laboratory budget for the 2015 sediment core samples.

Table 4. 2015 Laboratory Budget.

Parameter	Field Samples (# of samples)	QA Samples* (# of samples)	Total Number of Samples	Cost per Sample	MEL Subtotal	Contract Lab Subtotal	MEL Contract Fee
Pb	20	4	24	$50	$1,200	---	---
Hg	20	4	24	$52	$1,248	---	---
Pb, Hg, As, Cd, Cu, Cr, Sb, Ti, Zn*	10	2	12	$220	$2,640		
TOC	30	2	32	$46	$1,472	---	---
^{210}Pb	45	3	48	$120	---	$5,760	$1,440
Grain Size	3	2	5	$100	---	$500	$125
BFRs	27	0	27	$845	---	$22,815	$5,704
MEL subtotal					**$6,560**	**---**	**---**
Contracting Subtotal					**---**	**$36,344**	
Lab Grand Total					**$42,904**		

* includes only QA samples that are not free of charge with the analysis.
^additional metals only analyzed in Pierre Lake core. ERO TCP will provide funding for additional metals analyses.

6.0 Quality Objectives

6.2 Measurement Quality Objectives

Measurement Quality Objectives (MQOs) for the analysis of brominated flame retardants and additional metals are described in Table 5. MQOs for lead and mercury are unchanged.

Table 5. Measurement Quality Objectives.

Analyte	LCS (recovery)	Lab Duplicates (RPD)	Method Blanks	Matrix Spike (recovery)	Matrix Spike Duplicates (% recov.)	Surrogate Standards (% recov.)	Lowest Concentration of Interest
BFRs	70 - 130%	<40%	< LOQ	NA	NA	50 - 150%	0.01 - 200 pg/g*
As	85 - 115%	<20%	< LOQ	75-125%	<20%	NA	0.1 mg/kg
Cd	85 - 115%	<20%	< LOQ	75-125%	<20%	NA	0.1 mg/kg
Cu	85 - 115%	<20%	< LOQ	75-125%	<20%	NA	0.1 mg/kg
Cr	85 - 115%	<20%	< LOQ	75-125%	<20%	NA	0.1 mg/kg
Sb	85 - 115%	<20%	< LOQ	75-125%	<20%	NA	0.2 mg/kg
Ti	86 - 115%	<20%	< LOQ	75-125%	<20%	NA	0.1 mg/kg
Zn	85 - 115%	<20%	< LOQ	75-125%	<20%	NA	5 mg/kg

*The lowest concentration of interest is equal to the reporting limit for individual compounds (see Table 7 for individual reporting limits).
BFRs = brominated flame retardants. LCS = laboratory control samples. RPD = relative percent difference

8.0 Sampling Procedures

8.2 Containers, preservation methods, holding times

Table 6 provides information on sample containers, preservation techniques, and holding times for brominated flame retardants. Additional metals analyzed in the Pierre Lake core will be taken from the same sample already analyzed for lead and mercury.

Table 6. Sample Containers, Preservation, and Holding Times.

Parameter	Matrix	Container	Sediment needed	Field preservation	Sample Holding Time*	Holding Time from Extraction*
BFRs	sediment	4 oz. glass	10-15 g dw	cool to < 4° C	1 year (stored at < -10° C)	1 year (stored at < -10° C)

*Maximum holding times have not been established for this method.

8.3 Invasive Species

All study locations are in areas of moderate concern for invasive species. Boat and sampling gear will be inspected and cleaned following Ecology's SOP EAP070, *Procedures to Minimize the Spread of Invasive Species Version 2.0* (Parsons et al., 2012).

8.4 Equipment Decontamination

Field staff will follow Ecology's SOP EAP090, *Decontaminating Field Equipment for Sampling Toxics in the Environment* (Friese, 2014), to clean the sampling equipment prior to field collection. Acrylic liners and subsectioning equipment will be scrubbed with Liquinox and hot tap water, followed by sequential rinses with 10% nitric acid, deionized water, acetone, and hexane. Equipment will be dried in a hood, and then wrapped in aluminum foil for transport to the field location.

9.0 Measurement Methods

9.2 Lab procedures table

The lab procedures for brominated flame retardants and additional metals are outlined in Table 7.

Table 7. Lab Procedures.

Analyte	Number of Samples	Expected Range of Results	Reporting Limit	Analytical Method	Method Description
PBDEs	27 (to be sent to lab on 07/01/15)	< 0.1 - 10,000 pg/g	0.1 pg/g	EPA 1614A	HRMS; isotopic dilution
PBEB		< 0.01 - 2,000 pg/g	0.01 pg/g		
HBB		< 0.1 - 2,000 pg/g	0.1 pg/g		
BTBPE		< 30 - 2,000 pg/g	30 pg/g		
DBDPE		< 200 - 2,000 pg/g	200 pg/g		
As	10 (to be sent to lab on 07/01/15)	<0.1 - 10 mg/kg	0.1 mg/kg	EPA 6020	ICP-MS
Cd		<0.1 - 1 mg/kg	0.1 mg/kg		
Cu		<0.1 - 50 mg/kg	0.1 mg/kg		
Cr		<'0.1 - 50 mg/kg	0.1 mg/kg		
Sb		<0.2 - 0.5 mg/kg	0.2 mg/kg		
Ti		<0.1 - 2000 mg/kg	0.1 mg/kg		
Zn		<5 - 100 mg/kg	5 mg/kg		

9.3 Sample preparation method(s)

Brominated flame retardant samples will be extracted in a Soxhlet/Dean-Stark extractor prior to analysis, as per EPA Method 1614.

9.4 Special method requirements

Analysis of the non-BDE compounds (PBEB, HBB, BTBPE, and DBDPE) is a special addition to the 1614 method. The laboratory awarded the contract for analysis will be required to have experience analyzing and reporting the non-BDE compounds.

9.5 Lab(s) accredited for method(s)

The laboratory awarded the contract for brominated flame retardants will be required to hold accreditation by Ecology for EPA Method 1614.

10.0 Quality Control (QC) Procedures

10.1 Table of field and lab QC required

Table 8 presents laboratory QC sample types and the frequency at which they will be tested during the brominated flame retardant analysis. No change was made to the field QC procedures from the original QAPP. QC sample types and frequency for additional metals will follow that outlined for lead in the original QAPP (Coots, 2006).

Table 8. QC Samples, Types, and Frequency.

Parameter	Matrix	Field		Laboratory			
		Blanks	Replicates	LCS	Method blanks	Analytical duplicates	Surrogates
BFRs	sediment	n/a	none	1/batch	1/batch	1/batch	each sample

Batch = 20 samples or fewer

10.2 Corrective action processes

If QC tests are below MQOs during the analysis, the laboratory will contact the project manager to discuss possible corrective action.

11.0 Data Management Procedures

11.2 Laboratory data package requirements

The laboratory will be required to deliver an EPA Tier IV data package to MEL with analytical results, including all raw sample data, raw QC data, and instrument calibration data.

11.3 Electronic transfer requirements

MEL will send the project manager an electronic data deliverable in the form of an Excel spreadsheet with the analytical results, along with MEL-amended data qualifiers.

12.0 Audits and Reports

12.1 Number, frequency, type, and schedule of audits

No field or laboratory audits will be made for this study.

12.3 Frequency and distribution of report

The additional metals being analyzed in the Pierre Lake sediment core will be reported to TCP ERO staff in a separate data submittal memo.

13.0 Data Verification

13.2 Lab data verification

MEL's QA officer will review the Tier IV data package from the contract laboratory to verify that the analytical method was followed correctly and data were reported without omissions or errors. MEL will provide a case narrative to the project manager documenting holding times, instrument calibrations, QC test results, and any other information regarding the quality of the data analysis.

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