

# Addendum 2 to Quality Assurance Project Plan

Freshwater Fish Contaminant Monitoring Program, 2021: Lake Chelan

September 2021 Publication 21-03-117

# **Publication Information**

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# Addendum 2 to Quality Assurance Project Plan

# Freshwater Fish Contaminant Monitoring Program, 2021: Lake Chelan

by

Keith Seiders and Patti Sandvik

September 2021

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ERO: Eastern Regional Office	
MEL: Manchester Environmental Laboratory	
SCS: Statewide Coordination Section	
TSU: Toxics Studies Unit, EAP	
WQP: Water Quality Program	

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The numbered headings in this document correspond to the headings in the original programmatic QAPP for the FFCMP (Publication 20-03-026). Only relevant sections are included in this document; therefore, some numbered headings may be missing.

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# 2.0 Abstract

Since 2001, the Freshwater Fish Contaminant Monitoring Program characterized persistent, bioaccumulative, and toxic chemicals in freshwater fish throughout Washington. In 2009, a Long-Term Monitoring component was added to determine if changes in contaminant levels occur over time.

Lake Chelan was sampled in 2003 for a total maximum daily load (TMDL) study, and in 2010 for a follow-up study. The 2021 sampling will repeat historical sampling work to see whether changes in fish tissue contaminant concentrations can be discerned.

The goals of this 2021 sampling are to: (1) measure concentrations of DDT analogs and PCBs in lake trout from Lake Chelan and compare results to the 2003 and 2010 study results. and (2) characterize concentrations of other contaminants in lake trout collected from Lake Chelan.

Results will inform resource managers about potential risks to human health from eating fish that may be contaminated and will help to evaluate progress towards goals of the Lake Chelan TMDL.

This document is an addendum to the most recent Quality Assurance Project Plan (Seiders and Sandvik, 2020) and gives information that is specific to the 2021 sampling results in Lake Chelan.

# 3.0 Background

This document is an addendum to the most recent programmatic Quality Assurance Project Plan (QAPP) (Seiders and Sandvik, 2020) for the Washington State Department of Ecology's (Ecology's) Freshwater Fish Contaminant Monitoring Program (FFCMP). This document gives specific details about the 2021 sampling in Lake Chelan and addresses only those sections in Ecology's current QAPP format where such detail is needed. For additional information, refer to the 2020 programmatic QAPP referenced above.

### 3.1 Introduction and problem statement

Past sampling of Lake Chelan fish tissue have shown elevated concentrations of the pesticide DDT (dichloro-diphenyl-trichloroethane) and its metabolites DDD (dichloro-diphenyl-dichloroethane) and DDE (dichloro-diphenyl-dichloroethylene). These three may be collectively termed DDx in this document. Other chemicals such as PBDEs (polybrominated diphenyl ethers), PCBs (polychlorinated biphenyls), and PCDD/Fs (polychlorinated dibenzo-p-dioxins and -furans) were also found in fish. These findings led to multiple 303(d) listings since 1998 in Lake Chelan for toxic chemicals in fish tissue.

To address these listings and sources of pollution, Ecology undertook several studies:

- In 2003, a Total Maximum Daily Load (TMDL) study was done for DDT and PCBs (Coots and Era-Miller, 2005).
- In 2006, a Water Quality Improvement Report (Schneider and Coots, 2006) was completed and the Washington State Department of Health issued a Fish Consumption Advisory for the lake (Health, 2006).
- In 2008, a Water Quality Implementation Plan was developed in 2008 (Anderson and Peterschmidt, 2008) and spelled out various actions that would lead to improvements in water quality. Periodic monitoring was also recommended in order to determine changes in pollution levels in fish tissue.

The local communities and organizations around Lake Chelan have also worked to address toxic contaminants and other water quality issues in the watershed. Chelan County's Natural Resources Department (NRD) is the lead entity for implementation work related to the TMDL (<u>https://www.co.chelan.wa.us/natural-resources/pages/watershed-plan-lake-chelan</u>). Watershed Planning Units and Water Quality Subcommittees comprised by local stakeholders are part of the cumulative response to water quantity and water quality concerns in Lake Chelan. This TMDL process occurs across Washington for to improve water quality (<u>https://ecology.wa.gov/Water-Shorelines/Water-quality/Water-improvement/Total-Maximum-Daily-Load-process</u>).

In 2010, fish were sampled again, and DDx concentrations in lake trout were found to be higher than levels found in 2003 (Seiders et al., 2012). The 2021 sampling will be the third round of sampling to help determine changes in concentrations of DDx and PCBs. The 2021 sampling will also characterize concentrations of other harmful chemicals including arsenic and per- and poly-fluoroalkyl substances (PFAS).

### 3.2 Study area and surroundings

The documents referenced in the preceding section provide information about the study area and surroundings. Lake Chelan is within Water Resources Inventory Area (WRIA) 47.

### 3.2.2 Summary of previous studies and existing data

There are eight known studies of Lake Chelan where fish were analyzed for chemicals. Table 1 summarizes these sampling studies by showing tissue type and number of samples, sampling years, locations, species, and target analytes. Results from the earlier studies raised concerns due to high levels of pesticides in fish and resulted in 303(d) listings. The 303(d) listings led to the 2003 DDT and PCB TMDL study (Coots and Era-Miller, 2005), the most comprehensive study to date. In 2010, only lake trout from the Wapato Basin were sampled in order to determine if DDT and PCB levels had changed since the 2003 study.

Study:	BWMP <sup>1</sup>	W	QA <sup>2</sup>	WSPMP <sup>3</sup>	WSPMP <sup>4</sup>	EPA Lakes <sup>5</sup>	ТМ	DL <sup>6,a</sup>	WSTMP <sup>7,a</sup>	WSTMP <sup>8</sup>
EIM Study ID:	BHOP0002	r	na	WSPMP92T	WSPMP94T	EPALAKES	RCO	O0004	WSTMP03T	WSTMP10
Sample Year:	1984	19	87	1992	1994	2000	20	003	2003	2010
Basin:	Wapato	Wapato	Lucerne	Wapato	Wapato	Lucerne	Wapato	Lucerne	Wapato	Wapato
Species										
BLS	f-1									
BUR		f-1	f-1				f-7	f-3 <sup>b</sup>		
CHIN			f-1							
KOK		w-1	w-1	f-1	f-1		f-7			
LKT						f-1	f-10 ª		f-32i ª	f-9
LSS		w-3	w-1	w-1	w-1	w-1				
NPM	f-1	w-2	w-2							
RBT			f-1	f-1	f-1		f-7			
SMB					f-1					
Analytes										
CPs	х	х	х	x	х	х	х	х	х	х
PCBs	A	A	А	A	A	A,C	A,C	А	A,C	А
PBDEs										х
PCDD/Fs						х	х	х		х
Mercury	х	Х	х			х			х	х
Metals	x	Х	Х			Х				
Lipids	х	Х	Х	X	Х	Х	х	х	х	Х
Other						Х				

Table 1. Species, tissue types, sample numbers, and target analytes from Lake Chelan studies that included fish tissue.

Study references: 1-Hopkins, et al., 1985; 2-Pelletier et al., 1989; 3-Davis and Johnson, 1994; 4-Davis and Serdar, 1996; 5-EPA 2009; 6-Coots and Era-Miller, 2005; 7-Seiders, 2007; 8-Seiders et al., 2012.

All samples were composites of tissue from multiple fish except those designated with "i" indicating individual fish.

a - Some samples were split with splits sent to different labs for same or different analyses; different sample IDs for the split samples were often used in EIM Study IDs RCOO0004 and WSTMP03T.

b - Some samples were split with split sent to different labs for same or different analyses.

i- Fish analyzed as individuals; f – fillet tissue; w – whole fish; for example, "f-7" indicates that 7 composite samples of fillet tissue were analyzed.

A = PCB Aroclors; C = PCB congeners.

Species codes: See Table 2.

Table 2 shows the names for fish used in past contaminant studies of Lake Chelan. The species codes are used in this addendum and in some historical reports.

Common Name	Scientific Name	Species Code
Burbot	Lota lota	BUR
Bridgelip sucker	Catostomus columbianus	BLS
Chinook salmon	Oncorhynchus tshawytscha	CHI
Cutthroat trout	Oncorhynchus clarkii	CTT
Lake trout	Salvelinus namaycush	LKT
Largescale sucker	Catostomus macrocheilus	LSS
Northern pikeminnow	Ptychocheilus oregonensis	NPM
Kokanee (Sockeye) salmon	Oncorhynchus nerka	KOK
Rainbow trout	Oncorhynchus mykiss	RBT
Smallmouth bass	Micropterus dolomieu	SMB

Table 2. Fish species from Lake Chelan analyzed for contaminants in past studies.

The 2010 study concluded that total DDx levels in lake trout remain high, and that concentrations were higher than they were in 2003. Lake trout also had high levels of PCDD/Fs and PBDE flame retardants; while levels of PCBs were mildly elevated. Table 3 summarizes the most recent results from multiple studies for DDE, PCBs, and PCDD/Fs in lake trout. More information about historical results can be found in the studies referenced in Table 1 above.

Analyte	4,4'-DDE (ug/kg)		t-PCB congeners (ug/kg)		2,3,7,8-TCDD (ng/kg)		
Year and Study	2003 TMDL	2010 WSTMP	2000 EPA Lakes	2003 TMDL, WSTMP	2000 EPA Lakes	2003 TMDL	2010 WSTMP
Number of Samples	10	9	1	20	1	3	5
Average	924.0	1262.2	32.6	25.7	0.400	0.373	0.380
Standard Deviation	324.4	446.3	-	9.7	-	0.076	0.106

Table 3. Concentrations of 4,4'-DDE, total PCBs, and dioxin in lake trout from past studies.

### 3.2.3 Parameters of interest and potential sources

The primary target analytes for long term trend assessment are DDT analogs (4,4'-DDD, -DDE, and -DDT), and PCBs. Other analytes of interest include other chlorinated pesticides, PCDD/Fs, PBDEs, and per- and poly-fluoroalkyl substances (PFAS), mercury, and arsenic.

Many of the other analytes are of interest because of associated 303(d) listings. There are listings for the chlorinated pesticides alpha-BHC, chlordanes, and dieldrin. Also listed are PCDD/Fs. Concentrations of PBDEs and mercury found in the 2010 study were elevated with some samples exceeding lower limits for the protection of human health. Perfluoroalkyl substances are of interest because of the growing recognition that these chemicals are widely dispersed in the environment (Mathieu and McCall, 2017). Arsenic is of interest because of its use as a component in lead-arsenate pesticides historically used in orchards: arsenic has also been found

in drainages leading to Lake Chelan (Patmont and Long, 2021). Potential sources for parameters of interest are given in Table 4.

Pollutant	Potential Source			
Dioxins/Furans	Contaminant in some pesticides, fires, incinerators, atmosphere			
PCBs	Electrical transformers, hydraulic fluids, caulks, atmosphere			
DDT and metabolites	Pesticides, soil erosion			
Dieldrin	Pesticides, soil erosion			
Chlordanes	Pesticides, soil erosion			
Arsenic	Pesticides, soil erosion			
Mercury	Gold mining, fossil fuels, atmosphere			
Flame retardants	Furniture, plastics in consumer products, atmosphere			
PFAS	Firefighting foams, consumer products, atmosphere			

Table 4. Pollutants and their potential sources in the Lake Chelan basin.

### 3.2.4 Regulatory criteria or standards

Washington's water quality standards and the Water Quality Assessment process are described in the programmatic QAPP for the FFCMP (Seiders and Sandvik, 2020). The most recent statewide Water Quality Assessment was approved by EPA in 2016 (Ecology, 2016a) and resulted in 17 Category 5, 4A, or 2 listings for nine toxic pollutants in Lake Chelan. (Table 5). The Category 5 listings are also known as 303(d) listings.

When a water cleanup plan (an EPA-approved TMDL) is created, Category 5 listings are reassigned as Category 4A listings. Category 4A listings indicate that full implementation of the cleanup plan is expected to result in the standards being met.

The Lake Chelan DDT and PCB TMDL Water Quality Implementation Plan (Anderson and Peterschmidt, 2008) states on page 24 that:

"the success of this TMDL will be determined by directly measuring fish tissue concentrations and comparing them to historical data and the fish tissue standards for protection of human health".

Washington's water quality standards were revised in 2016 after the TMDL Implementation Plan for Lake Chelan was developed. Ecology's Rule Implementation Plan (Ecology, 2016b) provides guidance and tools to entities involved with implementing the TMDL transition towards meeting the new water quality standards. For example, where a TMDL has been formally approved (such as for Lake Chelan), the TMDL and original water quality targets are kept in place, implementation continues as scheduled, and monitoring results are compared to targets as well as the new water quality standards. If TMDL Effectiveness Monitoring shows that the new standards are not being met, then the TMDL would be amended in order to address the new standards.

Location	Assessment Unit ID	Listing ID	WQA Category 2014	Parameter Name	Species used in basis of listing
Outlet: bridge to dam	47120I0D1	14327	5	Alpha-BHC	BLS, NPM
Manata Desin		72230	5	4,4'-DDE	LKT
(eastern grid)	47120I1G2	75066	5	4,4'-DDT	LKT
(eastern griu)		78813	5	PCBs	LKT
		14324	4A	4,4'-DDT	LKT, RBT
	47120I1G4	14325	4A	4,4'-DDD	BLS*, NPM*
Wapato Basin		14326	4A	4,4'-DDE	LKT, BUR, KOK, RBT
(western grid)		14328	4A	PCBs	LKT, BUR, KOK, RBT
		78558	2	2,3,7,8-TCDD TEQ	LKT
		78610	5	2,3,7,8-TCDD (Dioxin)	LKT
Lucama Dasin			5	Dieldrin	LKT
Lucerne Basin	47120J1D9	43061	5	Dioxin	LKT
		43078	5	Chlordane	LKT
Lucerne Basin		8963	4A	4,4'-DDE	LKT, KOK, RBT, SMB
(between Graham	491204406	8964	4A	PCBs	LKT, KOK, RBT, SMB
Harbor Cr and	40120A4G6	36426	4A	4,4'-DDT	LKT
Corral Cr)		78627	2	2,3,7,8-TCDD TEQ	BUR

Table 5. Category 5, 4A, and 2 listings for fish tissue from Lake Chelan.

\* Species shown is likely an error in WATS: BLS and NPM were not collected in this location

TMDL Effectiveness Monitoring is a fundamental component of any TMDL implementation activity. It measures to what extent the water body has improved and whether it has been brought into compliance with the state water quality standards. Effectiveness monitoring takes a comprehensive look at TMDL implementation, watershed management plan implementation, and other watershed-based cleanup work. Success may be measured against TMDL load allocations or targets, correlated with baseline conditions or desired future conditions.

While not a formal Effectiveness Monitoring project, this 2021 study can contribute to such a project which has not yet been conducted. The 2021 fish tissue monitoring will determine if the original TMDL targets (old water quality standards) for fish tissue and the new water quality standards are being met. Other elements of formal Effectiveness Monitoring, such as monitoring the implementation of Best Management Practices (BMPs) for agriculture, stormwater, and rural development are beyond the scope of this 2021 study. Ecology's guidance for formal Effectiveness Monitoring (Collyard and Onwumere, 2013) presents a strategy for monitoring the effectiveness of TMDLs and other pollution control plans.

Table 6 shows various thresholds for the protection of human health from contaminants of concern for the 2021 study. The table includes Washington's revised and previous water quality standards and the Department of Health's Screening Levels. The contaminants shown are those which are:

- Subject of the TMDL: 4,4'-DDD, -DDE, -DDT, and total PCBs.
- Reason for 303(d) listings and Categories 2 and 4 in the latest water quality assessment.
- New target analytes for this study: arsenic and PFAS.

Table 6. Thresholds used by Ecology and Health for protecting human health from contaminants in fish tissue.

Analyta	Pick	Ecolog in N	y's Threshold Iarrative Crite	Health's Screening Levels (2018)		
(ppb ww) <sup>1</sup>	Effect	TECn (2018)	10x TECc (2018)	Old FTEC (1996- 2016)	FCASL: Higher FCR	FCASL: Lower FCR
2,3,7,8-TCDD <sup>3</sup>	nc	0.32		0.065	0.280	0.821
2,3,7,8-TCDD TEQ <sup>3, 4</sup>	nc	0.32			0.280	0.821
4,4'-DDD	nc	230	19	44	1.7	4.9
4,4'-DDE	nc	230	27	32	1.2	3.4
4,4'-DDT	nc	230	13	32	200 1.2	586 3.4
Total DDT <sup>5</sup>	nc				200 1.2	586 3.4
Alpha-BHC	nc	3700	0.73			
Arsenic (inorganic)	nc	140		6.16	120 0.27	352 0.78
Chlordane <sup>6</sup>	nc	230	13	8	200 1.1	586 3.4
Dieldrin	nc	23	0.29	0.65	20 0.025	58.6 0.073
Mercury <sup>7</sup>	nc	30		770	34	101
Total PBDEs	nc				34	101
Total PCBs <sup>2</sup>	nc	9.1	2.3	5.3	8.0 0.20	23 0.59
Total PFAS <sup>8</sup>	nc				8.0	23.5

#### Key to Table 6:

Health: Washington State Department of Health.

Bold values: water quality targets per the Lake Chelan DDT and PCB TMDL Water Quality Implementation Plan. FCASL: Fish Consumption Advisory Screening Level.

FCR: Fish Consumption Rate.

FTEC: Fish Tissue Equivalent Concentration (old water quality narrative standard).

c: carcinogenic effects

nc: non-carcinogenic effects

TEC: Tissue Exposure Concentration; c=for carcinogenic effect; n=for non-carcinogenic effects.

1 - Values in parts per billion wet-weight ( $\mu g/kg$  ww) unless otherwise noted.

2 - Total PCBs is sum of Aroclors or congeners.

3 - Values in parts per trillion wet-weight (ng/kg ww).

4 - The cumulative toxicity of a mixture of congeners in a sample can be expressed as a TEQ to 2,3,7,8-TCDD. EPA (2010) states that the criterion for dioxin is expressed in terms of 2,3,7,8-TCDD and should be used in

conjunction with the international convention of Toxic Equivalency Factors (TEFs) and Toxic Equivalency (TEQs) to account for the additive effects of other dioxin-like compounds. When the TEQ is used, the toxicity of the single congener 2,3,7,8-TCDD is incorporated.

5 - Total DDT is typically the sum of the 2,4'- and 4,4'- isomers of DDD, DDE, and DDT.

DDD: 4,4'-dichlorodiphenyldichloroethane. DDE: 4,4'-dichlorodiphenyldichloroethylene. DDT: 4,4'-

dichlorodiphenyltrichloroethane. Where data for the 2,4' isomers are lacking, the sum of the 4,4'- isomers is used.

6 - The criterion for chlordane is interpreted as the sum of five chlordane components; these can be individually quantified through laboratory analyses while chlordane cannot. The EPA screening values are for "Total Chlordanes" which is the sum of five compounds: cis- and trans- chlordane, cis- and trans-nonachlor, and oxychlordane.

7 - The criterion for methylmercury is a true numeric criterion for fish tissue as opposed to a narrative criterion, which incorporates a TEC. The interpretation of tissue methylmercury results uses the TECn pathway described in Policy 1-11. Fish tissue was analyzed for total mercury, which has been deemed to adequately represent the concentration of methylmercury.

8 - Sum of PFOA and PFOS compounds. The state Department of Health considers these Screening Levels as provisional.

# **4.0 Project Description**

### 4.1 Project goals

- Characterize temporal trends for DDTs, PCBs, PCDD/Fs (dioxins), PBDEs, and mercury; and determine concentrations of PFAS and arsenic in lake trout.
- Compare results to water quality standards and screening levels for the protection of human health.
- Support fish consumption risk assessments conducted by health jurisdictions.
- Inform current and future water quality improvement work.

### 4.2 Project objectives

- Collect 50 or more lake trout of various sizes from the Wapato Basin.
- Process and analyze nine composite samples for target analytes.
- Compile and review laboratory analytical results; upload results to EIM database.
- Characterize contaminant levels found in the sampled area: evaluate temporal trends, water quality standards, and other thresholds for the protection of human health.
- Share results through various media such as reports, Ecology website, and presentations.

### 4.3 Information needed and sources

Previous studies and associated data described above were obtained from Ecology project files, EIM database, and reports from other entities. All information was reviewed to guide development of project objectives and the sampling plan. This project will use data collected through past monitoring studies conducted by Ecology and others to characterize temporal trends.

# 5.0 Organization and Schedule

### 5.1 Key individuals and their responsibilities

#### Table 7. Organization of project staff and responsibilities.

Staff All staff are with EAP	Title	Responsibilities
Jessica Archer SCS 360-407-6698	Client and SCS Manager	Reviews the project scope and budget, tracks progress, reviews the draft QAPP and addendums, and approves the same. Works with management team to help resolve issues affecting the project.
Keith Seiders Toxics Studies Unit SCS 360-407-6689	TSU 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.
Patti Sandvik Toxics Studies Unit SCS 360-407-7198	Project Assistant, Field and EIM Lead	Leads 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. Helps write report.
Jim Medlen Toxics Studies Unit SCS 360-407-6194	Unit Supervisor for the TSU Project Manager	Provides internal review of the QAPP, addendums, and reports, and approves the same. Manages budget and staffing needs. Works with management team to help resolve issues affecting the project.
Alan Rue Manchester Environmental Lab Phone: 360-871-8801	Director	Reviews and approves the final QAPP and addendums. Ensures MEL performs all chemical analyses as requested, including work contracted out. Ensures laboratory results are validated in timely manner.
Christina FransLaboratoryManchesterQualityEnvironmental LabAssurancePhone: 360-871-8801Coordinator		In addition to QA Coordinator role, leads technical aspects related to contract lab work. Develops Statements of Work, reviews labs' capabilities to meet project needs, reviews data packages from contract labs for compliance with contracts, leads data validation work (in-house or through vendor). Works with MEL's Project Coordinator and TSU Project Manager to accomplish tasks described within for contract lab data.
Nancy Rosenbower Manchester Environmental Lab Phone: 360-871-8801	Laboratory Project Coordinator	Coordinates communication between MEL staff and TSU Project Manager. Conducts sample receipt, tracking, storage, shipment to other labs. Disseminates labs result reports. Works with MEL QA Coordinator and TSU Project Manager to accomplish tasks described within for contract lab data.
<b>Arati Kaza</b> EAP Manager's Unit Phone: 360-407-6964	Ecology Quality Assurance Officer	Reviews and approves the draft QAPP and the final QAPP and addendums. Ensures EAP adheres to QC-related SOPs and practices.

See Notes on next page.

#### Notes for Table 7

EAP: Environmental Assessment Program EIM: Environmental Information Management database QAPP: Quality Assurance Project Plan MEL: Manchester Environmental Laboratory SCS: Statewide Coordination Section TSU: Toxics Studies Unit

This 2001 FFCMP study on Lake Chelan requires the use of contract labs because Manchester Environmental Laboratory (MEL) is not equipped to conduct all of the needed analyses. The process for obtaining contract lab services involves varied staff having different expertise and roles. In order to help communication among all involved with this process, the checklist in Appendix A was developed. The Project Manager will use this checklist to track the process.

### 5.3 Organization chart

Table 0	Ouganizations	that may h	a invialuad	with the	FECMD.	2021 :m	Lake Chalan
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Organization	Role	Persons
Ecology WQP HQ	Review draft report and provide guidance on interpretation of water quality standards	Melissa Gildersleeve, Chad Brown, Benjamin Rau
Ecology WQP CRO	Support coordination with Lake Chelan Watershed Planning Unit and other local interests involved with the Lake Chelan TMDL for DDTs and PCBs.	Mark Peterschmidt, Lloyd Stevens (CRO)
Ecology CRO	Regional EAP staff: liaison with regional staff, field support	George Onwumere (CRO)
Ecology MEL	Analytical services at MEL and contract labs	Alan Rue, Christina Frans, Nancy Rosenbower, John Weakland, Heidi Chuhran
WDFW HQ	Fish Age Lab: fish age determination	Andrew Claiborne
WDFW HQ	Scientific Collection Permits	Bruce Baker, others at ScientificCollection.Permits @dfw.wa.gov
WDFW District	Fish Program Biologist: local knowledge, possible collaboration	Graham Simon
NOAA	Scientific Collection Permits	Claire McGrath, Mitch Dennis
WDOH and Chelan-Douglas Health District	Uses FFCMP data to conduct risk assessments for Fish Consumption Advisories	Dave McBride
Lake Chelan Watershed Planning Unit	Outreach and coordination with watershed community about water quantity and quality related to Lake Chelan.	Mike Kaputa, Lisa Dowling
Local Government	Local governments: counties, cities, PUDs, special districts: permissions to sample as needed	Not specified

CRO: Central Regional Office

EAP: Environmental Assessment Program

ERO: Eastern Regional Office

HQ: Headquarters

MEL: Manchester Environmental Laboratory

NOAA: National Oceanic and Atmospheric Administration

WDFW: Washington Department of Fish and Wildlife

WDOH: Washington Department of Health

WQP: Water Quality Program

### Proposed project schedule

Table 9. Proposed schedule for completing field and laboratory work, data entry in	to EIM,
and reports.	

Field and laboratory work	Due date	Lead staff		
Field work (varies annually, depends on site characteristics)	Jun 2021	Patti Sandvik		
Sample processing	Sep 2021	Patti Sandvik		
Lab analyses and data validation completed (varies, depends on time of sample delivery and lab capacity)	Jan to Dec 2022	Alan Rue, Christina Frans, Nancy Rosenbower		
Environmental Information System (EIM	) database			
EIM Study ID	FFCMP21			
Product	Due date	Lead staff		
EIM data loaded <sup>1</sup>	Mar 2023	Patti Sandvik		
EIM data entry review <sup>2</sup>	Apr 2023	Varies by year		
EIM complete <sup>3</sup>	May 2023	Patti Sandvik		
Final Report for 2021 Study				
Author lead / Support staff	Keith Seiders / Pa	tti Sandvik		
Schedule (variable – dependent on resource	ces)			
Draft due to supervisor	Jun 2023			
Draft due to client/peer reviewer	Jul 2023			
Draft due to external reviewer(s)	Sep 2023			
Final (all reviews done) due to publications coordinator	Nov 2023			
Final report due on web	Mar 2024 to Sep 2	2024		
QAPP Addendum for 2021 Sampling				
Author lead / Support staff	Keith Seiders / Pa	tti Sandvik		
Schedule:				
Draft due to supervisor	May 2021			
Draft due to client/peer reviewer	May 2021			
Draft due to external reviewer(s)	Jun 2021			
Final (all reviews done) due to publications team	Aug 2021			
Final QAPP Addendum due on web	Jan 2022 to Oct 2	022		

<sup>1</sup> All data entered into EIM by the lead person for this task.

<sup>2</sup> Data verified to be entered correctly by a different person; any data entry issues identified. Allow one month. <sup>3</sup> All data entry issues identified in the previous step are fixed (usually by the original entry person); EIM Data Entry Review Form signed off and submitted to EIM coordinator (Melissa Petersen, who then enters the "EIM Completed" date into Activity Tracker; allow one month for this step). The final EIM completion date is usually targeted to be no later than the final report publication date. Table 10 shows estimated analytical costs based on the sampling plan with sites, target species, target number of analyses of composite samples for each suite of analyses.

Analyte group	Total # field sample analyses	Total # lab QC analyses	Total # analyses	Cost per analysis (\$)	Subtotal: Field + QC Cost (\$)	MEL contracting surcharge (\$)	Subtotal: all costs (\$)
Fish Age*	50	-	50	30	1,500*	-	1,500*
Mercury	14	4	18	50	900	-	900
Lipid	14	3	17	35	595	-	595
3 DDx, 3 PCB Aroclors	14	3	17	300	5,100	-	5,100
PBDE	14	3	17	240	4,080	-	4,080
PFAS	14	4	18	800	14,400	-	14,400
PCB congeners	14	2	16	895	14,320	4,296	18,616
PCDD/Fs	14	2	16	705	11,280	3,384	14,664
Chlor Pest	14	2	16	900	14,400	4,320	18,720
Arsenic	14	4	18	306	5,508	1,652	7,160
	70,583	13,652	84,235				

Table 10. Estimated laboratory costs, FFCMP 2021.

\* The costs for fish age are excluded from laboratory cost totals because fish aging services are purchased separately from analyses for toxic chemicals in fish tissue.

# 6.0 Quality Objectives

### 6.1 Data quality objectives

The data quality objective for this project is to obtain data of sufficient quantity and quality for use in comparisons to results from previous and future studies and thresholds for the protection of human health. This objective will be achieved through attention to sample design, sample collection and processing, laboratory measurement of target analytes, collection and review of historical data, data management, and quality control (QC) procedures described or referenced in this plan.

### 6.2 Measurement quality objectives

The measurement quality objectives (MQOs) for calibration verification, ongoing precision and recovery, and labeled compound recovery correspond to the QC acceptance limits of the analytical methods. Even though fish tissue is a challenging matrix for organics analyses and subject to interferences due to lipids and other compounds, certain lab practices (e.g., sample preparation and cleanup) allow MQOs to be achieved most of the time.

These MQOs correspond to MEL's QC limits (metals and ancillary parameters) or the acceptance limits specified in the analytical methods (organic compounds). The lowest concentrations of interest shown in the tables below are currently attainable by MEL and contract laboratories, in most cases. MEL and contract labs are expected to meet the MQOs in Table 11. Results not meeting these MQOs will be evaluated for possible corrective action or use with qualification.

For most analytes, the designated method's achievable reporting limits (RL) will be adequate for this project. For organics, MEL will continue the current practice of reporting results down to their in-house DL (detection limit) and qualify results between the DL and PQL (practical quantitation limit) or EQL (estimated quantitation limit) as estimates. For PCDD/Fs, contract labs will be required to report down to their in-house DL for all congeners and qualify results between the DL and PQL or EQL as estimates. These reporting practices improve the ability to compare results to thresholds for the protection of human health and aquatic life.

### 6.2.1 Targets for precision, bias, and sensitivity

The MQOs for laboratory analyses are expressed in terms of acceptable precision, bias, and sensitivity for each analytical method in Table 11. Tables 12-14 expand on the sensitivity for individual analytes within a suite of analytes. These MQOs are then briefly discussed. Laboratory Case Narratives will discuss the outcomes of QC practices and address these MQOs for each batch of sample analyses.

Analytical		Precision (RPD)		E	Bias (% recove	Sensitivity	
Parameter	Method	Lab Duplicate	Matrix Spike Duplicate	Lab Control Sample	Surrogate <sup>a</sup>	Matrix Spike <sup>a</sup>	Reporting Limits <sup>b</sup>
Mercury	EPA 245.6 (CVAA)	0%-20% (for results > 5x RL)	0%-20%	85%-115%	NA	75%-125%	17 ug/kg
Arsenic species	EPA 1632A or equiv	0%-20% (for results > 5x RL)	0%-20%	85%-115%	NA	75%-125%	0.05 - 0.10 ug/g (wet wt)
4,4'-DDD, -DDE, - DDT (low resolution)	EPA 8081 (GC/ECD); MEL SOP 730002	0%-40%	0%-40%	50%-150%	20%-120%, 30%-130	50%-150%	most 0.5-3.0 ug/kg °
Chlorinated pesticides (high resolution)	EPA 1669 (HR GC/MS) or equivalent	0%-40%	NA	g	NA	NA	0.01-0.10 ug/kg <sup>c</sup>
PCB Aroclors 1248, 1254, 1260 (low resolution)	EPA 8082 (GC/ECD); MEL SOP 730002	0%-40%	0%-40%	50%-150%	50%-150%	50%-150%	1.1 - 10 ug/kg <sup>d</sup>
PCB congeners (high resolution)	EPA 1668C (HR GC/MS)	0%-40%	NA	g	NA	NA	0.003-0.01 ug/kg
PCDD/Fs (high resolution)	EPA 1613B (HR GC/MS)	0%-40%	NA	g	NA	NA	EQL 0.03 - 0.5 ng/kg
PBDEs	EPA 8270 (SIM); SOP 730104	0%-40%	NA	50%-150%	50%-150%	50%-150%	0.10-2.6 ug/kg; PBDE 209 1.9-4.3 ug/kg
PFAS	EPA 8327 Modified (LC-MS/MS with isotopic dilution; MEL SOP 730133)	0%-40%	0%-40%	50%-150%	20%-200%	50%-150%, 40%-160%	0.5-2.50 ug/kg <sup>f</sup>
Lipids	MEL SOP 730009	0%-20%	0%-40%	NA	NA	NA	0.10%

#### Table 11. Measurement quality objectives by analyte and method.

a - Different ranges of limits can be specific to the surrogate used or to different target analytes.

b - Value reflects typical range.

c - See Table 12 for analyte-specific RLs for chlorinated pesticides by different methods.

d - Typical RL; yet interferences may drive the RL higher.

e - See table 13 for analyte-specific RLs for PCDD/Fs.

f - See table 14 for analyte-specific RLs for PFAS.

g - Per method for Ongoing Precision and Recovery (OPR), Internal Standards, and Labelled Compounds.

NA - Not applicable.

Analyte	CAS #	RL for low-res (EPA 8081) <sup>a</sup>	EDL for Hi-res (EPA 1699 or similar)	EQL for Hi-res (EPA 1699 or similar)	Expected range of results
2,4'-DDD	53-19-0	0.5 - 1.0	0.02	0.2	ND - 5
2,4'-DDE	3424-82-6	0.5 - 1.0	0.02	0.2	ND – 10
2,4'-DDT	789-02-6	0.5 - 1.0	0.02	0.2	ND - 15
4,4'-DDD	72-54-8	0.5 - 1.0	0.02	0.2	5 - 40
4,4'-DDE	72-55-9	0.5 - 1.0	0.02	0.2	200 - 2000
4,4'-DDT	50-29-3	0.5 - 1.0	0.02	0.2	10 - 100
Aldrin	309-00-2	0.5 - 2.0	0.02	0.4	ND - 3.0
alpha-BHC (alpha-HCH)	319-84-6	0.5 - 2.0	0.02	0.4	ND – 2.0
beta-BHC (beta-HCH)	319-85-7	0.5 - 1.0	0.02	0.4	ND - 2.0
Chlordane, total (sum of 5 addends)	-	0.4 <sup>c</sup> - 1.0 <sup>c</sup>	0.02 °	0.4 °	ND - 10
Chlorpyriphos	2921-88-2	0.25 - 0.5	0.02	0.4	ND - 10
Chlorthal-dimethyl (Dacthal)	1861-32-1	0.25 - 0.5	0.02	0.2	ND - 5.0
cis-Chlordane (alpha-Chlordane) <sup>b</sup>	5103-71-9	0.5 - 1.0	0.02	0.4	ND - 5
cis-Nonachlor <sup>b</sup>	5103-73-1	0.5 - 1.0	0.02	0.4	ND - 5
DDMU	1022-22-6	0.25 - 0.5	nt	nt	ND - 15
delta-BHC (delta-HCH)	319-86-8	0.5 - 1.0	0.05	0.20	ND - 2.0
Dieldrin	60-57-1	0.5 - 2.0	0.05	0.16	ND - 5.0
Endosulfan I	959-98-8	1.0 - 2.0	0.05	0.16	ND - 5.0
Endosulfan II	33213-65-9	1.0 - 2.0	0.05	0.16	ND - 5.0
Endosulfan Sulfate	1031-07-8	1.0 - 2.0	0.05	0.16	ND - 5.0
Endrin	72-20-8	1.0 - 2.0	0.05	0.16	ND - 5.0
Endrin Aldehyde	7421-93-4	1.0 - 2.0	0.05	0.16	ND - 5.0
Endrin Ketone	53494-70-5	0.5 - 1.0	0.05	0.16	ND - 5.0
Heptachlor	76-44-8	0.5 - 2.0	0.02	0.2	ND - 5.0
Heptachlor Epoxide	1024-57-3	0.5 - 2.0	0.05	0.16	ND - 5.0
Hexachlorobenzene	118-74-1	0.5 - 1.0	0.01	0.2	ND - 5.0
Lindane (gamma-BHC, -HCH)	58-89-9	0.5 - 1.0	0.02	0.4	ND - 5.0
Methoxychlor	72-43-5	0.5 - 1.0	0.10	0.16	ND - 5.0
Mirex	2385-85-5	0.5 - 2.0	0.02	0.2	ND - 5.0
Oxychlordane <sup>b</sup>	27304-13-8	0.5 - 1.0	0.02	0.4	ND – 5.0

Table 12. Reporting limits for chlorinated pesticide analyses by different methods and expected range of results for fish tissue (ug/kg).

Analyte	CAS #	RL for low-res (EPA 8081) <sup>a</sup>	EDL for Hi-res (EPA 1699 or similar)	EQL for Hi-res (EPA 1699 or similar)	Expected range of results
Pentachloroanisole	1825-21-4	0.25 - 0.5	nt	nt	ND - 5.0
Toxaphene <sup>d</sup>	8001-35-2	2.0 - 10	0.10	0.4	ND – 5.0
trans-Chlordane (gamma-Chlordane) <sup>b</sup>	5103-74-2	0.5 - 1.0	0.02	0.4	ND – 5.0
trans-Nonachlor <sup>b</sup>	39765-80-5	0.5 - 1.0	0.02	0.4	ND – 5.0

Analytes in **bold** are the more commonly detected pesticides of concern.

a = Typical RL for past FFCMP, extract split).

b = One of five addends used for determining "Chlordane, total".

c = As the sum of five addends.

d = While not a target analyte of EPA 1699, HR GCMS can be used to quantify major components of this analyte.

nt = Not listed as a target analyte.

Congener	CAS Number	Quantitation Limit (ng/kg)	Detection Limit (ng/kg)	TEF (WHO 2005)
2,3,7,8-TCDD	1746-01-6	0.03	0.013	1
1,2,3,7,8-PeCDD	40321-76-4	0.03	0.022	1
1,2,3,4,7,8-HxCDD	39227-28-6	0.1	0.018	0.1
1,2,3,6,7,8-HxCDD	57653-85-7	0.1	0.019	0.1
1,2,3,7,8,9-HxCDD	19408-74-3	0.1	0.019	0.1
1,2,3,4,6,7,8-HpCDD	35822-46-9	0.2	0.034	0.01
OCDD	3268-87-9	0.5	0.034	0.0003
2,3,7,8-TCDF	51207-31-9	0.05	0.019	0.1
1,2,3,7,8-PeCDF	57117-41-6	0.1	0.023	0.03
2,3,4,7,8-PeCDF	57117-31-4	0.05	0.019	0.3
1,2,3,4,7,8-HxCDF	70648-26-9	0.1	0.024	0.1
1,2,3,6,7,8-HxCDF	57117-44-9	0.1	0.023	0.1
1,2,3,7,8,9-HxCDF	72918-21-9	0.1	0.031	0.1
2,3,4,6,7,8-HxCDF	60851-34-5	0.1	0.025	0.1
1,2,3,4,6,7,8-HpCDF	67562-39-4	0.2	0.008	0.01
1,2,3,4,7,8,9-HpCDF	55673-89-7	0.2	0.012	0.01
OCDF	39001-02-0	0.5	0.042	0.0003

Table 13. Quantitation and detection limits, and TEFs, for PCDD/F congeners.

Analyte	LLOQ	MDL
Analyte	(ug/kg)	(ug/kg)
Perfluorobutanoic acid	0.500	0.238
Perfluoropentanoic acid	0.500	0.126
Perfluorobutanesulfonic acid	0.500	0.0211
4:2 Fluorotelomer sulfonic acid	2.50	
Perfluorohexanoic acid	0.500	0.0798
Perfluorohexanesulfonic acid	0.500	0.152
Perfluoroheptanoic acid	0.500	0.0861
Perfluorooctanoic acid	0.500	0.0602
Perfluorooctanesulfonic acid	0.500	0.0638
Perfluoropentanesulfonic acid	0.500	0.112
6:2 Fluorotelomer sulfonic acid	2.50	
Perfluorononanoic acid	0.500	0.0483
Perfluorodecanoic acid	0.500	0.0370
N-methyl perfluorooctanesulfonamidoacetic acid	0.500	0.0608
N-ethyl perfluorooctanesulfonamidoacetic acid	0.500	0.0632
Perfluoroheptanesulfonic acid	0.500	0.171
Perfluoroundecanoic acid	0.500	0.0248
8:2 Fluorotelomer sulfonic acid	2.50	
Perfluorododecanoic acid	0.500	0.0294
Perfluorotridecanoic acid	0.500	0.0214
Perfluorooctanesulfonamide	0.500	
Perfluorotetradecanoic acid	0.500	0.0394
Perfluorononanesulfonic acid	0.500	0.0884
Perfluorodecanesulfonic acid	0.500	0.0862

#### Table 14. Quantitation and detection limits for PFAS.

LLOQ: Lower Limit of Quantitation

MDL: Method Detection Limit

# 7.0 Study Design

### 7.1 Study boundaries

The study boundary is Lake Chelan's southernmost area known as the Wapato Basin. Figure 1 shows the Wapato Basin and the likely fish collection areas: these are the same as in the 2010 study.



Figure 1. Areas in the Wapato Basin of Lake Chelan where fish may be collected in 2021.

### 7.2 Field data collection

### 7.2.1 Sampling strategy, frequency, and locations

#### Strategy

The selection of sampling location, species, fish size, and tissue type for the 2021 study was determined mostly by historical sampling, particularly the 2010 and 2003 studies. Lake trout were chosen because they:

- Exhibit high concentrations of multiple contaminants from which declines in concentration should be measureable.
- Are the subject of a Fish Consumption Advisory.
- Are available in adequate numbers and desirable size ranges; therefore, they can be collected with reasonable level of effort.

While fillet tissue will be used for characterizing contaminant concentrations and temporal trends, the remaining carcasses of fish will also be analyzed. Together, the matched results from

carcass and fillet analyses can be used to estimate whole body concentrations of target analytes which can yield information about the recycling of target contaminants within the Lake Chelan system. The carcass tissue may also have higher concentrations of some analytes, such as arsenic, which allows for more accurate measurement.

#### Sample Size

Estimates of sample sizes needed to detect given changes in mean concentrations were conducted for key analytes. A series of calculations were made using the 2010 sample variances and different Minimum Detectable Changes (MDCs) to estimate sample sizes. Results from these estimates were plotted to show the sample sizes needed for given MDCs. For these cases, we set the significance level (alpha) to 0.05 and power (B-1) to 0.8.

Figure 2 shows the plots for DDE and PCBs in lake trout from Lake Chelan. The curve for DDE shows that a sample size of 9 should be adequate for a MDC of 631 ug/kg, which corresponds to a 50% change from the 2010 mean value of 1262 ug/kg. For t-PCBs, 11 samples should provide a MDC of 18 ug/kg, which corresponds to a nearly 50% change in the 2010 mean value of 35.5 ug/kg.



Figure 2. Sample size estimates and MDCs for DDE and PCBs in lake trout.

Plots for other analytes showed that a sample size of 9 (for fillet samples) would yield acceptable sensitivity (as MDCs) for trend detection given the costs for sampling and laboratory analyses. These other analytes and MDCs are: mercury - 25%; t-PBDEs - 58%; TCDD-TEQ - 40%; and 2,3,7,8-TCDD - 40%.

For 2021, a sample size of 9 composite samples of fillet tissue for all analytes should produce the data needed to detect trends in key analytes as well as produce a robust data set for other analytes for future comparisons. For carcass tissue, a sample size of 5 should be adequate for initial characterization of this sample media. Each composite sample will be formed using five individual fish of similar size. For five sets of composite samples, the same five fish that are used in forming a single composite of sample fillet tissue will also be used to form a corresponding sample of carcass tissue. Table 15 shows the historical and target fish size range for lake trout from the Wapato Basin. The target number of individual fish is based on using 5 individual fish per composite sample. If desired numbers of fish are low, 3 fish per composite sample could be acceptable.

Sample grouping	Target #	We	ight	Total Length		
Sample grouping	of fish	grams	pounds	mm	inches	
Small size: 5 samples	25	445-1390	1.0-3.1	381-546	15.0-21.5	
Medium size: 3 samples	15	1392-1803	3.1-4.0	533-603	21.0-23.8	
Large size: 1 sample	5	2071-2212	4.6-4.9	578-603	22.8-23.8	

 Table 15. Target numbers and size ranges for Lake Chelan lake trout, FFCMP 2021.

# **8.0 Field Procedures**

### 8.4 Equipment Decontamination

Decontamination procedures for this year's FFCMP will be slightly modified in order to accommodate the addition of PFAS analytes. The modification substitutes methanol for acetone and hexane for the final solvent rinses of equipment used to process samples. The original decontamination procedure is documented in Standard Operating Procedure EAP007, *Resecting Finfish Whole Body, Body Parts, or Tissue Samples* (Sandvik, 2018) and Ecology's Chemical Hygiene Plan (Ecology, 2019). The modified practice is found in Standard Operating Procedure EAP090, *Decontamination of Sampling Equipment for Use in Collecting Toxic Chemical Samples* (Friese, 2014).

# 9.0 Laboratory Procedures

Multiple laboratories will be used to analyze samples collected from Lake Chelan. Many analyses will be performed by MEL whereas contract labs will conduct analyses for arsenic speciation, PCB congeners, PCDD/Fs, and chlorinated pesticides using High Resolution Mass Spectrometry (HRMS).

The process for obtaining contract lab services involves varied staff having different expertise and roles. In order to help communication among all involved with this process, the checklist in Appendix A was developed. The Project Manager will track the progress of the process by using the checklist.

### 9.1 Lab procedures table

Parameter	Analysis Frequency, Number of Samples, Arrival Date	Expected Range of Results	Reporting Limits	Analytical Method	Sample Preparation Method
Mercury	n=14, Oct 2021	10 - 500 ug/kg	17 ug/kg	EPA 245.6 (CVAA)	EPA 245.6
Arsenic: MMA, DMA, InorgAs	n=14, Oct 2021	Likely non- detect to low (up to 5x RL)	0.05-0.10 ug/kg (wet wt)	EPA 1632A or equivalent	per method
4,4'-DDD, -DDE, - DDT ª (low resolution)	n=14, Oct 2021	10 - 2000 ug/kg	most 0.5- 3.0 ug/kg	EPA 8081B (GC/ECD) MEL SOP	Prep: EPA 3541 Modified, Cleanup: EPA 3620C/3665A, EPA 60014-81-045
Chlorinated pesticides <sup>a</sup> (high resolution)	n=14, Oct 2021	0.01 - 20 ug/kg for most; DDx 10-2000 ug/kg	0.01-0.10 ug/kg	EPA 1669 (HR GC/MS) or equivalent	EPA 1669 or equivalent lab SOPs
PCB Aroclors: 1248, 1254, 1260 (low resolution)	n=14, Oct 2021	1.0 - 50 ug/kg	1.0 - 10 ug/kg	EPA 8082 (GC/ECD) MEL SOP	Prep: EPA 3541 Modified, Cleanup: EPA 3620C/3665A, EPA 60014-81-045
PCB congeners (high resolution)	n=14, Oct 2021	0.005 - 100 ug/kg, depending on congener	0.003-0.01 ug/kg	EPA 1668C (HR GC/MS)	EPA 1668C, lab SOPs
PCDD/Fs (high resolution)	n=14, Oct 2021	0.005 - 5.0 ng/kg, depending on congener	0.017 - 0.5 ng/kg <sup>b</sup>	EPA 1613B (HR GC/MS)	EPA 1613B, lab SOPs
PBDEs	n=14, Oct 2021	0.1 - 50 ug/kg	0.10-2.6 ug/kg; BDE 209 1.9-4.3 ug/kg	EPA 8270E (SIM) SOP 730104	Prep: EPA 3541 Modified, Cleanup: EPA 3620C Modified, EPA 3665A Modified
PFAS	n=14, Oct 2021	Likely non- detect to low (up to 5x RL)	0.5 - 2.5 ug/kg	EPA 8327 modified (LC- MS/MS, isotopic dilution)	QuEChERS MEL SOP 730124
Lipids	n=14, Oct 2021	0.1 - 20 %	0.10%	MEL SOP 730009	EPA 3541 Modified

Table 16. Measurem	ent methods	(laboratory).
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a - See Table 12 for analyte-specific RLs and expected range of results for chlorinated pesticides.

b - See Table 13 for analyte-specific RLs for PCDD/Fs.

# **10.0 Quality Control Procedures**

### **10.1 Table of field and laboratory quality control**

Table 17.	Laboratory	quality	control	sample	types	and fr	eauencies.
I abic I /.	Laboratory	quanty	control	sampic	ypus	and m	equencies.

Parameter	Analytical Method	Lab Duplicates	Lab Control Standards	Surrogates	MS/ MSD	Method Blanks
Mercury	EPA 245.6 (CVAA)	1/ batch ª	1/batch	NA	1/batch	1/batch
Arsenic: MMA, DMA, InorgAs	EPA 1632A or equiv	1/batch	1/batch	NA	1/batch	1/batch
4,4'-DDD, -DDE, -DDT <sup>b, e</sup> (low resolution)	EPA 8081 (GC/ECD), MEL SOP	1/batch	1/batch	each sample	1/batch	1/batch
Chlorinated pesticides <sup>c</sup>	EPA 1699 or equivalent (HR GC/MS)	1/batch	each sample & 1/batch <sup>b</sup>	NA	NA	1/batch
PCB Aroclors <sup>e</sup> 1248, 1254, 1260	EPA 8082 (GC/ECD), MEL SOP	1/batch	1/batch	each sample	1/batch	1/batch
PCB congeners <sup>c</sup>	EPA 1668C (HR GC/MS)	1/batch	each sample & 1/batch <sup>b</sup>	NA	NA	1/batch
PCDD/Fs °	EPA 1613B (HR GC/MS)	1/batch	each sample & 1/batch <sup>b</sup>	NA	NA	1/batch
PBDEs °	EPA 8270 (SIM), MEL SOP 730104	1/batch	1/batch	each sample	1/batch	1/batch
PFAS <sup>d</sup>	EPA 8327 modified (LC-MS/MS with isotopic dilution)	1/batch	1/batch	each sample	1/batch	1/batch
Lipids	MEL SOP 730009	1/batch	1/batch	NA	NA	1/batch

a - Batch is defined as up to 20 samples analyzed together.

b – Labeled compounds in each sample and Ongoing Precision and Recovery standards in each batch.

c – Certified Reference Material "CARP-2" from National Research Council Canada to be analyzed once per sample delivery group.

d- Standard Reference Material (SRM) 1947 from National Institute for Standards and Technology (NIST) to be analyzed once per batch.

e – No SRM to be analyzed.

# **11.0 Data Management Procedures**

### **11.2 Laboratory data package requirements**

Laboratory results from MEL analyses will be sent to the Project Manager in printed format (from LIMS) and be accompanied by a Case Narrative. The Case Narrative will address various data verification and validation checks described in Section 13 below.

Results from contract laboratories will be delivered to MEL. These results will contain information specified in one of two documents, depending on how contract labs are selected. For labs that are already on the State Master Contract List, one document called a Statement of Work (SOW) will describe the project needs for analysis and reporting. For other labs, the same SOW can be used. If a bid process is needed, the SOW will be incorporated into a document called a Request for Quotes (RFQ).

For work conducted by contract labs, a MEL-designated expert will review the Level 4 data package from the contract lab and summarize findings in a Case Narrative similar to that for MEL-generated data. The Level 4 data package, as well as the Electronic Data Deliverable (EDD) specifications, are described in Section 11.3 below.

### **11.3 Electronic transfer requirements**

MEL staff will enter lab data generated by MEL into the Laboratory Information System (LIMS). When notified of the availability of data, project staff can then access LIMS data and receive the data in an Excel file formatted similar to the EIM loading template.

Results from contract labs will be provided in Excel-compatible (e.g., .csv) format for ease of review, validation, editing, and transfer into EIM. At a minimum, the EDD must contain the information shown in Table 18. Some items in Table 18 could be provided in the lab's Case Narrative instead of within the EDD format. For example, items 2, 3, and 4 could be provided in a separate table (sometimes called a Sample Correlation Table). Another example is for items 7, 8, 9, and 16: these items will be common to all samples, so do not necessarily need to be in the EDD as long as the Case Narrative contain this information. Other items may be included in the EDD and Case Narrative as needed to help reviewers understand the data package and have the information needed for validation.

When the verification/validation process for contract lab data results in changes to qualifiers and reported values, the person conducting the verification/validation will (1) create three new fields in the EDD and (2) enter the amended values, along with the reason for the change (as in items #27-29 in Table 18, italicized for emphasis).

For PFAS compounds, there are different reporting conventions among laboratories and methods. To help promote more consistent nomenclature, the Interstate Technology Regulatory Council (2020) addresses naming conventions for individual and groups of PFAS compounds.

Ref #	Field Name	Example Value
1	Study ID (Project Name provided to contract lab)	FFCMP 2018
2	Field Station Identification (Ecology Field ID provided to contract lab)	STA5-CCC
3	Contract Lab Sample ID	L180327-5
4	MEL Work Order Sample ID (Ecology Sample ID provided to contract lab)	1803015-01
5	Field Collection Date (listed in COC)	10/25/2018
6	Date of Receipt at Contract Lab	3/15/2019
7	Sample Matrix (provided to contract lab)	Tissue
8	Sample Preparation Method	1668C
9	Analysis Method	1668C
10	Parameter Name (the 7-character format for PCBs is required)	PCB-001
11	CAS Number	2051-60-7
12	Sample Extraction Date	3/30/2018
13	Analysis Date	4/10/2018
14	Analysis Time	12:22
15	Lab Batch ID (to associate results with QC samples)	L80882
16	Contract Lab Name	MegaMSLab
17	Result Value	0.743
18	Result Value Units	ng/g
19	Result Reporting Limit	4.33
20	Result Reporting Limit Type (e.g., LOQ/MRL)	LOQ
21	Result Detection Limit	0.743
22	Result Detection Limit Type (e.g., EDL/CRDL/MDL)	EDL
23	Result Value Qualifier	UJ
24	Result Basis (Wet/Dry)	Wet
25	Lab Duplicate (Y/N)	N
26	Lab Reanalysis (Y/N)	Ν
27	Amended Result Value (entered by data reviewer)	0.743
28	Amended Result Value Qualifier (entered by data reviewer)	U
29	Reason for Amendment(s) (entered by data reviewer)	Blank contamination

Table 18. Required fields for electronic data deliverables from contract labs.

For PFAS compounds, there are different reporting conventions among laboratories and methods. To help promote more consistent nomenclature, the Interstate Technology Regulatory Council (2020) addresses naming conventions for individual and groups of PFAS compounds. The analytical method for PFAS used by MEL (SW 8327, modified) actually measures the anionic form of PFAS compounds: it is the anionic form that is present in the environment.

However, MEL reports results for the acid form of the compounds because the EPA Method 8327 publication lists the acids (not the anions); and the analytical standards come in the acid form. Because the anionic form is what is actually measured, this project will report the anionic form to EIM. Table 19 shows the corresponding anionic and acid forms for 35 PFAS compounds.

Abbreviation	Name	CAS#
DEDA	Perfluorobutanoate	45048-62-2
FFDA	Perfluorobutyric acid	375-22-4
DEDoA	Perfluoropentanoate	45167-47-3
FFFEA	Perfluoropentanoic acid	2706-90-3
	Perfluorohexanoate	92612-52-7
FFRXA	Perfluorohexanoic acid	307-24-4
DEUpA	Perfluoroheptanoate	120885-29-2
егпрА	Perfluoroheptanoic acid	375-85-9
DEOA	Perfluorooctanoate	45285-51-6
PFUA	Perfluorooctanoic acid	335-67-1
	Perfluorononanoate	72007-68-2
PFINA	Perfluorononanoic acid	375-95-1
	Perfluorodecanoate	73829-36-4
PFDA	Perfluorodecanoic acid	335-76-2
	Perfluoroundecanoate	196859-54-8
PFUNA	Perfluoroundecanoic acid	2058-94-8
DEDoA	Perfluorododecanoate	171978-95-3
FFDOA	Perfluorododecanoic acid	307-55-1
	Perfluorotridecanoate	862374-87-6
PEIIDA	Perfluorotridecanoic acid	72629-94-8
	Perfluorotetradecanoate	365971-87-5
PFIEDA	Perfluorotetradecanoic acid	376-06-7
DEBS	Perfluorobutanesulfonate	45187-15-3
PFDS	Perfluorobutanesulfonic acid	375-73-5
	Perfluoropentanesulfonate	175905-36-9
PFPeo	Perfluoropentanesulfonic acid	2706-91-4
DEHVO	Perfluorohexanesulfonate	108427-53-8
	Perfluorohexanesulfonic acid	355-46-4

Table 19. PFAS abbreviations with corresponding anionic and acid forms.

Abbreviation	Name	CAS#
DELLas	Perfluoroheptanesulfonate	146689-46-5
РЕпро	Perfluoroheptanesulfonic acid	375-92-8
DEOS	Perfluorooctanesulfonate	45298-90-6
PF03	Perfluorooctanesulfonic acid	1763-23-1
DENS	Perfluorononanesulfonate	474511-07-4
FFINO	Perfluorononanesulfonic acid	68259-12-1
DEDS	Perfluorodecanesulfonate	126105-34-8
FFD3	Perfluorodecanesulfonic acid	335-77-3
DEDos	Perfluorododecanesulfonate	343629-43-6
PFD05	Perfluorododecanesulfonic acid	79780-39-5
4.2 ETS	4:2 fluorotelomersulfonate	414911-30-1
4.2 F 13	4:2 fluorotelomersulfonic acid	757124-72-4
	6:2 fluorotelomersulfonate	425670-75-3
0.2 F13	6:2 fluorotelomersulfonic acid	27619-97-2
0.0 ГТС	8:2 fluorotelomersulfonate	481071-78-7
0.2 F13	8:2 fluorotelomersulfonic acid	39108-34-4
	3:3 perfluorohexanoate	1169706-83-5
3.3 FTCA	3:3 perfluorohexanoic acid	356-02-5
	5:3 perfluoroctanoate	1799325-94-2
5.3 FTCA	5:3 perfluorooctanoic acid	914637-49-3
	7:3 perfluorodecanoate	1799325-95-3
7.3 FICA	7:3 perfluorodecanoic acid	812-70-4
	N-Methylperfluorooctanesulfonamidoacetate	n.a.
IN-INIEFOSAA	N-Methylperfluorooctanesulfonamidoacetic acid	2355-31-9
	N-Ethylperfluorooctanesulfonamidoacetate	n.a.
N-EIFUSAA	N-Ethylperfluorooctanesulfonamidoacetic acid	2991-50-6
	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3- heptafluoropropoxy)propanoate	122499-17-6
HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3- heptafluoropropoxy)propanoic acid	13252-13-6
	Dodecafluoro-3H-4,8-dioxanonanoate	2127366-90-7
ADONA	Dodecafluoro-3H-4,8-dioxanonanoic acid	919005-14-4
	9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	1621485-21-9
301-5430113	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1
	11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	2196242-82-5
1101-7730003	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9

Abbreviation	Name	CAS#
	Perfluoro-3,6-dioxaheptanoate	39187-41-2
NEDHA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6
	Perfluoro-3-methoxypropanoate	n.a.
Frivifa	Perfluoro-3-methoxypropanoic acid	377-73-1
	Perfluoro-4-methoxybutanoate	1432017-36-1
PFINIBA	Perfluoro-4-methoxybutanoic acid	863090-89-5
	Perfluoro(2-ethoxyethane)sulfonate	220689-13-4
FFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7
PFOSA	Perfluorooctanesulfonamide	754-91-6
N-MeFOSA	N-Methylperfluorooctanesulfonamide	31506-32-8
N-EtFOSA	N-Ethylperfluorooctanesulfonamide	4151-50-2
N-MeFOSE	N-Methylperfluorooctanesulfonamidoethanol	24448-09-7
N-EtFOSE	N-Ethylperfluorooctanesulfonamidoethanol 1691-99	

# **13.0 Data Verification**

# 13.2 Laboratory data verification, requirements, and responsibilities

For results generated by MEL, a "same-party validation" will be performed by MEL staff according to MEL's internal procedures. For example, MEL SOP 730022 describes the peer and final review of organics data. This SOP performs the same tasks through Stage 2B validation, and includes some tasks in Stage 3 validation.

For results generated by a Contract Lab, the verification includes checks to see whether specific requirements described in the contracts' Statement of Work (SOW) were followed, such as providing items in the EDD format and analyzing QC samples as specified in the SOW.

The verification and validation process is summarized in workflow format in Figure 3. Table 20 lists the items to be checked during the data completeness and compliance review prior to the data undergoing more intensive examination of Stage 3 or 4 data validation. Items in Table 20 are also contained within MEL's SOP 770043.

MEL's Quality Assurance Coordinator will:

- Conduct verification of contract lab data according to MEL SOP 770043 and the checklist that is Table 20. Note that the verification process consists of checking that the item is merely present in the data package and, for some items, the requirements in the SOW are met (e.g. QC limits).
- Arrange for validation to be done within MEL or by a qualified vendor.
- Shepherd the data package through the process shown in Figure 3.
- Provide the Project Manager with the final validated data package along with a Case Narrative.

The Case Narrative will summarize:

- The nature of the verification and validation work.
- The location where results and related details are stored, such as: the analytical method used, sample ID scheme, QC results, and batch IDs.
- Compliance with analytical method, lab QA/QC limits, and the MQOs described in this QAPP or subsequent QAPP addendums.
- Explanations and discussion about circumstances that affect the quality of the data.
- The assignment, and definitions, of data qualifiers.



Figure 3. Workflow for Contract Lab Data Verification and Validation.



#### Figure 3, continued.

Proposed	Line from SOD 770042 1 (1 - 2010)	Present and		
Alternate	Items from SOP 770043 v1 (June 2019):	Compliant?		Comments
SOP Ref #	Section 4 and Appendix A: "Data Completion Unecklist"	Yes	No	
1	1. Cover Page			
2	2. Project Narrative			
3	3. Sample Correlation			
4	4. Analytical Method Summary (SOP)			
5	5. Sample Shipment and Lab Receipt Documentation			
5.01	Chain-of-Custody			
5.02	Work Order Number			
5.03	Sample Matrix			
5.04	• Field Blanks (if applicable)			
5.05	• Field duplicates (if available)			
5.06	• Field spikes (if available)			
5.07	• PE, SRM, CRM samples (if available)			
5.08	Sample collection date			
5.09	Sample collection time			
5.10	Samplers name/initials			
5.11	Shipping date			
5.12	Preservatives			
5.13	Types of Analysis			
	• Name, address and signature of sample receipt from the			
5.14	contract lab			
6	6. Sample pre-treatment Documentation			
7	7. Sample Extraction /Prep Bench sheets			
8	8. Sample Clean-up Bench sheets			
9	9. Preparation logs for standards			
10	10. Summary of Analytical Results			
10.a	a. Sample Results Data Analysis Sheet (the EDD*)			
10.a.01	• EDD format complies with SOW			
10.a.02	· Result Detection Limits comply with SOW specifics			
10.a.03	· Result Reporting Limits comply with SOW specifics			
10.a.04	· Laboratory flags/qualifiers are used per SOW			
10.a.05	· Laboratory flags/qualifiers are defined per SOW			

Table 20. Items to be checked during the data review or verification process.

#### Table 20 continued.

Proposed Alternate	Items from SOP 770043 v1 (June 2019):	Present and		
		Compliant?		Comments
SOP Ref #	Section 4 and Appendix A. Data Completion Checkist	Yes	No	
10.b	b. Chromatogram which includes			
10.b.01	Sample Number			
10.b.02	Date and Time of Analysis			
10.b.03	Instrument Identifier			
10.b.04	Date and Time of Analysis			
10.b.05	Laboratory File Identifier			
10.b.06	Analyst ID			
10.c	c. Quantitation Report which includes			
10.c.01	Sample Number			
10.c.02	Date and Time of Analysis			
10.c.03	Absolute RT and RRT			
10.c.04	• Ions or M/Zs used for Quantitation with measured areas			
10.c.05	• Copy of area table from data system			
10.c.06	Total area of two component channels			
10.c.07	On-column concentration including units			
10.c.08	• Final concentration with units			
10.c.09	Analyte Name			
	• M/Z Ration and a Yes or No indicator whether within			
10.c.10	acceptable limit or not			
10.c.11	Instrument Identifier			
10.c.12	Laboratory Filename			
10.c.13	Analyst ID			
11	11. Quality Control Data			
11.01	Method Blanks			
11.02	Equipment Blank			
11.03	Proof Blank			
11.04	• Field Blank			
11.05	Rinsate Blank			
11.06	Laboratory Control Sample (OPR)			
11.07	Duplicate Sample Analysis			
11.08	• PE, SRM, or CRM Analytical Data			
11.09	Matrix Spike/Matrix Spike Duplicate Data			

#### Table 20 continued.

Proposed	Items from SOP 770043 v1 (June 2019): Section 4 and Appendix A: "Data Completion Checklist"	Present and		
Alternate SOP Ref #		Yes	No	Comments
12	12. Standard Data			
	• Instrument Performance Checks in the order of analysis: date			
12.01	and time completed for each 12 hour period per instrument used			
12.02	Window Defining Mix			
12.03	Chromatographic Resolution Summary			
	• Initial Calibration Data Summary, arranged in chronological order by instrument, date and time of analysis with %RSD, % recoveries, relative response factor (RF/FFF), retention time,			
12.04	relative retention time (RT/RRT), Ion Abundance Ratio			
12.04.a	Mass Resolution Data			
12.04.b	Standards, SICP, and complete data system reports			
	• Continuing Calibration Verification Data must be arranged in			
12.05	chronological order by instrument, date and time of analysis			
13	13. Clean-up Data			
13.01	• GPC Calibration, chromatograms and data system reports			
14	14. Miscellaneous			
14.01	Communication logs			
14.02	Non-conformance memos and corrective action reports			
14.03	Standards Certificate			
14.04	Accreditation Certificate			

### 13.3 Validation requirements, if necessary

Some elements of validation will be conducted as described in Section 13.2 above. How validation is conducted depends to some degree on which laboratory analyzes samples for which parameters. For example, for parameters analyzed by MEL, MEL conducts data review and some elements of validation that are part of EPA's Stage 4 validation. For parameters analyzed by a contract lab, data review (some level of verification) will be conducted by MEL, whereas the broader data validation work could be conducted by MEL or a qualified vendor. The decision of who will conduct data validation will be determined at a later date and be somewhat dependent on MEL's capacity for data validation.

Appendix B shows validation requirements for contract lab work associated with this project: these requirements are in addition to the workflow and checklist described in Section 13.2 above. The checklist in Appendix B was developed as a tool to communicate validation needs to MEL.

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# 16.0 Appendices

# Appendix A. Steps for Obtaining Contract Lab Services through Manchester Environmental Laboratory (MEL)

This checklist can serve as an internal tool to aid communication about the contracting process.

#### **Staff Roles**

MEL Lab Director – Alan Rue MEL QA Coordinator – Christina Frans MEL Project Coordinator – Nancy Rosenbower MEL Contract Manager – Deborah Clark TSU Project Manager – Keith Seiders

#### **Initiating Project**

- Project Manager (PM) defines needs for contract lab services: target analytes, methods, sample size, RLs, DLs, EDD, etc.
- PM contacts MEL Project Coordinator (Nancy) via email with notification of project/contract lab needs. Cc MEL QA Coordinator (Christina) and MEL Contract Manager (Deborah).
- \_\_\_\_\_ MEL QA Coordinator does the waiver for analysis if needed.
- \_\_\_\_\_ Decision to contract conducted by MEL QA Coordinator (or designee). Either here or later in the process. Need to know the # of samples, full budget amount and duration.
- MEL Project Coordinator replies to PM email with follow-up questions (if necessary) and works with the PM to determine whether the analysis will go through a formal bid process (RFQ), go to DES Master Contract, or direct buy (<\$30k). Projects with anticipated value between \$25K - \$30K that are not on the DES Master Contract, must go through the RFQ process to ensure buffer for coverage, billable QC and other miscellaneous charges that could arise. MEL QA Coordinator provides most recent template for SOW to PM. (allow 1-2 weeks).
- MEL Project Coordinator notifies MEL Contract Manager, Lab Director, and QA Coordinator of the project and need for contract lab services.

### **Drafting Scope of Work (SOW)**

- PM drafts SOW (draft 1) and sends to MEL QA Coordinator for review.
- MEL QA Coordinator reviews draft 1 of PM's SOW and works with PM to ensure project needs can be met and revises as needed. (allow 2 weeks if non-RFQ, 3 weeks if RFQ); if project is complex QA Coordinator will communicate to PM a more realistic time frame)
- \_\_\_\_\_ MEL QA Coordinator sends the draft 1 SOW with comments/edits back to PM.
- PM works with MEL QA Coordinator to reconcile issues. Final SOW is produced by MEL QA Coordinator and sent to PM. (allow 1 week).
- For an RFQ: PM approves the final SOW and sends the final SOW to MEL Contract Manager, with MEL QA Coordinator Cc'd. MEL Contract Manager approves final SOW as to appropriate bidder documentation requirements and scoring. (allow 3 days).

Direct Buy or DES Master Contract: PM approves the final SOW and sends the final SOW to MEL Project Coordinator with MEL QA Coordinator Cc'd.

### Direct Buy (not on DES Master Contract or RFQ)

- \$0 to \$5,000 Informal bid. Competition is encouraged but not required. MEL Project Coordinator will contact 1-3 vendors for quotes for an informal bid.
- \$5001 to \$30,000 (\$40,000\*) Informal competition is required. MEL Project Coordinator must contact a minimum of 3 vendors if available for quotes for an informal bid. If only one vendor can provide the item a brief explanation will need to be provided. \*\$40,000 exception if you can document that it is in-state Small Businesses, certified Minority & Women's Business Enterprises (MWBE), or certified Veteran-owned businesses.
- The Purchase Request number (PR#) will need to be on the Chain of Custody (COC) when you send your samples. This helps the contract labs and MEL to keep the project and the PR# connected. Email MEL Project Coordinator and MEL QA Coordinator to get the PR# for your project.

#### DES Master Contract

- If using the DES Master Contract 01519, MEL Project Coordinator will email the SOW to the contract lab confirming they can deliver all that is requested on the SOW for the contract price. If they cannot honor the contract price, then it becomes either a Direct Buy or RFQ (Allow 3-4 weeks).
- The PR# will need to be on the COC when you send your samples. This helps the contract labs and MEL to keep the project and the PR# connected. Email MEL Project Coordinator and MEL QA Coordinator to get the PR# for your project.

#### **Request for Quotes (RFQ)**

- If a formal bid solicitation is necessary for the project, the MEL QA Coordinator drafts the necessary bid solicitation RFQ and sends onto PM for final review. Timeline varies with complexity of the project requirements, but generally takes at least 2-3 weeks.
- MEL Contract Manager finalizes RFQ then posts the bid solicitation with final SOW/RFQ language following contract guidelines to Washington's Electronic Business Solution (WEBS) following WEBS requirements. Allow one week.
- MEL Contract Manager notifies PM that the bid solicitation has been posted and provides final copy to PM.
- When the bidding period (14 21 calendar days) has ended, MEL Contract Manager forwards bids received to MEL QA Coordinator who compiles bid information and scores bids.
- \_\_\_\_\_ MEL QA Coordinator evaluates bids and emails PM and MEL Contract Manager with the results of scoring.
- PM reviews bid information and MEL's recommendation and comes to an agreement with MEL QA Coordinator on lab to use.
- PM approves the selection of lab to use via email to MEL's Contract Manager (CC: MEL QA Coordinator). MEL QA Coordinator ensures all PM's requirements are being met, however, bids submitted are the binding documents for all RFQ awards.

- MEL Contract Manager posts Apparent Successful Bidder notifications to WEBS (all labs that submitted a bid will be able to view on WEBS). There is 3-day (72 hour) required debriefing period.
- If unsuccessful bidders challenge the award (within 5-day challenge period), MEL Contract Manager informs MEL's QA Coordinator and PM and all work together as needed to resolve the challenge.
- In the unusual case that a different lab is awarded the bid, PM must approve the selection of the lab. (Scoring Matrix, as published in the RFQ is the binding document. However, input into the scoring of the matrix initially is done jointly with the PM, Contracts Management Training is required for PMs if PMs are going to have a roll deciding on the Successful Bidder.)
- The PR# will need to be on the COC when you send your samples. This helps the contract labs and MEL to keep the project and the PR# connected. Email MEL Contract Manager to get the PR# for your project.

#### Helpful Hints

Any project can have an RFQ, if desired, but must be RFQ if >\$30k and not on contract. Example: if you want to lock in one lab.

We now have DES contracts for certain parameters and media. Example: We had a contract for dioxin in tissue but not for water.

### Appendix B. Checklist for Communicating Requirements for Validation of Contract Lab Data Packages for Organics Analyses

This checklist provides guidance from Project Managers (PMs) to Manchester Environmental Laboratory's (MEL) validator of contract lab (CL) data packages for the tasks to be completed by MEL's validator for organics data CL packages. These data packages typically include HRMS analyses, but can also include low level LC-MS/MS methods.

Tier 4 data packages should always be requested in the Statement of Work for contracts unless otherwise stated. This does not mean that all data packages require Stage 4 validation, but the information necessary to conduct Stage 4 validation is available if needed.

#### Validation checklist

- 1. Follow MEL SOP #770043: Data Validation of Contracted HRMS Analytical Data.
- 2. Check that the data package and electronic data deliverables (EDDs) comply with all items in the Statement of Work and the Quality Assurance Project Plan. Make corrections (or have lab correct) where needed. Be certain to check the following:
  - a. Check the EDD for required formatting of all fields, particularly the "parameter name" for PCB congeners.
  - b. Check that the SOW-required LOQs for all analytes were met for each sample result (includes lab reps and SRM/CRM).
- Conduct the following level of Verification and Validation as defined in Appendix A of EPA's "Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use": EPA 540-R-08-005, January 2009.

4. Amend the original EDD as described in MEL SOP#770043 by adding and populating the MEL Amended Result and MEL Amended Qualifier. Add "Reason for MEL qualification" code definitions to the case narrative.

MEL Amended	MEL Amended	Reason for MEL
Result	Qualifier	Qualification

- 5. If the CL provided multiple EDDs, amend each one as appropriate (do not combine multiple EDDs into one final EDD).
- 6. Do not recalculate homolog totals, totals or TEQs, except as requested by the PM. If requested, state in the case narrative whether totals or TEQs have been recalculated based on the validated data.
- 7. Exclude evaluation of the standard or certified reference materials (SRM/CRM) other than treating it as another sample.
- 8. Censor results on the Laboratory Method Blank. Do not use other types of blanks.
- 9. Use the following basis for censoring (recommend 5x the Laboratory Method Blank as default for HRMS):

10. Do not conduct the following as part of validation unless requested:

- Do not change NJ qualified results that are greater than EQL (or LOQ) to J based on chromatograms.
- Do not re-censor results based on the IRV (instrument response value) of method blanks where the CL qualified these as U but review of the chromatogram suggests the analyte is present.