



DEPARTMENT OF  
**ECOLOGY**  
State of Washington

**Addendum 6 to  
Quality Assurance Project Plan**

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**Freshwater Fish Contaminant  
Monitoring Program: 2017**

October 2017

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**Addendum 6 to  
Quality Assurance Project Plan**

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**Freshwater Fish Contaminant  
Monitoring Program: 2017**

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Signatures are not available on the Internet version.

EAP: Environmental Assessment Program

TSU: Toxics Studies Unit

WQP: Water Quality Program

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## 3.0 Background

This document describes the 2017 sampling effort for the Washington State Department of Ecology (Ecology) Freshwater Fish Contaminant Monitoring Program (FFCMP) and is an addendum to the Quality Assurance Project Plan (Seiders, 2013). The 2017 sampling effort will focus on the Okanogan River basin in Washington. The main goals are to characterize current contaminant levels in resident fish, establish a robust baseline for future monitoring efforts, and determine changes over time by comparing results with historical data.

Previous studies and associated data were reviewed to guide development of project objectives and the sampling plan. Contaminants assessed in fish from previous studies focused mainly on CPs (chlorinated pesticides) and PCBs (polychlorinated biphenyls). Limited sampling has been done for other contaminants, such as PBDEs (polybrominated diphenyl ethers), PCDD/Fs (polychlorinated dibenzo-p-dioxins and -furans), mercury, and other metals.

The pesticide DDT and its breakdown products, as well as PCBs, were often found in fish at elevated levels from which a decrease could likely be detected over time, assuming that inputs decrease. Reductions in contaminant levels might also approach levels seen in similar species from “background” areas – those areas not directly impacted by human activities.

Collectively, data from historical sampling efforts comprise a mix of sites, species, tissue types, collection seasons, and analytical methods. The most recent monitoring effort in 2008 (Newell, 2011) did not detect significant temporal changes in fish contaminant concentrations largely because of typical challenges associated with such efforts, such as small sample sizes and high variability associated with fish tissue.

Information about previous work on contaminants in fish from the 2017 target locations is summarized below.

### Okanogan River

Three areas in the Okanogan River mainstem within Washington have been sampled in the past: the upper, middle and lower reaches between Oroville and the confluence with the Columbia River at Lake Pateros.

Sampling in the 1980s and 1990s were part of statewide screening-level studies for various contaminants and some focused studies. High levels of DDT and PCBs led to a larger Total Maximum Daily Load (TMDL) technical study in 2001 (Serdar, 2003). The Water Quality Implementation Plan for this TMDL (Peterschmidt, 2006) recommended that fish tissue be monitored periodically for changes in contaminant levels. Fish were sampled in 2008 as part of the TMDL Effectiveness Monitoring effort (Newell, 2011). The FFCMP sampled Lake Pateros in 2013 as part of a Columbia River focus (Seiders et al, 2015) and found high levels of DDT in carp and northern pikeminnow.

Throughout these studies, the Washington Department of Health has been consulted on sampling plans and results to ensure that the data met its needs for conducting risk assessments. Health issued a Fish Consumption Advisory after the TMDL study and revised it after the 2008 study (Health, 2011).

Table 1 shows the timeframe, species, and target analytes for studies conducted in the Okanogan River.

Results from the studies described above showed that concentrations of several chemicals in fish tissue were above the Fish Tissue Equivalent Concentration (FTEC) used by Ecology to determine whether Washington water quality standards are met. These results led to 303(d) listings for DDT compounds and PCBs (Table 2). The 303(d) listings are also known as Category 5 listings in Ecology's periodic statewide Water Quality Assessment (<http://www.ecy.wa.gov/programs/Wq/303d/index.html>). When a water cleanup plan is created (i.e. an EPA-approved TMDL), Category 5 listings are re-assigned as Category 4A listings which indicate that full implementation of the cleanup plan is expected to result in standards being met.

Table 1. Summary of fish tissue samples from the Okanogan River by study, site, and species.

Study ->	BWMP <sup>1</sup>			WSPMP <sup>2</sup>			Osoyoos <sup>3</sup>	TMDL <sup>4</sup>			TMDL EM <sup>5</sup>		
Sample Year ->	1984			1994			1995	2001			2008		
Reach->	Upper	Middle	Lower	Upper	Middle	Lower	Lake Osoyoos	Upper	Middle	Lower	Upper	Middle	Lower
<b>Species</b>													
BLS			1f										
CCP						1f	3f	3f	3f		1f	2f	4f
LMB			1f										
LSS						2w	2w						
LWF							1f, i						
MWF							1f, i	3f	3f **	3f	3f*		
SMB							3f	3f	3f	3f	2f	5f	3f
YP							7f						
<b>Target Analytes</b>													
CPs			x			x	x	x	x	x			
PCBs			x			x	x	x	x	x			
PBDEs											xx	xx	xx
PCDD/Fs											xx	xx	
Mercury			x								xx	xx	xx
Metals			x										
Lipids			x			x	x	x	x	x	xx	xx	xx
EIM Study ID	BHOP0002			WSPMP94T			DDT-OSOY	DSER0009			CCOF0003		

Study Codes: BWMP - Basic Water Monitoring Program; TMDL - Total Maximum Daily Load Study; TMDL EM - TMDL Effectiveness Monitoring study; WSPMP - Washington State Pesticide Monitoring Program; WSTMP - Washington State Toxics Monitoring Program.

References: 1- Hopkins, 1985; 2- Davis and Serdar, 1996; 3 – Serdar et al, 1998; 4- Serdar, 2003; 5- Newell, 2011; 6- Seiders and Deligeannis, 2009.

Most samples are composites of fillets (f) or whole fish (w) from multiple fish.

xx - some fish also analyzed by the WSTMP <sup>6</sup> for PBDEs, PCDD/Fs, and mercury (EIM Study ID WSTMP08)

\* fish collected at mouth of Similkameen River

\*\* composites were formed from 27 fish which were also analyzed individually for DDTs and PCBs by the WSTMP (EIM Study ID WSTMP03T)

Species Codes: BLS - Bridgelip sucker; CCP – Common carp; LMB - Largemouth bass; LSS -: Largescale sucker; LWF – Lake whitefish; MWF - Mountain whitefish; SMB - Smallmouth bass; YP – Yellow perch.



Table 2. Category 5 and 4A Listings for the Okanogan River.

River Section	Listing ID	Category	Parameter	Assessment Unit ID	EIM User Location ID	Sample Year	Species Not Meeting Standards
Lake Osoyoos	8970	4A	4,4'-DDD	48119J4H3	OSOYOOSLAKE	1995	CCP, LMB, LWF
Lake Osoyoos	8971	4A	4,4'-DDE	48119J4H3	OSOYOOSLAKE	1995	CCP, LMB, LWF, MWF, SMB, YP
Upper	72174	5	4,4'-DDE	17020006005138	OKANOTMDL16	2001	CCP, MWF, SMB
Upper	78944	5	PCBs	17020006005138	OKANOTMDL16	2001	CCP, MWF
Upper	72173	5	4,4'-DDE	17020006005059	CCOF0003-OKU	2008	CCP, SMB
Middle	76156	5	4,4'-DDE	17020006008524	CCOF0003-OKM	2008	CCP, SMB
Middle	78946	5	PCBs	17020006008524	CCOF0003-OKM	2008	CCP
Middle	74499	5	4,4'-DDD	17020006000066	OKANOTMDL14	2001	MWF
Middle	72171	5	4,4'-DDE	17020006000066	OKANOTMDL14	2001	CCP, MWF, SMB
Middle	75053	5	4,4'-DDT	17020006000066	OKANOTMDL14	2008	MWF
Middle	78943	5	PCBs	17020006000066	OKANOTMDL14	2001	CCP, MWF, SMB
Lower	8992	4A	4,4'-DDD	17020006018013	OKARNM	1994	CCP
Lower	8991	4A	4,4'-DDE	17020006018013	OKARNM	1994	CCP
Lower	8993	4A	PCBs	17020006018013	OKARNM	1994	CCP
Lower	14353	4A	4,4'-DDD	17020006000055	OKANOGANRIVER(49A070)	1984	BLS, LMB
Lower	14354	4A	4,4'-DDE	17020006000055	OKANOGANRIVER(49A070)	1984	BLS, LMB
Lower	14352	4A	4,4'-DDT	17020006000055	OKANOGANRIVER(49A070)	1984	BLS, LMB
Lower	14356	4A	PCBs	17020006000055	OKAN@MALOTT	1984	LMB
Lower	74498	5	4,4'-DDD	17020006000049	OKANOTMDL13 CCOF0003-OKL	2008	CCP
Lower	72170	5	4,4'-DDE	17020006000049	OKANOTMDL13 CCOF0003-OKL	2008	CCP, MWF, SMB
Lower	78942	5	PCBs	17020006000049	OKANOTMDL13 CCOF0003-OKL	2008	CCP, MWF, SMB

Species Codes: BLS - Bridgelip sucker; CCP - Common carp; LMB - Largemouth bass; LWF -Lake whitefish; MWF - Mountain whitefish; SMB - Smallmouth bass; YP - Yellow perch.

Figures 1-6 show results from past studies for 4,4'-DDE (DDE) and t-PCBs in fish tissue. Levels of DDE are well above the FTEC of 32 ug/kg and show differences among species and sites. Many past samples exceeded FTECs for t-PCBs (5.3 ug/kg) while others did not. Washington's water quality standards were revised in the fall of 2016 and Ecology is reconsidering how fish tissue data will be used in the Water Quality Assessment.

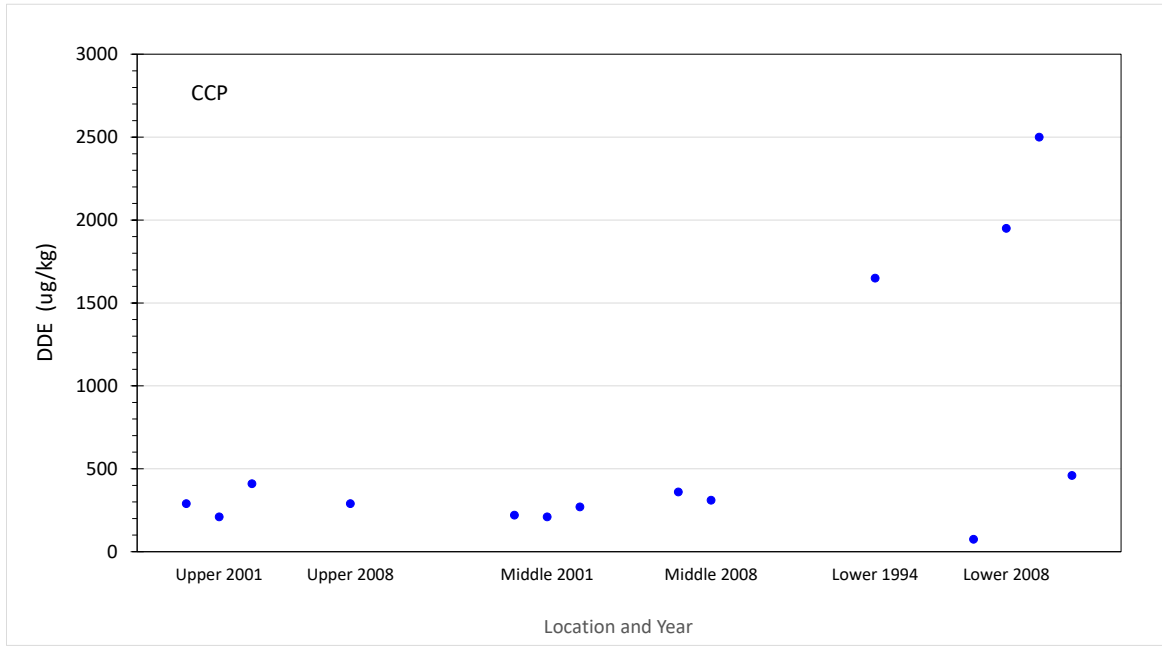


Figure 1. Results for DDE in Common carp (CCP) from the Okanogan River.

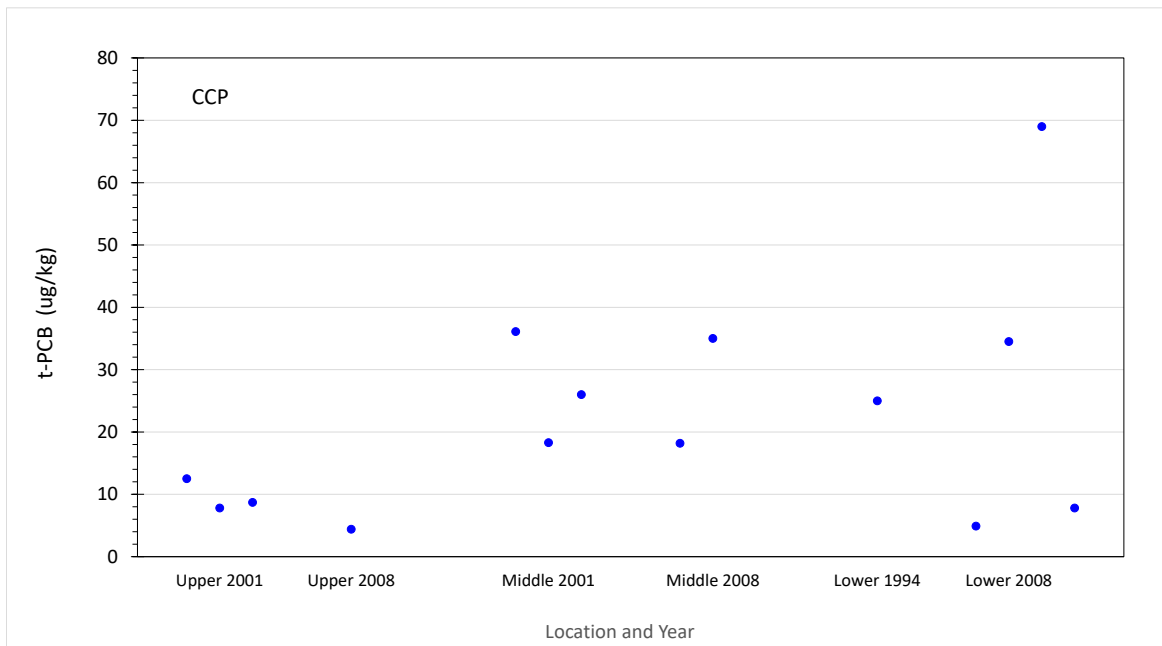


Figure 2. Results for t-PCBs in Common carp (CCP) from the Okanogan River.

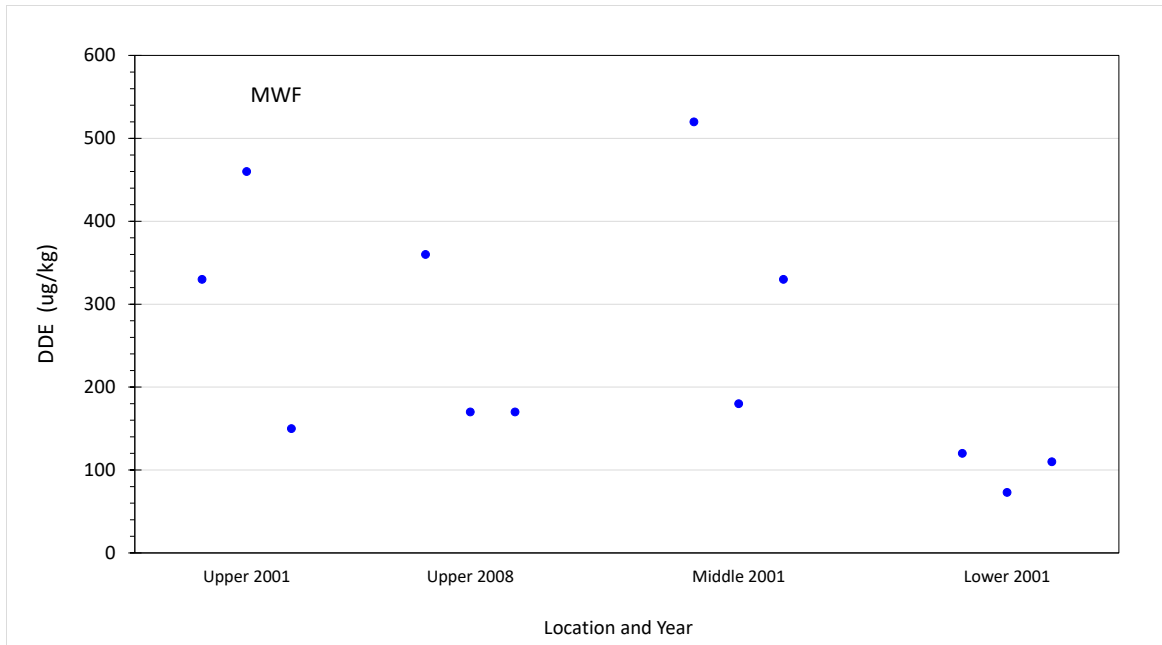


Figure 3. Results for DDE in Mountain whitefish (MWF) from the Okanogan River.

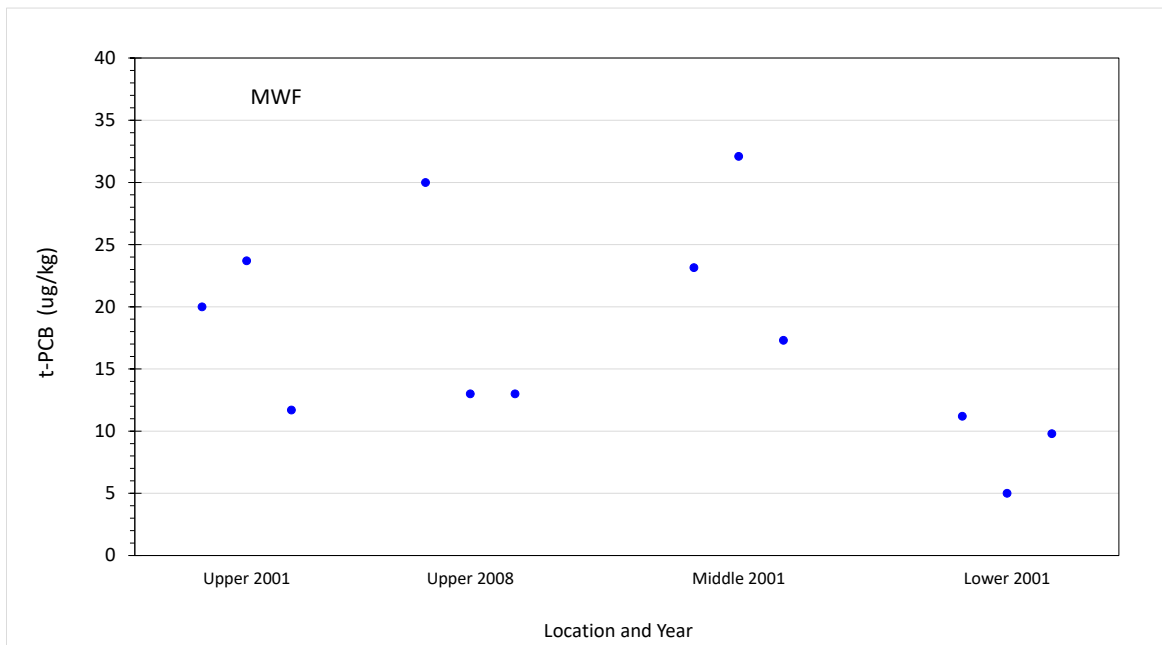


Figure 4. Results for t-PCBs in Mountain whitefish (MWF) from the Okanogan River.

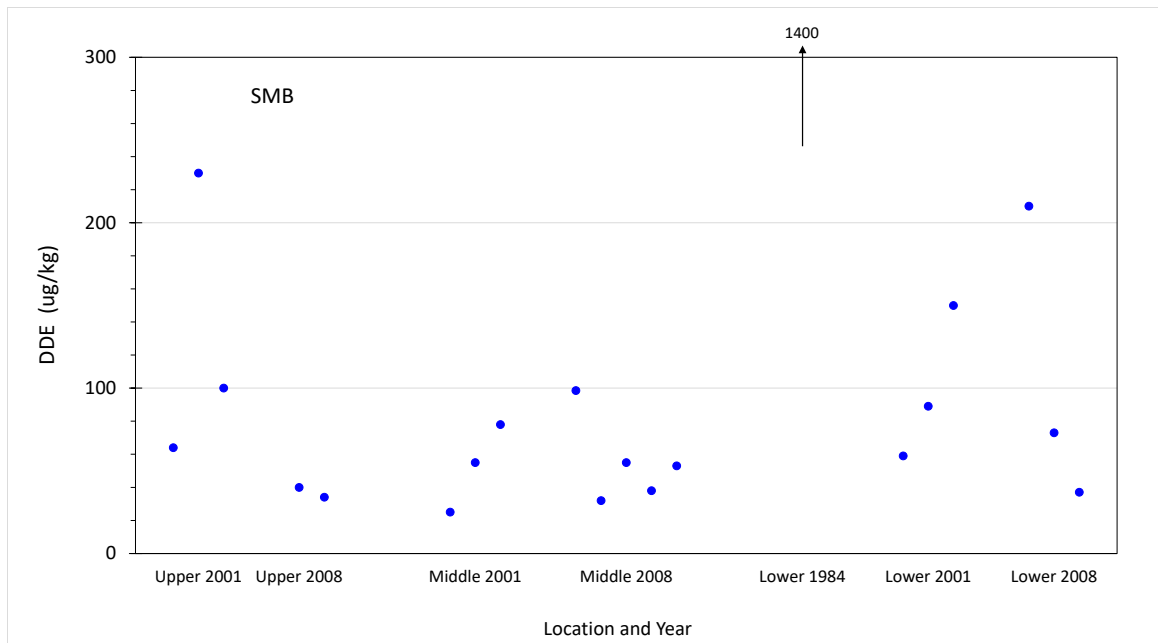


Figure 5. Results for DDE in Smallmouth bass (SMB) from the Okanogan River.

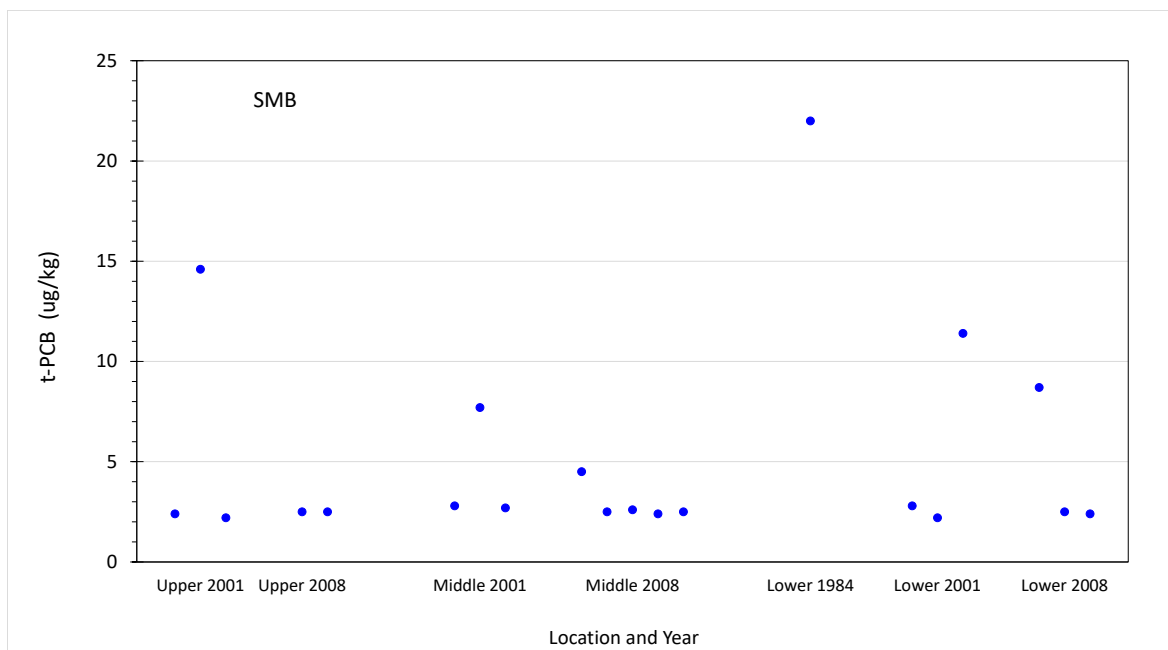


Figure 6. Results for t-PCBs in Smallmouth bass (SMB) from the Okanogan River.

## 4.0 Project Description

The goal of the 2017 monitoring effort is to develop a robust data set of contaminant concentrations in fish from the Okanogan River in order to:

- Characterize temporal trends by comparisons to historical and future data.
- Compare results to water quality standards.
- Support fish consumption risk assessments by health jurisdictions.
- Inform water quality management efforts such as the current TMDL for the river.

Site selection was described in the original QAPP and is refined here for the 2017 effort. The key characteristics of sites selected for long term monitoring are:

- Concentrations of key contaminants are elevated in fish tissue.
- Likelihood of detecting change in contaminant levels over time.
- Presence of historical data that can be used for temporal comparisons.
  - Multiple samples taken during each of the previous efforts.
  - Multiple sampling efforts at different times in the past.
  - Potential for pooling data to increase statistical sensitivity.
- Waters impaired: Category 5, 4, or 2 from the most recent Water Quality Assessment.
- Ability to collect desired species: sampling access, permits, species abundance.

Figure 7 shows historic sampling locations which are also the target areas for the 2017 effort. The sampling locations correspond to the “Map Reference” column in Table 3.

Table 3 shows location information for sites that were sampled in the past. The mainstem river sites are grouped into the general areas of Upper, Middle, and Lower river segments. The 2017 effort will focus on these same areas of the Okanogan River from Lake Osoyoos to Lake Pateros. Lake Pateros, between Brewster and Chief Joseph State Park, is included because the Lower Okanogan River Fish Consumption Advisory includes part of this lake.

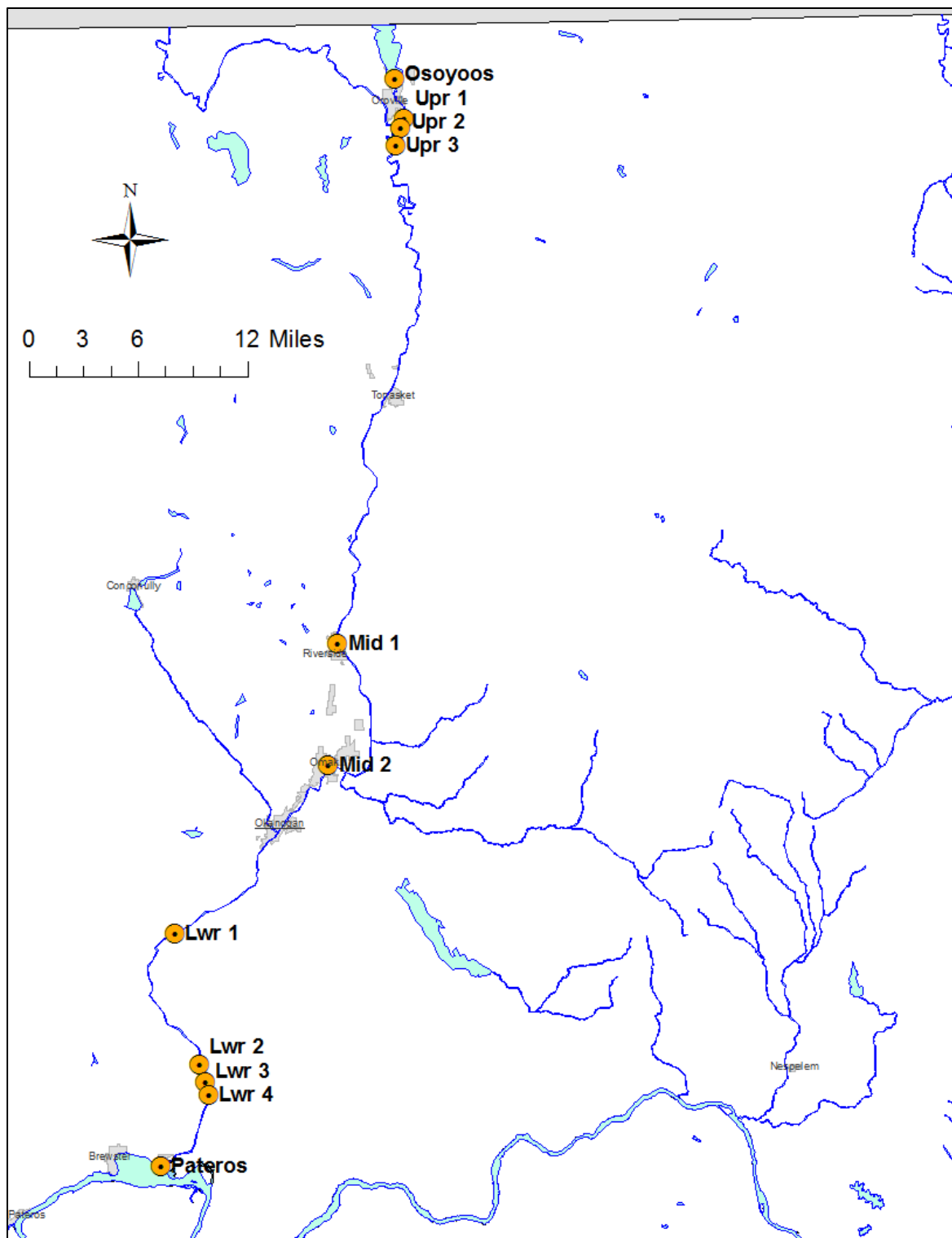


Figure 7. Proposed sampling locations for the FFCMP in 2017.

Table 3 Site information for proposed sample locations, FFCMP 2017.

2017 Sampling Area	Location Description	Map Reference	Historic EIM Location ID	NHD Reach Code	Latitude*	Longitude*	Historic EIM Study ID
Lake Osoyoos	Lake Osoyoos, south of the Canadian border	Osoyoos	OKANOTMDL10	17020006001115, 48119J4H3	48.9572	-119.4306	DDTOSOY
Upper	Stretch of river between the fork that flows into the Similkameen River RM 76.3 and just before Oroville RR crossing RM 77.3	Upr 1	OKANOTMDL16	17020006005138	48.9250	-119.4194	DSER0009
	Upper Okanogan River between approximately RM 76.2-76.4	Upr 2	CCOF0003-OKU	17020006005059	48.9178	-119.4235	CCOF0003
	Similkameen River Mouth	Upr 3	CCOF0003-SIM	17020007004433	48.9043	-119.4304	CCOF0003
Middle	Middle Okanogan River; reach started at primitive boat launch w/in Riverside and proceeded upstream (approximately RM 40-42)	Mid 1	CCOF0003-OKM	17020006008524	48.5093	-119.5074	CCOF0003
	Two segments: reach between Omak Ave. bridge and Highway 97 bridge; reach between RM 39.4 AND RM 41.0 that passes through the town of Riverside	Mid 2	OKANOTMDL14	17020006000066	48.4128	-119.5201	DSER0009
Lower	Okanogan River below Malott	Lwr 1	OKANOGRIVER		48.2805	-119.7052	BHOP0002
	Stretch of river between Monse bridge RM 4.8 and closest upstream RR crossing at RM 10.5	Lwr 2	OKANOTMDL13	17020006000049	48.1770	-119.6782	DSER0009
	Lower Okanogan River; reach started at Monse Road bridge and proceeded upstream 3 to 4 miles (approximately RM 5-8)	Lwr 3	CCOF0003-OKL	17020006000049	48.1625	-119.6705	CCOF0003
	Okanogan River above Brewster	Lwr 4	OKARNM		48.1528	-119.6664	WSPMP04T
Lake Pateros	Lake Pateros near confluence with Okanogan River; roughly from Brewster to Chief Joseph State park	Pateros	-	17020005000040, 17020005000042, 17020005000043	48.0965	-119.7248	FFCMP17

RM: River mile

\* approximate center of previously sampled stream segment; NAD83 HARN

The primary target analytes for long term trend assessment are CP and PCB compounds. Other contaminants for which there are little or no historical data will be characterized, such as, mercury, polybrominated diphenyl ethers (PBDEs), and polychlorinated dibenzo-p-dioxins and -furans (PCDD/Fs).

This project will use data collected through past monitoring efforts conducted by Ecology as described above. These data and associated documentation (e.g., project plans, project reports, and laboratory data reports) will be reviewed to assess their usability in this project.

For the long-term monitoring strategy at selected sites in the target watersheds, multiple replicates of composite samples for each species at each site are anticipated to provide an adequately robust data set that will meet objectives. Review of field replicate data from previous FFCMP work showed that variance is inconsistent and can be high for organic contaminants, ranging up to 100% Relative Percent Difference (RPD) for PCBs, DDTs, and PCDD/Fs. A sample size of five to seven composite samples should reduce the variability associated with the mean and median tissue concentrations and improve the ability to determine change among sample results over time. The use of three to five fish of the same species and similar size in each composite sample also helps reduce variability.

Table 4 shows the sites, target species, and target number of analyses of composite samples for each suite of analyses. Three composite species will analyzed for most analytes while seven composite samples will be analyzed for the most frequently detected DDT and PCB compounds: 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, and PCB Aroclors 1248, 1254, and 1260. These compounds are analyzed in two different suites of analyses so the total number of samples analyzed is the sum of values in the 4<sup>th</sup> and 5<sup>th</sup> columns in Table 4 (i.e. 3+4=7 samples).

The 2017 sampling will attempt to collect fish of the same species and size ranges that were collected in the past. Actual numbers of samples to be analyzed will likely be adjusted depending on success of fish collection efforts: past FFCMP efforts have met about 60-80% of the target number of fish.

While the 2008 study (Newell, 2011) recommended that smallmouth bass not be analyzed for PCBs due to relatively low concentrations found in this species, the 2017 plan retains this analysis until revision of Water Quality Program Policy 1-11 is completed: revisions to this policy, in conjunction with the revised water quality standards, may warrant the analysis of smallmouth bass for PCBs.

Other species may be collected and analyzed if interest and resources allow. Local fish biologists have expressed interest in contaminant levels in walleye from Lake Pateros and its extension into the lower reach of the Okanogan River. Walleye is a popular sports fishery in the area. Freshwater mussels may also be collected and analyzed if feasible. Freshwater mussels have high site fidelity as adults, can live for decades, and accumulate various contaminants.



Table 4. Sample plan and estimated laboratory costs, FFCMP 2017.

Site	Species Code	Number of Composite Samples for Each Analysis Suite					Total # fish for 5 fish per composite
		Mercury	Cl Pest, PCB Aroclors, PBDE, lipid	3 PCB Aroclors, 3 DDTs, lipid	PCDD/F	CL Pest, HiRes **	
Lake Osoyoos	CCP	3	3	4	3	3	35
	LSSw		3	4			35
	NPM	3	3	4	3	3	35
	SMB	3	3	4			35
Upper Okanogan River	CCP	3	3	4	3	3	35
	LSSw		3	4			35
	MWF	3	3	4	3	3	35
	SMB	3	3	4			35
Middle Okanogan River	CCP	3	3	4	3	3	35
	LSSw		3	4			35
	MWF	3	3	4	3	3	35
	SMB	3	3	4			35
	MUS	3	3				15
Lower Okanogan River	CCP	3	3	4	3	3	35
	LSSw		3	4			35
	MWF	3	3	4	3	3	35
	SMB	3	3	4			35
Confluence: Okanogan River and Lake Pateros	CCP	3	3	4	3	3	35
	LSSw		3	4			35
	NPM	3	3	3	3		30
	SMB	3	3				15
	WAL	3	3				15
	Total # field samples	51	66	75	30	27	705
	Total # lab QC analyses	6	6	8	2	1	
	Total # analyses	57	72	83	32	28	
	Cost per analysis	\$50	\$620	\$264	\$600	\$1,100	
	Subtotal costs	\$2,850	\$44,640	\$21,912	\$19,200	\$30,800	
	<b>Grand Total</b>		<b>\$88,602</b>			<b>\$30,800</b>	

\*\* High resolution analysis performed only if selected pesticides are not detected in analyses using EPA Method 8081: Additional funding may be needed.

Species codes: CCP - Common Carp; LSSw - Largescale sucker (as whole fish); MWF - Mountain whitefish; MUS - Freshwater mussel; NPM - Northern pikeminnow; SMB - Smallmouth bass; WAL - Walleye.

## 5.0 Organization and Schedule

Table 5 lists the people involved in this project. All are employees of the Washington State Department of Ecology. Table 6 is the proposed schedule for this project.

Table 5. Organization of project staff and responsibilities, FFCMP 2017.

EAP Staff	Title	Responsibilities
Jessica Archer EAP-SCS 360-407-6698	Client	Provides internal review of the QAPP, addendums, and reports. Approves the final QAPP and addendums.
Keith Seiders Toxics Studies Unit EAP-SCS 360-407-6689	Project Manager and Principal Investigator	Writes the QAPP, addendums, and reports. Reviews historical data and develops sample strategy for different sites on annual basis. Works with laboratories to obtain analytical services. Reviews, analyzes, and interprets data. Guides field assistants in various roles and tasks.
Patti Sandvik Toxics Studies Unit EAP-SCS 360-407-7198	Field and EIM Lead, Project Assistant	Leads efforts for sample collection, processing, and transportation of samples to the laboratory. Ensures that field and processing information is recorded. Enters field and laboratory data into EIM. Compiles and summarizes historical and current-year data. Assists report effort.
Debby Sargeant Toxics Studies Unit EAP SCS 360-407-6139	Unit Supervisor for the Project Manager	Provides internal review of the QAPP, addendums, and reports. Approves the final QAPP and addendums. Manages budget and staffing needs.
George Onwumere EAP-EOS 360-407-6730	Supervisor, EOS, Central Regional Office	Helps coordinate CRO EAP staff involvement as needed.
Mark Peterschmidt CRO WQP 509-454-7843	Supervisor, WQP, Central Regional Office	Helps coordinate CRO and stakeholder involvement, particularly with the Colville Tribe and other interested parties.
Alan Rue EAP-Manchester Environmental Lab 360-871-8801	Acting Laboratory Director	Approves the final QAPP. Oversees all operations at MEL regarding in-house analyses and processes for contracting analyses to commercial labs.
William R. Kammin EAP-QA 360-407-6964	Ecology Quality Assurance Officer	Reviews the draft QAPP and addendums. Approves the final QAPP and addendums.

EAP: Environmental Assessment Program

EIM: Environmental Information Management database

QAPP: Quality Assurance Project Plan

SCS: Statewide Coordination Section

EOS: Eastern Operations Section

CRO: Central Regional Office

WQP: Water Quality Program

Table 6. Schedule for completing field, laboratory, and report tasks, FFCMP 2017.

Field and laboratory work	Due date	Lead staff
Field work completed	September 2017	Patti Sandvik
Sample processing completed	December 2017	Patti Sandvik
Ecology lab analyses completed	July 2018	MEL, Alan Rue
Contract lab analyses completed	July 2018	MEL, Alan Rue
Environmental Information System (EIM) database		
EIM user study ID	FFCMP17	
Product	Due date	Lead staff
EIM data loaded	September 2018	Patti Sandvik
EIM data verification	October 2018	To be determined
EIM complete	November 2018	Patti Sandvik
Final report		
Author lead / Support staff	Keith Seiders / Patti Sandvik	
Schedule		
Draft due to supervisor	March 2019	
Draft due to client/peer reviewer	April 2019	
Draft due to external reviewer(s)	April 2019	
Final (all reviews done) due to publications coordinator	May 2019	
Final report due on web	June 2019	

## 6.0 Quality Objectives

Table 7 shows measurement quality objectives (MQOs).

Table 7. Measurement quality objective, FFCMP 2017.

Parameter	Analytical Method	Lab Duplicate (as RPD)	Lab Control Sample (% recovery)	Surrogates (% recovery)	Matrix Spike/Spike Duplicate (% recovery)
Mercury	EPA 245.6 (CVAA)	0%-20% (for results > 5x RL)	85%-115%	NA	75%-125%; RPD limit 20%
Chlorinated pesticides	EPA 8081 (GC/ECD); MEL SOP	0%-40%	50%-150%	20%-130% <sup>a</sup>	50%-150%; RPD limit 40%
Chlorinated pesticides (HiRes short list: if needed)	EPA 1699 or equivalent (HiRes GC/MS)	0%-40%	Per method for OPR, Internal Standards, and Labeled Compounds each sample & 1/batch <sup>b</sup>	NA	NA
PCB Aroclors	EPA 8082 (GC/ECD); MEL SOP	0%-40%	50%-150%	50%-150%	50%-150%; RPD limit 40%
PCB congeners (if needed, none planned for 2017)	EPA 1668A (HiRes GC/MS)	0%-40%	Per method for OPR, Internal Standards, and Labeled Compounds	NA	NA
PCDD/Fs	EPA 1613B (HiRes GC/MS)	0%-40%	Per method for OPR, Internal Standards, and Labeled Compounds	NA	NA
PBDEs	EPA 8270 (SIM); SOP 730104	0%-40%	50%-150%	50%-150%	50%-150%; RPD limit 40%
Lipids	MEL SOP 730009	0%-20%	NS	NA	NA

<sup>a</sup> Surrogate recovery limits were recently revised by MEL and are specific to surrogates used: some limits are 20%-120%, others are 30%-130%.

<sup>b</sup> Labeled compounds in each sample and Ongoing Precision and Recovery standards in each batch.

## 8.0 Sampling Procedures

Samples will be collected and processed as described in the project plan for the FFCMP (Seiders, 2013). Fish collection methods may include the use of gillnets, seines, electrofishing, and angling. Federal and state scientific collection permits provide guidance for minimizing the disturbance of anadromous salmon and steelhead that may be present. These include National Marine Fisheries Permit # 1386-R9, U.S. Fish and Wildlife Service Permit # TE058381-8, Washington Department of Fish and Wildlife Permit SANDVIK 12-298i. This project will also apply to the Confederated Tribes of the Colville Reservation for a collection permit where segments of the Okanogan River are under tribal jurisdiction.

Table 8 shows sample containers, preservation, and holding times for fish tissue samples.

Table 8. Containers, preservation, and holding times for samples, FFCMP 2017.

Parameter	Sample Container	Minimum Amount Required (wet weight)	Preservation	Holding Time
Mercury	2 oz. precleaned glass jar w/teflon lid	5g	freeze, -10° C	6 months to extraction, then 28 days to analysis
Chlorinated Pesticides	4 oz. precleaned glass jar w/teflon lid	30g, 60g preferred	freeze, -10° C	1 year to extraction, then 40 days to analysis
PCB Aroclors	4 oz. precleaned glass jar w/teflon lid	30g, 60g preferred	freeze, -10° C	1 year to extraction, then 40 days to analysis
PCB congeners	4 oz. precleaned glass jar w/teflon lid	30g, 60g preferred	freeze, -10° C	1 year to extraction, then 40 days to analysis
PCDD/Fs	4 oz. precleaned glass jar w/teflon lid	30g, 60g preferred; ~220g if base digestion	freeze, -10° C	1 year to extraction, then 40 days to analysis
PBDEs	4 oz. precleaned glass jar w/teflon lid	30g, 60g preferred	freeze, -10° C	1 year to extraction, then 40 days to analysis
Lipids	4 oz. precleaned glass jar w/teflon lid	30 g	freeze, -10° C	1 year to extraction, then 40 days to analysis

### **8.3 Invasive species evaluation**

Invasive or unwanted aquatic species may be encountered during fish collections for this project. Environmental ethics and Washington law prohibit the transportation of all aquatic plants, animals, and many noxious weeds. Sample collection efforts for this project will follow the Ecology Environmental Assessment Program's SOP to Minimize the Spread of Invasive Species (Parsons et al., 2012) and Washington Department of Fish and Wildlife's Invasive Species Management Protocols (Tweit et al., 2011). For this year's target sites, the New Zealand mudsnail, an invasive species of extreme concern, is not known to be present.

## 9.0 Measurement Methods

The analytical methods are consistent with the most recent FFCMP monitoring events. Laboratory analyses of most samples will be conducted by the Ecology Manchester Environmental Laboratory (MEL). Analyses for PCB congeners, PCDD/Fs, and chlorinated pesticides (if needed) will be done by an accredited laboratory through a contract managed by MEL. Both MEL and the contract laboratories are expected to meet the QC requirements of the analytical methods being used and any other requirements specified by MEL or the Project Officer in the Statement of Work part of the contract.

Table 9 shows the parameters to be analyzed, analytical methods, desired reporting limits, and ranges of expected results (all results expressed as wet weight).

Table 9. Laboratory measurement methods for fish tissue samples, FFCMP 2017.

Parameter	Methods, Reporting Limits, and Sample Number			
	Number of Samples and Arrival Date <sup>a</sup>	Expected Range of Results <sup>b</sup>	Reporting Limits <sup>c</sup>	Analytical Method
Mercury	39, January 2018	10 - 1000 ug/kg	17 ug/kg	EPA 245.6 (CVAA)
Chlorinated pesticides	51-full suite plus 67 for 3 DDTs only, January 2018	0.1 - 1000 ug/kg for DDTs; 0.1 - 50 ug/kg for others	most 0.5 - 3.0 ug/kg	EPA 8081 (GC/ECD); MEL SOP
Chlorinated pesticides (HiRes short list IF NEEDED)	24, April, 2018	Varies by analyte: see Table 11	Varies by analyte: see Table 11	EPA 1699 or equivalent (HiRes GC/MS)
PCB Aroclors	51-full suite, plus 67 for 3 Aroclors only, January 2018	0.5 - 100 ug/kg, depending on Aroclor	1.1 - 5 ug/kg	EPA 8082 (GC/ECD); MEL SOP
PCB congeners (none planned for 2017)	None planned	0.005 - 10 ug/kg, depending on congener	0.003-0.01 ug/kg	EPA 1668A (HiRes GC/MS)
PCDD/Fs	27, January 2018	0.005 - 5.0 ng/kg, depending on congener and extraction method	EQL (Estimated Quantitation Limit) 0.017 - 0.5 ng/kg	EPA 1613B (HiRes GC/MS)
PBDEs	51, January 2018	0.1 - 100 ug/kg	0.10 - 2.6 ug/kg; PBDE 209 1.9 - 4.3 ug/kg	EPA 8270 (SIM); MEL SOP 730104
Lipids	108 (51+67), January 2018	0.1 - 20%	0.10%	MEL SOP 730009

<sup>a</sup> MEL will be informed of numbers and arrival dates when the sampling effort concludes.

<sup>b</sup> Values reflect historical data from the study area.

<sup>c</sup> Value reflects typical range.

Analytical methods need to be adequately sensitive to compare to FTECs or other numerical values that will be used in the Water Quality Assessment. Fish tissue is a challenging matrix and presents various interferences that often raise reporting limits for six chlorinated pesticides. These pesticides are aldrin, alpha-BHC, dieldrin, heptachlor, heptachlor epoxide, and toxaphene. These pesticides were identified in Table D-1 of the original QAPP as possibly needing extra effort by labs to achieve desired reporting limits (Seiders, 2013).

In order to obtain results that can be compared to FTECs or other numerical values to be used in the Water Quality Assessment, a second round of analyses for pesticides may be conducted after reviewing results from the first round of analyses. An HRGC/HRMS method for chlorinated pesticides, such as method EPA 1699, or an equivalent or more sensitive method, will be used for this second round of analyses. A qualified laboratory will be selected through the Department of Enterprise Services bid solicitation process.

Table 10 shows the pesticides which might be analyzed with a more sensitive method, along with detection limits, current FTEC, and expected range of results. Pesticides other than the six mentioned above are also being analyzed to help inform MEL about issues related to interferences and interpretations of their analysis using EPA 8081.

Table 10. Characteristics of chlorinated pesticides to be analyzed using HR GC/MS for the FFCMP 2017 (ug/kg wet weight).

Analyte	CAS #	Required Detection Limit	FTEC	Expected Range of Results
Aldrin	309-00-2	0.01	0.654	ND - 1.0
alpha-BHC (alpha-HCH)	319-84-6	0.02	1.69	ND - 1.0
Dieldrin	60-57-1	0.01	0.654	ND - 10
Heptachlor	76-44-8	0.02	2.35	ND - 10
Heptachlor Epoxide	1024-57-3	0.02	1.23	ND - 10
Toxaphene	8001-35-2	0.20	9.56	ND - 50
beta-BHC (beta-HCH)	319-85-7	0.02	5.98	ND - 1.0
Hexachlorobenzene	118-74-1	0.02	6.69	ND - 20

CAS: Chemical Abstract Service.

FTEC: Fish Tissue Equivalent Concentration.

ND: Non detect



## 10.0 Quality Control

Table 11 shows laboratory quality control procedures.

Table 11. Laboratory quality control sample types and frequencies, FFCMP 2017.

Parameter	Analytical Method	Lab Duplicates	Lab Control Standards	Surrogates	MS/MSD	Method Blanks
Mercury	EPA 245.6 (CVAA)	1/ batch <sup>a</sup>	1/batch	NA	1/batch	1/batch
Chlorinated pesticides	EPA 8081 (GC/ECD); MEL SOP	1/batch	1/batch	each sample	1/batch	1/batch
Chlorinated pesticides (HiRes short list)	HiRes GC/MS (EPA 1699 or equivalent)	1/batch	each sample & 1/batch <sup>c</sup>	NA	NA	1/batch
PCB Aroclors	EPA 8082 (GC/ECD); MEL SOP	1/batch	1/batch	each sample	1/batch	1/batch
PCB congeners <sup>b</sup> (none planned for 2016)	EPA 1668A (HiRes GC/MS)	1/batch	each sample & 1/batch <sup>c</sup>	NA	NA	1/batch
PCDD/Fs <sup>b</sup>	EPA 1613B (HiRes GC/MS)	1/batch	each sample & 1/batch <sup>c</sup>	NA	NA	1/batch
PBDEs	EPA 8270 (SIM); SOP 730104	1/batch	1/batch	each sample	1/batch	1/batch
Lipids	MEL SOP 730009	1/batch	1/batch	NA	NA	1/batch

<sup>a</sup> “Batch” is defined as up to 20 samples analyzed together.

<sup>b</sup> Includes one analysis of Certified Reference Material for the project (WMF-01 preferred; CARP-2 acceptable)

<sup>c</sup> Labeled compounds in each sample and Ongoing Precision and Recovery standards in each batch.

## 15.0 References

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