DEPARTMENT OF
ECOLOGY
State of Washington

Quality Assurance Project Plan

Sampling for Per- and Polyfluroalkyl Substances (PFAS) in Landfill Leachate in the State of Washington

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Data for this project will be available on Ecology's Environmental Information Management (EIM) website: EIM Database. Data will be located in the EIM study for the individual landfill from which the samples were collected.

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Quality Assurance Project Plan

Measuring Per- and Polyfluroalkyl Substances (PFAS) in Landfill Leachate in the State of Washington

January 2020

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2.0 Abstract

PFAS are synthetic chemicals that have properties of oil and water repellency, temperature resistance, and friction reduction. Because of these unique properties, these man-made substances have been used in a wide variety of commercial products since the 1940s. Many of these products, such as the waterproofing materials used on carpets and clothing or packaging used for foods, can end up in landfills. As these materials degrade, PFAS can be released to the air, wastewater treatment plants, and eventually to drinking water supplies.

The chemistry of PFAS makes the substances persistent in nature and bio-accumulative. They can have serious health effects including detrimental effects to the liver, reproductive, and cardiovascular systems, and may be carcinogenic. Several states that have water contaminated with PFAS have identified severe health effects from drinking that water.

Washington State has not had its landfills tested for the presence of PFAS. The first phase of this program is to sample leachate from landfills. Later studies will sample landfill gas and groundwater to help determine if landfills in the state could be a source for PFAS in the environment.

The EPA has a health advisory of 70 parts per trillion for two of the PFAS substances in drinking water, but there are no established regulatory limits. Phase I of the study will help determine which, if any, landfills have the potential to contaminate the environment.

3.0 Background

3.1 Introduction and problem statement

PFAS, or Per- and Polyfluroalkyl substances (PFAS), are manufactured substances invented in the 1930s that are now widespread in the environment. Blood testing shows that nearly every human being, including newborns, has some PFAS in their body (ITRC, 2017). Increased cancer, birth defects, and other health problems are present around some PFAS-producing facilities that have contaminated groundwater.

There are over 4,700 different PFAS substances. Many materials used in our everyday lives contain PFAS including Teflon, fabric protectors, fire-fighting foam, Gore-Tex, cosmetics, and other household products. Two of the most common PFAS are PFOS (perfluorooctane sulfonates) and PFOA (perfluorooctanoic acid). PFOS was voluntarily withdrawn from the market by 3M in 2002. The EPA developed a global stewardship program to eliminate PFOA by 2015. Industry implemented this ban in much of the world; however, studies show that substitute PFAS compounds are also toxic (Wang et. al. 2017).

Products containing PFAS often end up in landfills where they can degrade and accumulate in leachate. In landfills that are unlined, or have compromised liner systems, leachate may contaminate groundwater. PFAS from leachate sent to wastewater treatment plants may be discharged to streams. Eventually, PFAS from leachate may be introduced into public water supply systems. PFAS have also been detected in the air around landfills and wastewater treatment plants.

This is why the Washington State Department of Health and the Department of Ecology (Ecology) are developing a joint Chemical Action Plan (CAP). The state legislature named PFAS a priority chemical in Senate Bill 5135. Preliminary recommendations include the following:

1. Ensure safe drinking water,
2. Manage environmental contamination,
3. Reduce PFAS in products, and
4. Research and manage PFAS in waste.

Several states, particularly those with PFAS-producing facilities, have already completed extensive sampling of groundwater, landfills, soils, and air. There are no known PFAS-producers in the state of Washington.

In Washington, most of the known PFAS groundwater contamination is attributed to the use of fire-fighting foam agents termed aqueous film forming foam concentrates (AFFF). However, other possible sources, such as leachate from landfills, have not been investigated.

3.2 Study area and surroundings

The study area includes landfills throughout the state of Washington. In Washington State, solid waste landfills are regulated under different administrative codes, depending on the type of landfill. Only Limited Purpose Landfills (Chapter 173-350 WAC) and Municipal Solid Waste Landfills (Chapter 173-351 WAC) are required to have leachate collection systems. This includes most of the active landfills, with the exception of Inert Waste Landfills. A few closed landfills regulated under Chapter 173-304 WAC also have leachate collection systems. Landfills were contacted for permission to collect PFAS samples. Selection of the specific landfills to be sampled will not be determined until responses are received from the landfills.

Jurisdictional Health Departments (JHDs) are the regulating authority for these facilities. The Solid Waste Management program (SWM) writes the state landfill regulations and serves as a technical consultant to the JHDs. Some of the landfills are undergoing remediation either through independent cleanups or through agreements under the state Model Toxics Control Act (MTCA). The Solid Waste Management program oversees some landfill cleanups, and several older landfills in the state are undergoing cleanups administered by Ecology’s Toxics Cleanup Program.

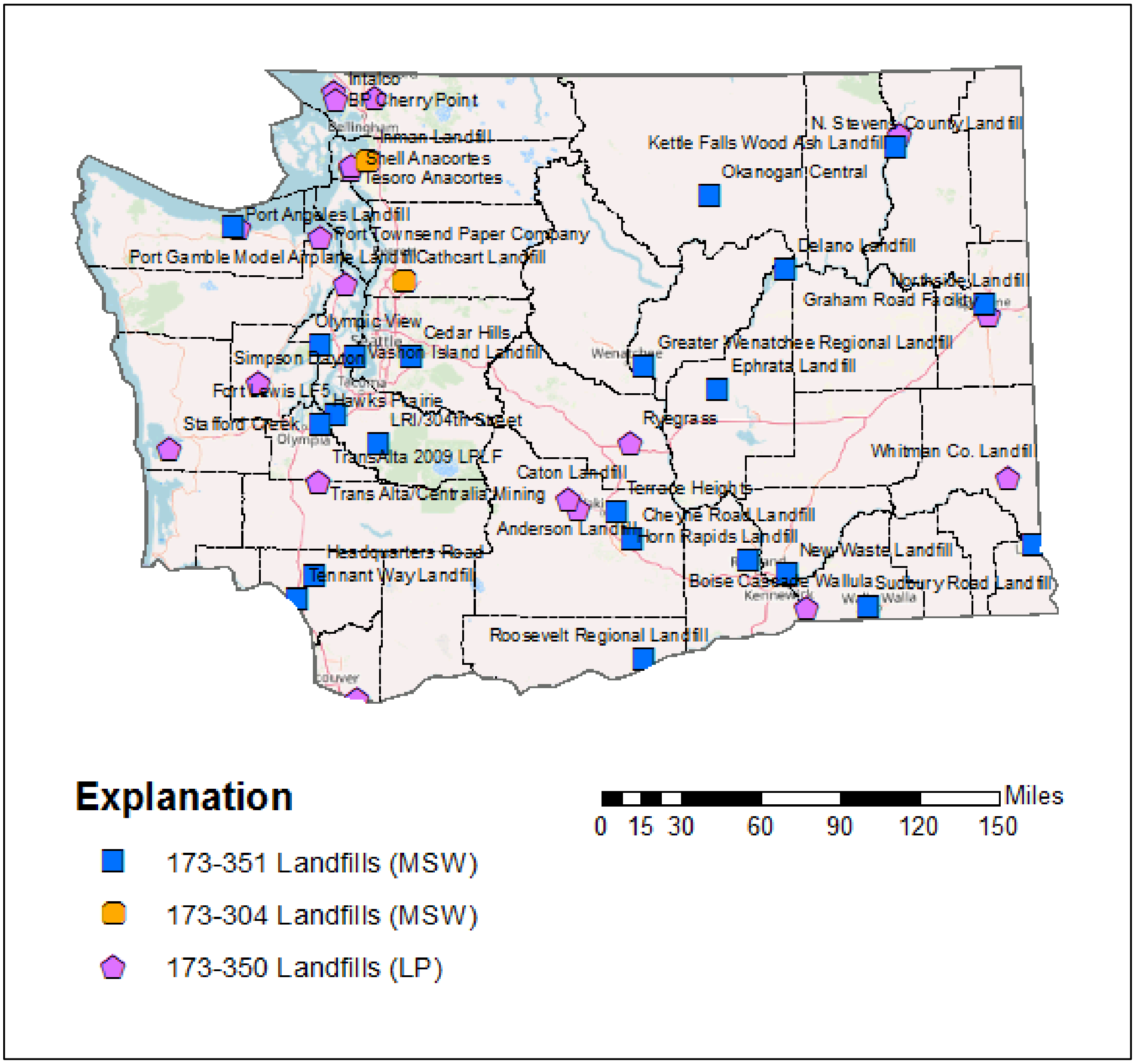

Figure 1. Landfills considered for the Phase 1, Step 1 sampling program.

Explanation refers to applicable WAC regulation and the type of landfill, municipal solid waste (MSW) or limited purpose (LP).

3.2.1 History of study area

Samples will be collected from facilities that are permitted as landfills by the JHD. All landfills that received municipal solid waste on or after November 26, 1993, are required to be permitted under chapter 173-351 WAC. Chapter 173-350 WAC applies to limited purpose landfills in operation after February 3, 2003.

3.2.2 Summary of previous studies and existing data

There are no previous studies of PFAS in landfills in Washington State. This project is part of the joint Department of Ecology/Department of Health PFAS Chemical Action Plan.

3.2.3 Parameters of interest and potential sources

The parameters of interest are PFAS. There are over 4700 identified PFAS substances. The selected analytical laboratory will use a method that can identify up to 33 of those compounds (see Table 6). To determine how many PFAS are not identified with this analysis, a total oxidizable precursor (TOP) analysis will also be conducted on some samples. The TOP analysis can help measure the concentration of PFAS compounds that are not determined by conventional analytical methods (Buechler 2017).

Potential sources for PFAS in landfills includes waterproofing products on carpets and other items, aqueous fire-fighting foam, cosmetics, food packaging, non-stick cookware, and hundreds of other items (Lang et. al. 2017) .

3.2.4 Regulatory criteria or standards

There are no regulatory standards for PFAS in the state of Washington. Some states have set their own limits. The EPA has a health advisory for drinking water of 70 parts per trillion for two of the PFAS substances.

4.0 Project Description

In order to develop the Chemical Action Plan, Ecology will sample landfills across the state for PFAS using a phased approach.

The sampling phases of this study are as follows:

Phase I:

- Step 1: Collect samples of leachate from selected landfills that consent to sampling throughout the state.

- Step 2: Follow-up sampling to Phase 1 at some facilities, if warranted, such as if higher concentrations show up in test results from particular facilities. Also, may collect samples of leachate from additional landfills if investigations suggest a higher probability of PFAS-containing materials in historic waste streams. Cooperation of Jurisdictional Health Departments (JHDs) to include PFAS-sampling as part of their permit requirements may be necessary to sample at some facilities. Some landfills administered by the Toxics Cleanup Program may be included in this phase.

Phase II: Phase II sampling will be dependent on the results of Phase I. Groundwater will be sampled at selected facilities with higher PFAS levels in leachate. A more detailed investigation of historical waste streams may be undertaken, and groundwater may be sampled at some facilities that do not have leachate collection systems. Landfill gas sampling will also be part of this phase. A separate QAPP will be prepared for Phase II.

4.1 Project goals

The goal of Phase I, Step 1 of the project is to determine the concentration of PFAS in leachate in the landfills. Subsequent phases will use this information to guide sampling for PFAS in the air and groundwater in the state and to see what concentrations of PFAS are discharged to Wastewater Treatment Plants.

4.2 Project objectives

Phase I, Step 1 of the project consists of the following:

- Collect approximately 20 or more PFAS samples from landfills.
- Analyze all of the samples for approximately 33 PFAS (dependent on analysis method of selected laboratory).
- Analyze approximately half the samples with a Total Oxidized Precursor method.
- Compare the results with those from landfills in other states.
- Identify landfills that warrant follow-up sampling during subsequent phases of the program.

4.3 Information needed and sources

The new data will be compared to published data from other states.

4.4 Tasks required

Required tasks include sample collection, sample analysis, quality control, review of data on PFAS in leachate from other states, and report preparation.

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Project personnel will include staff from several regions and programs as listed in Table 1 below.

Table 1. Organization of project staff and responsibilities.

Staff	Title	Responsibilities
Cole Carter Solid Waste Management Eastern Regional Office Phone: 509-329-3515	Program Hydrogeologist	Writes the QAPP. Oversees field sampling and transportation of samples to the laboratory. Conducts QA review of data, analyzes and interprets data, and enters data into EIM. Writes the draft report and final report.

Staff	Title	Responsibilities
Tim O'Connor Solid Waste Management Northwest Regional Office Phone: 425-649-7051	Hydrogeologist	Liaison with facilities in his region. Assists with sampling. Report review.
Eugene Radcliff Solid Waste Management Southwest Regional Office Phone: 360-407-6392	Hydrogeologist	Liaison with facilities in his region. Assists with sampling. Report review.
Pat Shanley Solid Waste Management Central Regional Office Phone: 509-484-7293	Hydrogeologist	Liaison with facilities in her region. Assists with sampling. Report review.
Marni Solheim Solid Waste Management Eastern Regional Office Phone: 509-329-3438	Section Manager for the Project Manager	Reviews the project scope and budget, tracks progress, reviews the draft QAPP, and approves the final QAPP. Report review.
Alan Rue Manchester Environmental Laboratory Phone: 360-871-8801	Laboratory Director	Provides input an analyses. Manages contract bidding and approves bid award. Reviews QAPP.
Ginna Grepo-Grove Chemist 4 Manchester Environmental Laboratory Phone: 360-871-8829	Chemist	Quality assurance coordinator. Prepares and reviews bids for laboratory contracts. Ensures quality assurance goals at lab are met.
Contract Laboratory (to be determined)	Project Manager	Supervises analyses of samples. Coordinates with MEL QA Coordinator.
Cathrene Glick Environmental Assessment Program Eastern Regional Office Phone: 509-329-3425	Supervisor	Reviews the draft QAPP. May review and comment on the draft project report.
Mark Dirkx Solid Waste Management Headquarters Phone: 360-407-6937	Facility Engineer	SWM QA Coordinator. Reviews draft QAPP for the Solid Waste Management Program.
Arati Kaza Environmental Assessment Program Headquarters Phone: 360-407-6964	Quality Assurance Officer	Review and approval of final QAPP and QAPP addendum for Ecology.
Brian Penttila Hazardous Waste and Toxics Reduction Program Headquarters Phone: 360-407-6758	Chemist	Provides input on analytical methods and project review.

5.2 Special training and certifications

Licensed hydrogeologists from the Solid Waste Management program who will collect samples. All have also completed the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) training and yearly 8-hour refreshers. All field personnel will received facility specific safety training prior to sampling. Other training includes statistical analysis, sampling techniques, and GIS.

5.4 Proposed project schedule

Sampling will begin shortly after competitive bids for laboratory analyses are received and awarded, and sampling schedules for the sites are arranged. We are targeting a start date of early-January 2020, and sampling completed by March 2020. We will collect approximately 40 samples during Phase I, Step 1 of the program. Table 2 shows the proposed timeline for key project tasks.

Table 2. Proposed schedule for completing field and laboratory work, data entry into EIM, and reports.

Field and laboratory work	**Due date**	**Lead staff**
Field work completed	March 2020	Cole Carter
Laboratory analyses completed	May 2020	Nancy Rosenbower
Level IV data validation completed	July 2020	Ginna Greppo-Grove
Environmental Information System (EIM) database		
EIM Study ID: TBD	ID number: TBD	
Product	Due date	Lead staff
EIM data loaded	July 2020	TBD
EIM data entry review	August 2020	EAP
EIM complete	August 2020	EAP
Final report		
Author lead / Support staff	To be determined / To be determined	
Schedule		
Draft due to supervisor	August 2020	
Draft due to client/peer reviewer	August 2020	
Draft due to external reviewer(s)	September 2020	
Final (all reviews done) due to publications coordinator (Joan)	September 2020	
Final report due on web	October 2020	

5.5 Budget and funding

Funding for this project comes from the Hazardous Waste Program’s budget for testing chemicals of concern in consumer products. The initial amount available is $80,000.

The principal expense for the project is laboratory analyses. The budget for Phase I includes rental for a YSI Pro multi-parameter meter and purchase of expendable items for sampling. Personnel time and travel expenses will be charged to the Solid Waste Management program.

Table 3 shows a proposed budget for the initial sampling.

Table 3. Project budget for Phase 1, step 1.

Item	Number of Samples	Number of Field QC Samples	Total Number of Samples	Cost Per Sample	Contract Lab Subtotal
Analysis Method					
TBD	38	4	42	$500	$21,000
TBD w/ TOP	20	2	22	$1000	$22,000
PFAS Contract Lab Total					$43,000
MEL Contract Lab Fee Total (30%)					$12,900
Analysis Total					**$55,900**
Field Expenses					
Equipment Rental and Supplies					$1,100
GRAND TOTAL:					**$57,000**

6.0 Quality Objectives

6.1 Data quality objectives

The main data quality objective (DQO) for this project is to collect a minimum of 20 leachate samples from selected landfills throughout the state and have them analyzed for PFAS. The exact analysis will depend on the laboratory that is awarded the bid. Generally, the analytical method will be consist of LC/MS-MS. This method will identify approximately 33 PFAS substances, depending on the laboratory selected. Additionally, there will be a TOP analysis on some of the samples.

6.2 Measurement quality objectives

6.2.1 Targets for precision, bias, and sensitivity

This section describes the MQOs for project results, expressed in terms of acceptable precision, bias, and sensitivity, and is summarized in Table 4 below.

Table 4. Measurement quality objectives for laboratory analyses of leachate samples.

Measurement Quality Objectives →	Precision	Bias		Sensitivity
	Laboratory Duplicate/Field Split Samples	Lab Control Standard[1]	Extracted Internal Standard Recovery[2]	Lowest Concentrations of Interest[3]
	Relative Percent Difference	Recovery Limits		Concentration Units
PFAS Analyses				
21+ substance analyses	± 40%	varies by lab	varies by lab	0.5 – 5 ppt
TOP analyses	± 40%	varies by lab	varies by lab	0.5 – 5 ppt

1. Laboratory Control Standard is also referred to as Ongoing Precision and Recovery (OPR) Standard, in which a laboratory blank sample is spiked with known quantities of analyte.
2. Internal Standard Recovery is also referred to as isotopic dilution using primarily 13C-labeled isotopes.
3. Method blank contamination should be less than reporting limits.

6.2.1.1 Precision

Precision is a measure of the variability between results of replicate measurements due to random error. It is assessed by calculating the relative percent difference (RPD) between the replicate measurements. Field splits are collected by taking two aliquots from one homogenized sample and analyzing them as separate samples. Precision of field splits is assessed in the same manner as field replicates.

For this project, field splits will be collected and analyzed. Field splits will be collected at about 10% of the total number of samples for each matrix. Laboratory duplicates will also be prepared and analyzed by the laboratory. The targets for acceptable precision for each sample matrix are shown above in Table 4.

6.2.1.2 Bias

Bias is the difference between the sample mean and the true value. For this project, bias will be measured as a percent recovery of laboratory blank spikes and percent recovery of labeled congener compounds. Targets for acceptable recoveries are shown above in Table 5.

6.2.1.3 Sensitivity

Sensitivity measures the capability of an analytical method to detect a substance above background level, and is often described as a detection or reporting limit.

6.2.2 Targets for comparability, representativeness, and completeness

6.2.2.1 Comparability

To ensure that data from this project are comparable to other studies, the Department of Ecology's *Guidelines for Preparing Quality Assurance Project Plans for Environmental Studies* (Ecology, 2004) will guide the sampling. Additional references include the following:

- *Standard Operating Procedures for Decontaminating Field Equipment for Sampling Toxics in the Environment, Version 1.1.* SOP Number EAP090 (Friese 2014).

- *MDEQ General PFAS Sampling Guidance*, Revised 10/16/2018, Michigan Department of Environmental Quality.

6.2.2.2 Representativeness

A limited number of landfills will be sampled during Phase I. Since only landfills with leachate collection systems will be sampled during Phase I, it will not be possible to collect samples from most older, closed landfills. However, the randomness involved with the landfills available for Phase I sampling will result in representative samples.

6.2.2.3 Completeness

This project will achieve completion if at least 95% of the planned samples are collected and analyzed successfully, and the data are deemed acceptable.

6.3 Acceptance criteria for quality of existing data

No known PFAS data exists for landfills in Washington State.

6.4 Model quality objectives

NA

7.0 Study Design

7.1 Study boundaries

As noted in section 3.2, samples will be collected at landfills throughout the state.

Refer to Figure 1.

7.2 Field data collection

To ensure consistency, Ecology personnel will collect the samples where possible. A landfill employee will accompany the samplers. If a landfill wants their employee to collect the samples, we will require they follow QAPP procedures to avoid contamination.

Ecology will collect leachate using grab samples, i.e., single samples taken at specific times. Some landfills have separate leachate collection systems for each cell. In those cases, we may collect more than one sample from a facility during Stage I. PFAS can concentrate at a water surface, so samples from leachate ponds will be collected from below the air/water interface.

7.2.1 Sampling locations and frequency

Samples will be collected from the most practical point nearest to where the leachate leaves the landfill unit. Leachate samples are often collected from a sample port where leachate is discharged to a wastewater treatment plant or transferred to a truck. At facilities that use leachate lagoons, samples will be collected at the discharge to the leachate pond or directly from the pond with a scoop sampler. In some cases, samples may be collected from a leachate sump.

Table 5. Landfills considered for Phase I PFAS sampling.

Landfill	Regulation[1]	Status	County	Region[2]
Horn Rapids Landfill	173-351	active	Benton	CRO
Greater Wenatchee Regional Landfill	173-351	active	Douglas	CRO
Ryegrass Landfill	173-350	active	Kittitas	CRO
Roosevelt Regional Landfill	173-351	active	Klickitat	CRO
Okanogan Central	173-351	active	Okanogan	CRO
Anderson Landfill	173-350	active	Yakima	CRO
Caton Landfill	173-350	active	Yakima	CRO
Cheyne Road Landfill	173-351	active	Yakima	CRO
Terrace Heights Landfill	173-351	active	Yakima	CRO
Asotin County Landfill	173-351	active	Asotin	ERO
New Waste Landfill	173-351	closed	Franklin	ERO
Ephrata Landfill	173-351	active	Grant	ERO
Graham Road Landfill	173-350	active	Spokane	ERO
Northside Landfill	173-351	active	Spokane	ERO
Kettle Falls Wood Ash Landfill	173-350	active	Stevens	ERO
Stevens County Landfill	173-351	active	Stevens	ERO
Sudbury Road Landfill	173-351	active	Walla Walla	ERO

Landfill	Regulation[1]	Status	County	Region[2]
Georgia Pacific Camas Landfill	173-350	active	Clark	Industrial
Shell Anacortes	173-350	active	Skagit	Industrial
Tesoro Anacortes	173-350	active	Skagit	Industrial
BP Cherry Point	173-350	active	Whatcom	Industrial
Intalco Landfill	173-350	closed	Whatcom	Industrial
Cedar Hills Landfill	173-351	active	King	NWRO
Vashon Island Landfill	173-351	closed	King	NWRO
Olympic View	173-351	closed	Kitsap	NWRO
Port Gamble Model Airplane Landfill	173-350	closed	Kitsap	NWRO
Inman Landfill	173-304	closed	Skagit	NWRO
Cathcart Landfill	173-304	closed	Snohomish	NWRO
County Construction Recyclers	173-350	closed	Whatcom	NWRO
Nippon Industries USA Lawson Landfill	173-350	active	Clallam	SWRO
Port Angeles Landfill	173-351	closed	Clallam	SWRO
Headquarters Road	173-351	active	Cowlitz	SWRO
Tennant Way Landfill	173-351	closed	Cowlitz	SWRO
Stafford Creek Landfill	173-350	active	Grays Harbor	SWRO
Port Townsend Paper Company	173-350	closed	Jefferson	SWRO
Trans Alta/Centralia Mining	173-350	active	Lewis	SWRO
Trans Alta 2009 Limited Purpose Landfill	173-350	closed	Lewis	SWRO
Simpson Dayton Landfill	173-350	closed	Mason	SWRO
Fort Lewis Landfill 5	173-351	closed	Pierce	SWRO
LRI/304th Street Landfill	173-351	active	Pierce	SWRO
Hawks Prairie Landfill	173-351	closed	Thurston	SWRO

1. "Regulation" refers to the chapter of the Washington Administrative Code.
2. "Region" refers to the Solid Waste Management Program that oversees the facility: CRO (Central Regional Office), ERO (Eastern Regional Office), NWRO (Northwest Regional Office), SWRO (Southwest Regional Office), or the Industrial Section which oversees facilities throughout the state.

7.2.2 Field parameters and laboratory analytes to be measured

A YSI Pro multi-parameter meter, or equivalent instrument, will measure field parameters before each sample is collected. The instrument is capable of measuring several parameters, including the following:

- Temperature
- Dissolved oxygen
- pH
- Specific conductance
- Total dissolved solids

7.3 Modeling and analysis design

NA

7.3.1 Analytical framework

NA

7.3.2 Model setup and data needs

NA

7.4 Assumptions in relation to objectives and study area

When leachate contacts air, it is subject to physical, chemical, and biological reactions that can change its composition. However, the persistence of PFAS in the environment minimizes the concern of avoiding aeration when collecting samples. The chemical and physical properties of PFAS makes samples exposed to air acceptable.

7.5 Possible challenges and contingencies

Because of the low detection limit values, measured in parts per trillion, associated with PFAS analyses, avoiding outside contamination during sampling is critical. The Michigan Department of Environmental Quality (now called Michigan Department of Environment, Great Lakes, and Energy) developed a list of prohibited items during PFAS sampling events. We will follow the guidance in the *MDEQ PFAS Sampling Quick Reference Guide* for what is prohibited, allowable, and needs screening. The long list of prohibited items includes items that contain fluoropolymers such as Teflon bottles or caps, notebooks with PFAS treated paper, Decon 90, new or unwashed clothing, anything made with Gore-Tex or other weather-resistant synthetics, anything washed recently with fabric softeners or protectors, any sun screen or insect repellent, and any prepackaged foods. The laboratory will provide PFAS-free decontamination water.

The use of equipment that has PFAS-containing components at landfill sites is common. For example, some bladder pumps used in low-flow well sampling are constructed of stainless steel and Teflon. We will use alternative methods of sample collection if necessary.

7.5.1 Logistical problems

Potential logistical problems include limited travel due to winter weather (pass closures, etc.), lack of permission to sample from facility owners, and shortage of personnel to complete project on time.

To address these problems, we will:

- Allow sufficient time in the schedule for inclement weather.
- Ask permission from more landfills than we plan on sampling during Phase 1.
- Expose other Ecology personnel to the project so they can assume responsibilities, if needed.

7.5.2 Practical constraints and schedule limitations

Safety is a primary concern. Safety concerns could preclude sampling at any facility.

Permission to sample a reasonable number of landfills is essential to meet the proposed start date of the Phase I program. If we are unable to secure permission, we will work with the regulating authority for each landfill to require PFAS sampling. That could take several months or longer.

Inclement weather and a number of other factors could affect the ability to collect samples. We will adjust the schedule as needed.

8.0 Field Procedures

8.1 Invasive species evaluation

There is no possibility of contamination from invasive species in leachate samples.

8.2 Sampling procedures

Samples will be collected from the most practical point nearest to where the leachate leaves the landfill unit. Leachate samples are often collected from a sample port where leachate is discharged to a wastewater treatment plant or transferred to a truck. At facilities that use leachate lagoons, samples will be collected at the discharge to the leachate pond or directly from the pond with a scoop sampler. In some cases, samples will be collected from a leachate sump.

8.3 Containers, preservation methods, holding times

The laboratory will provide HDPE appropriate sample bottles and shipping containers. Samples will be kept at the recommended temperature and shipped to the laboratory as soon as possible. Blue ice will be used if approved by the laboratory; otherwise, water ice in zip-lock bags will be substituted. The laboratory will notify us if samples are not received within the specified holding time, and will provide preservatives, if necessary. The holding time is 28 days from sample collection to preparation, and 30 days from preparation to analysis of sample extracts.

8.4 Equipment decontamination

Equipment will be decontaminated with Alconox (or approved equivalent), then triple rinsed with PFAS-free deionized water, and dried with a cotton cloth or untreated paper towel.

8.5 Sample ID

The Sample ID will consist of the facility name or abbreviation and successive numerals. For example, the first sample from the Ephrata Landfills will be “EphrataLF-1”. Sample IDs will be recorded in the field notebook and on the chain of custody.

8.6 Chain-of-custody

A chain of custody sheet will be provided by the laboratory. The sheet will be filled out after each sample is collected, and securely attached to the shipping container prior to sending it to the lab.

8.7 Field log requirements

The information below will be recorded in the field log.

- Name and location of project
- Field personnel
- Sequence of events
- Any changes or deviations from the QAPP
- Environmental conditions
- Date, time, location, ID, and description of each sample
- Field instrument calibration procedures
- Field measurement results
- Identity of QC samples collected
- Unusual circumstances that might affect interpretation of results

8.8 Other activities

NA

9.0 Laboratory Analyses

As a state agency, Ecology must use Manchester Environmental Laboratory (MEL), the state laboratory. MEL, however, does not have an analysis method developed for PFAS in leachate. A competitive bid is required because the budgeted cost of analyses exceeds the $30,000 limit for direct buy. For this project, MEL will make recommendations to Ecology and manage the bidding process for analyses from external labs. Additionally, they will review quality control procedures for the project. Since we do not know what lab will be awarded the contract, we are unable to discuss their specific analytical technique for PFAS or TOP.

Currently, the only EPA method for non-potable water is Method 8327, but it is a non-isotopic dilution, direct injection method with much higher reporting limits. However, because the draft method is still being finalized there are few, if any, laboratories accredited by the method. Another method that has gained an acceptance for leachate is ASTM D-7979. Region 5 of the EPA is currently using this method for their leachate analyses.

The laboratory chosen for this project must be accredited by the State of Washington for non-potable water or solids.

An additional oxidation, extraction, and analysis for TOP will be performed on some samples. Some of the PFAS in fire-fighting foams cannot be detected by analytical methods like EPA's 537 and 8327 or ASTM's D-7979. The TOP analysis can convert undetectable PFAS to detectable PFAS-derivatives. The TOP analysis only provides an estimate, but it is important to determine whether non-detects are due to a true absence of PFAS or are simply due to limitations in Methods 8327 or D-7979. Previous experience with many sampled media have shown that samples that first show only non-detects often will reveal "hidden" PFAS after the TOP preparation. TOP analyses will be performed on a limited number of samples during Phase 1 and will be evaluated for usefulness in later studies.

9.1 Sample parameters table

The PFAS substances to be analyzed will include most of the following parameters, depending on the selected laboratory's procedure (shown here in Table 6).

Table 6. PFAS substances (laboratory).

Abbreviation	Name - Acid Form	CAS#
PFBA	Perfluorobutyric acid	375-22-4
PFPeA	Perfluoropentanoic acid	2706-90-3
PFHxA	Perfluorohexanoic acid	307-24-4
PFHpA	Perfluoroheptanoic acid	375-85-9
PFOA	Perfluorooctanoic acid	335-67-1
PFNA	Perfluorononanoic acid	375-95-1
PFDA	Perfluorodecanoic acid	335-76-2
PFUnA	Perfluoroundecanoic acid	2058-94-8

Abbreviation	***Name - Acid Form***	***CAS#***
PFDoA	Perfluorododecanoic acid	307-55-1
PFTrDA	Perfluorotridecanoic acid	72629-94-8
PFTeDA	Perfluorotetradecanoic acid	376-06-7
PFBS	Perfluorobutanesulfonic acid	375-73-5
PFPeS	Perfluoropentanesulfonic acid	2706-91-4
PFHxS	Perfluorohexanesulfonic acid	355-46-4
PFHpS	Perfluoroheptanesulfonic acid	375-92-8
PFOS	Perfluorooctanesulfonic acid	1763-23-1
PFNS	Perfluorononanesulfonic acid	68259-12-1
PFDS	Perfluorodecanesulfonic acid	335-77-3
PFDoS	Perfluorododecanesulfonic acid	79780-39-5
4:2 FTS	4:2 fluorotelomersulfonic acid	757124-72-4
6:2 FTS	6:2 fluorotelomersulfonic acid	27619-97-2
8:2 FTS	8:2 fluorotelomersulfonic acid	39108-34-4
N-MeFOSAA	N-Methylperfluorooctanesulfonamidoacetic acid	2355-31-9
N-EtFOSAA	N-Ethylperfluorooctanesulfonamidoacetic acid	2991-50-6
HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propanoic acid	13252-13-6
ADONA	Dodecafluoro-3H-4,8-dioxanonanoic acid	919005-14-4
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9
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	1691-99-2

9.2 Sample preparation method(s)

Samples will be extracted following modified EPA methods or their equivalent. Extracted Internal Standards (EIS) are added prior to extraction as surrogates and used to monitor the extraction and recovery-correct the results.

For the TOP analysis, reaction monitoring surrogate(s) are added to the samples prior to the oxidation step, and then oxidized. EISs are then added to the samples as surrogates and used to monitor the extraction and recovery-correct the results *after* the oxidation step.

9.3 Special method requirements

Any special method requirements are dependent on the analytical procedures of the selected laboratory.

9.4 Laboratories accredited for methods

Manchester Environmental Laboratory will select a contract laboratory that is accredited by the State of Washington for the reported PFAS compounds. Because the only applicable EPA Method 8327 is still in draft, all of the potential laboratories are accredited to their SOP and not 8327.

10.0 Quality Control Procedures

The number and type of QC samples to be collected in the field and analyzed in a lab is summarized in Table 7. The bid-winning lab may have their own specific QC procedures, but at a minimum must perform the QC listed in Table 7.

10.1 Table of field and laboratory quality control

The selected laboratory will implement their laboratory quality control procedures. Field quality control will consist of submitting blank and duplicate samples.

Table 7. Quality control samples, types, and frequency.

PFAS Analyses	Field		Laboratory					
	Duplicates	Rinsate Blank	Lab Control Standard[1]	Method Blanks	Internal Standard Recovery[2]	OPR Duplicate	Reaction Monitoring Surrogate
Standard analyses	1 per 10 samples	1/batch[3]	1/batch[3]	1/batch[3]	All samples	1/batch[3]	
TOP analyses	1 per 10 samples	1/batch[3]	1/batch[3]	1/batch[3]	All sample	1/batch[3]	<5%

[1] Laboratory Control Standard is also referred to as Ongoing Precision and Recovery (OPR) Standard, in which a laboratory blank sample is spiked with known quantities of analyte.
[2] Internal Standard Recovery is also referred to as Surrogate or Labeled Compound Recovery, using ^{13}C-labeled congeners.
[3]A batch is a group of samples (typically of the same matrix) processed and analyzed in the laboratory together as a unit.

10.2 Corrective action processes

If activities are found to be inconsistent with the QAPP, if analysis or modeling results do not meet MQOs or performance expectations, or if some other unforeseen problem arises, the following actions may be taken.

- Collect new samples using the method described in the approved QAPP.
- Reanalyzing lab samples that do not meet QC.
- Convening project personnel and technical experts to decide on the next steps that need to be taken to improve model performance.

11.0 Data Management Procedures

11.1 Data recording and reporting requirements

Analytical results will be transferred to Ecology's EIM database. Most labs have an "EIM ready" format that they can send to us, but that depends on what lab is awarded the bid.

11.2 Laboratory data package requirements

The lab will provide a Level IV validatable package that includes the narrative, analytical results and raw data to include chromatograms for all samples, sample QC, and instrument QC. These requirements will be part of the posted RFQQ.

11.3 Electronic transfer requirements

EDD compatible with EIM will be requested from the lab.

11.4 EIM/STORET data upload procedures

The project manager will submit the data formatted for entry into Ecology's EIM data system.

11.5 Model information management

It is not anticipated that this data will be used for modeling.

12.0 Audits and Reports

12.1 Field, laboratory, and other audits

No audits are planned for this project.

12.2 Responsible personnel

Not applicable.

12.3 Frequency and distribution of reports

The final report for this project will be the only report for this project. Supervisors will be orally appraised of progress.

12.4 Responsibility for reports

Timing of the receipt of analytical results will determine who the author of the final report will be. Cole Carter will author as much of the report that can be completed by spring 2020. If additional work is needed to complete the report, it will be conducted by a "to be determined" member of the SWM program.

13.0 Data Verification

Data verification will be conducted by Manchester Environmental Laboratory (MEL). They will complete a Level IV data validation.

13.1 Field data verification, requirements, and responsibilities

Level III data validation includes review and assessment of all of the following:

- Chain of Custody, sample receipt conditions (e.g. pH, temperature).
- Sample QC (e.g. blanks, surrogates) and sample holding times.
- Instrument-related QC reports (e.g. ICAL, CCV).
- Recalculation of 10% of samples, sample QC, and instrument QC using instrument responses.

Gina Grepo-Grove with MEL is the lead for the Level III data validation.

13.2 Laboratory data verification

Lead staff for the laboratory data verification is Nancy Rosenbower with MEL.

13.3 Validation requirements, if necessary

A Level IV validation looks at the actual raw data including chromatograms. The validator reviews 100% of the detects and 10% of the non-detects. Level IV data validation also includes or evaluates the following:

- Summary of analytical results and the chain of custody,
- Surrogate recoveries,
- Method Blank,
- Matrix Spike,
- Matrix Spike Duplicate,
- Laboratory Control Sample,
- Duplicate sample (if available),
- Initial calibration, continuing calibration,

- Instrument performance checks, and
- Any specialized instrument QC (e.g. Instrument Sensitivity, Instrument Carryover) and other QC samples specified by the project like SRM, CRM, ICV, etc.

The instrument and sample results from laboratory instrument responses are recalculated and the recalculated results are compared to the laboratory reported results.

13.4 Model quality assessment

NA

13.4.1 Calibration and validation

NA

13.4.1.1 Precision

NA

13.4.1.2 Bias

NA

13.4.1.3 Representativeness

NA

13.4.1.4 Qualitative assessment

NA

13.4.2 Analysis of sensitivity and uncertainty

NA

14.0 Data Quality (Usability) Assessment

14.1 Process for determining project objectives were met

The goal of Phase I, step 1 of this project is to collect samples of leachate from several landfills in the state and analyze them for PFAS. Data from these samples will be compared to leachate samples collected in other states, and the PFAS levels will be used to guide follow-up sampling of groundwater and gas at landfills in subsequent phases. MEL's data validator will qualify data. The data quality assessment will be made based on the report that the data validator provides.

14.2 Treatment of non-detects

NA

14.3 Data analysis and presentation methods

NA

14.4 Sampling design evaluation

NA

14.5 Documentation of assessment

Data from the program will be stored in Ecology's EIM. Field notebooks will be stored in the Central Files at Ecology's Eastern Regional Office in Spokane. Data quality assessment will be discussed in the final report.

15.0 References

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16.0 Appendices

Appendix A. Standard Operating Procedure

Purpose and scope

This is the Standard Operation Procedure (SOP) for obtaining leachate samples for lab analysis from landfills for the Department of Ecology, Solid Waste Management (SWM) program,

The methods for sampling leachate should be followed unless it is determined that the SOP is not adequate or practical. If the SOP is not followed, the variations and the reasoning for not using the SOP must be documented.

Applicability

This SOP should be followed when collecting leachate samples from landfills.

This SOP is used in conjunction with the facility safety plan.

Personnel qualifications

This SOP pertains to all SWM staff, individuals taking samples for SWM staff, facility staff taking samples for SWM use or visitors accompanying inspections who will handle or assist sampling.

Due to the nature of the sampling material and facility sites all SWM personnel must have current 40 hr. Hazardous Waste Operations and Emergency Response (HAZWOPER) certification in order to take samples or be on the facility sites.

Staff should familiarize themselves with the selected analytical laboratory's recommended sample collection procedures and be familiar with specific instructions for lab tests.

Equipment, reagents, and supplies

- Coolers and ice.
- Nitrile gloves.
- Sample Tags.
- Chain of Custody seals.
- Appropriate sample bottles provided by analytical laboratory.
- Sample preservatives provided by laboratory.
- Ziploc bags.
- Sampling scoop and other specialized sampling equipment as needed.

Summary of procedure

Notify analytical laboratory of number of samples to be collected and type of analyses desired (e.g. Method 537M or TOP) in sufficient time before sampling event to allow receipt of sampling containers, forms, and other materials.

Inspect sample bottles to ensure that the correct bottles have been received, the appropriate preservatives have been provided, and that the bottles are in good condition. This should be done at least 2-3 days before sampling event, to provide time for laboratory to follow-up, if necessary.

Before embarking to sampling location, review facility safety plan and ensure that measures to minimize sample contamination discussed in section 5.7 are followed. Calibrate multi-parameter meter.

Steps for sampling are as follows:

1. Before embarking to sampling location, review facility safety plan.
2. Record information about sampling location in field notebook.
3. Prepare sample containers with labels.
4. Purge and decontaminate collection device, as needed.
5. Collect field parameters with multi-meter and record information in field notebook.
6. Fill to shoulder of sample jar.
7. Measure field parameters.
8. Fill out Chain of Custody form.
9. Package sample jars in container with ice.
10. Send samples to lab.

Appendix B. MDEQ Quick Reference Guide

Michigan Department of Environmental Quality

MDEQ PFAS Sampling Quick Reference Field Guide[1]

All Items Used During Sampling Event

Prohibited
• Items or materials that contain fluoropolymers such as ○ Polytetrafluoroethylene (PTFE), that includes the trademarks Teflon® and Hostaflon® ○ Polyvinylidene fluoride (PVDF), that includes the trademark Kynar® ○ Polycholotrifluoroethylene (PCTFE), that includes the trademark Neoflon ® ○ Ethylene-tetrafluoro-ethylene (ETFE), that includes the trademark Tefzel® ○ Fluorinated ethylene propylene (FEP), that includes the trademarks Teflon® FEP and Hostaflon® FEP • Items or materials that contain any other fluoropolymer

Pumps, Tubing, and Sampling Equipment

Prohibited	Allowable	Needs Screening[2]
• Items or materials containing any fluoropolymer (potential items include tubing, valves, or pipe thread seal tape)	• High-density polyethylene (HDPE) • Low-density polyethylene (LDPE) tubing • Polypropylene • Silicone • Stainless-steel • Any items used to secure sampling bottles made from: ○ Natural rubber ○ Nylon (cable ties) ○ Uncoated metal springs ○ Polyethylene	• Any items or materials that will come into direct contact with the sample that have **not** been verified to be PFAS-free ○ Do not assume that any sampling items or materials are PFAS-free based on composition alone

Sample Storage and Preservation

Prohibited	Allowable	Needs Screening[2]
• Polytetrafluoroethylene (PTFE): Teflon® lined bottles or caps	• Glass jars[4] • Laboratory-provided PFAS-Free bottles: ○ HDPE or polypropylene • Regular wet ice • Thin HDPE sheeting • LDPE resealable storage bags (i.e. Ziploc®) that will not contact the sample media[6]	• Aluminium foil[4] • Chemical or blue ice[5] • Plastic storage bags other than those listed as Allowable • Low-density polyethylene (LDPE) bottles

Field Documentation

Prohibited	Allowable	Needs Screening[2]
• Clipboards coated with PFAS • Notebooks made with PFAS treated paper • PFAS treated loose paper • PFAS treated adhesive paper products	• Loose paper (non-waterproof, non-recycled) • Rite in the Rain® notebooks • Aluminium, polypropylene, or Masonite field clipboards • Ballpoint pens, pencils, and Fine or Ultra-Fine Point Sharpie® markers	• Plastic clipboards, binders, or spiral hard cover notebooks • All markers not listed as Allowable • Post-It® Notes or other adhesive paper products • Waterproof field books

Decontamination

Prohibited	Allowable	Needs Screening[2]
• Decon 90® • PFAS treated paper towel	• Alconox®, Liquinox®, or Citranox® • Triple rinse with PFAS-free deionized water • Cotton cloth or untreated paper towel	• Municipal water • Recycled paper towels or chemically treated paper towels

www.michigan.gov/pfasresponse 800-662-9278 Revised 10/17/2018

Clothing, Boots, Rain Gear, and PPE

● Prohibited	■ Allowable	▲ Needs Screening[2]

• New or unwashed clothing
• Anything made of or with:
  ○ Gore-**Tex™ or other water**-resistant synthetics
• Anything applied with or recently washed with:
  ○ Fabric softeners
  ○ Fabric protectors, including UV protection
  ○ Insect resistant chemicals
  ○ Water, dirt, and/or stain resistant chemicals
	• Powderless nitrile gloves
	• Well-laundered synthetic or 100% cotton clothing, with most recent launderings not using fabric softeners
	• Made of or with:
	  ○ Polyurethane
	  ○ Polyvinyl chloride (PVC)
	  ○ Wax coated fabrics
	  ○ Rubber / Neoprene
	  ○ Uncoated Tyvek®
		• Latex gloves
		• Water and/or dirt resistant leather gloves
		• Any special gloves required by a HASP
		• Tyvek® suits, clothing that contains Tyvek®, or coated Tyvek®

Food and Beverages

● Prohibited	■ Allowable

• No food should be consumed in the staging or sampling areas, including pre-packaged food or snacks.
  ■ If consuming food on-site becomes necessary, move to the staging area and remove PPE. After eating, wash hands thoroughly and put on new PPE.
	• Brought and consumed only outside the vicinity of the sampling area:
	  ○ Bottled water
	  ○ Hydration drinks (i.e. Gatorade®, Powerade®)

Personal Care Products (PCPs) - for day of sample collection[6]

● Prohibited	■ Allowable	▲ Needs Screening[2]
• Any PCPs[6], sunscreen, and insect repellent applied in the sampling area.	PCPs[6], sunscreens, and insect repellents applied in the staging area, away from sampling bottles and equipment followed by thoroughly washing hands: **PCPs[6]:** • Cosmetics, deodorants/antiperspirants, moisturizers, hand creams, and other PCPs[6] **Sunscreens:** • Banana Boat® for Men Triple Defense Continuous Spray Sunscreen SPF 30 • Banana Boat® Sport Performance Coolzone Broad Spectrum SPF 30 • Banana Boat® Sport Performance Sunscreen Lotion Broad Spectrum SPF 30 • Banana Boat® Sport Performance Sunscreen Stick SPF 50 • Coppertone® Sunscreen Lotion Ultra Guard Broad Spectrum SPF 50 • Coppertone® Sport High Performance AccuSpray Sunscreen SPF 30 • Coppertone® Sunscreen Stick Kids SPF 55 • **L'Oréal**® Silky Sheer Face Lotion 50 • Meijer® Clear Zinc Sunscreen Lotion Broad Spectrum SPF 50 • Meijer® Sunscreen Continuous Spray Broad Spectrum SPF 30 • Meijer® Clear Zinc Sunscreen Lotion Broad Spectrum SPF 15, 30 and 50 • Meijer® Wet Skin Kids Sunscreen Continuous Spray Broad Spectrum SPF 70 • Neutrogena® Beach Defense Water+Sun Barrier Lotion SPF 70 • Neutrogena® Beach Defense Water+Sun Barrier Spray Broad Spectrum SPF 30 • Neutrogena® Pure & Free Baby Sunscreen Broad Spectrum SPF 60+ • Neutrogena® UltraSheer Dry-Touch Sunscreen Broad Spectrum SPF 30 **Insect Repellents:** • OFF® Deep Woods • Sawyer® Permethrin	• Products other than those listed as ■Allowable

[1] This table is not considered to be a complete listing of prohibited or allowable materials. All materials should be evaluated prior to use during sampling. The manufacturers of various products should be contacted in order to determine if PFAS was used in the production of any particular product.

[2] Equipment blank samples should be taken to verify these products are PFAS-free prior to use during sampling.

[3] **For surface water foam samples:** LDPE storage bags may be used in the sampling of foam on surface waters. In this instance, it is allowable for the LDPE bag to come into direct contact with the sample media.

[4] **For fish and other wildlife samples:** Depending on the project objectives, glass jars and aluminum foil might be used for PFAS sampling. PFAS has been found to bind to glass and if the sample is stored in a glass jar, a rinse of the jar is required during the sample analysis. PFAS are sometimes used as a protective layer for some aluminum foils. An equipment blank sample should be collected prior to any aluminum foil use.

[5] Regular ice is recommended as there are concerns that chemical and blue ice may not cool and maintain the sample at or below 42.8°F (6°C) (as determined by EPA 40 CFR 136 – NPDES) during collection and through transit to the laboratory.

[6] Based on evidence, avoidance of PCPs is considered to be precautionary because none have been documented as having cross-contaminated samples due to their use. However, if used, application of PCPs must be done at the staging area and away from sampling bottles and equipment, and hands must be thoroughly washed after the use of any PCPs prior to sampling.

●- Prohibited ■- Allowable ▲- Needs Screening

Appendix C. Glossaries, Acronyms, and Abbreviations

Glossary of General Terms

Conductivity: A measure of water's ability to conduct an electrical current. Conductivity is related to the concentration and charge of dissolved ions in water.

Effluent: An outflowing of water from a natural body of water or from a human-made structure. For example, the treated outflow from a wastewater treatment plant.

Landfill: A disposal facility or part of a facility at which solid waste is permanently placed in or on land including facilities that use solid waste as a component of fill.

Leachate: A liquid that has passed through or emerged from solid waste and contains soluble, suspended, or miscible materials removed from such waste.

pH: A measure of the acidity or alkalinity of water. A low pH value (0 to 7) indicates that an acidic condition is present, while a high pH (7 to 14) indicates a basic or alkaline condition. A pH of 7 is considered to be neutral. Since the pH scale is logarithmic, a water sample with a pH of 8 is ten times more basic than one with a pH of 7.

Pollution: Contamination or other alteration of the physical, chemical, or biological properties of any waters of the state. This includes change in temperature, taste, color, turbidity, or odor of the waters. It also includes discharge of any liquid, gaseous, solid, radioactive, or other substance into any waters of the state. This definition assumes that these changes will, or are likely to, create a nuisance or render such waters harmful, detrimental, or injurious to (1) public health, safety, or welfare, or (2) domestic, commercial, industrial, agricultural, recreational, or other legitimate beneficial uses, or (3) livestock, wild animals, birds, fish, or other aquatic life.

Total suspended solids (TSS): Portion of solids retained by a filter.

Acronyms and Abbreviations

CAP	Chemical Action Plan
CRO	Ecology's Central Regional Office
EAP	Ecology's Environmental Assessment Program
Ecology	Washington State Department of Ecology
EDD	Electronic data deliverable
EIM	Environmental Information Management database
EIS	Extracted Internal Standards
EPA	U.S. Environmental Protection Agency
ERO	Ecology's Eastern Regional Office
GIS	Geographic Information Software
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	High-Density Polyethylene resin
JHD	Jurisdictional Health Department
LC/MS-MS	Liquid Chromatography with tandem Mass Spectrometry
LP	Limited Purpose
MEL	Manchester Environmental Laboratory
MDEQ	Michigan Department of Environmental Quality
MQO	Measurement Quality Objective
MTCA	Model Toxics Control Act
MSW	Municipal Solid Waste
NA	Not Applicable
NWRO	Ecology's Northwest Regional Office
OPR	Ongoing Precision and Recovery
PFAS	Per- and Polyfluroalkyl substances
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctane sulfonates
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
SWM	Ecology's Solid Waste Management program
SWRO	Ecology's Southwest Regional Office
TBD	To be determined
TOP	Total organic precursors
WAC	Washington Administrative Code
WWTP	Wastewater Treatment Plant

Units of Measurement

ppt	Parts per Trillion. (1ppt = 1 nanogram/liter)

Quality Assurance Glossary

Accreditation: A certification process for laboratories, designed to evaluate and document a lab's ability to perform analytical methods and produce acceptable data. For Ecology, it is "Formal recognition by (Ecology)…that an environmental laboratory is capable of producing accurate analytical data." [WAC 173-50-040] (Kammin, 2010)

Accuracy: The degree to which a measured value agrees with the true value of the measured property. USEPA recommends that this term not be used, and that the terms *precision* and *bias* be used to convey the information associated with the term *accuracy*. (USGS, 1998)

Analyte: An element, ion, compound, or chemical moiety (pH, alkalinity) which is to be determined. The definition can be expanded to include organisms, e.g., fecal coliform, Klebsiella. (Kammin, 2010)

Bias: The difference between the sample mean and the true value. Bias usually describes a systematic difference reproducible over time and is characteristic of both the measurement system and the analyte(s) being measured. Bias is a commonly used data quality indicator (DQI). (Kammin, 2010; Ecology, 2004)

Blank: A synthetic sample, free of the analyte(s) of interest. For example, in water analysis, pure water is used for the blank. In chemical analysis, a blank is used to estimate the analytical response to all factors other than the analyte in the sample. In general, blanks are used to assess possible contamination or inadvertent introduction of analyte during various stages of the sampling and analytical process. (USGS, 1998)

Calibration: The process of establishing the relationship between the response of a measurement system and the concentration of the parameter being measured. (Ecology, 2004)

Check standard: A substance or reference material obtained from a source independent from the source of the calibration standard; used to assess bias for an analytical method. This is an obsolete term, and its use is highly discouraged. See Calibration Verification Standards, Lab Control Samples (LCS), Certified Reference Materials (CRM), and/or spiked blanks. These are all check standards but should be referred to by their actual designator, e.g., CRM, LCS. (Kammin, 2010; Ecology, 2004)

Comparability: The degree to which different methods, data sets and/or decisions agree or can be represented as similar; a data quality indicator. (USEPA, 1997)

Completeness: The amount of valid data obtained from a project compared to the planned amount. Usually expressed as a percentage. A data quality indicator. (USEPA, 1997)

Continuing Calibration Verification Standard (CCV): A quality control (QC) sample analyzed with samples to check for acceptable bias in the measurement system. The CCV is usually a midpoint calibration standard that is re-run at an established frequency during the course of an analytical run. (Kammin, 2010)

Control chart: A graphical representation of quality control results demonstrating the performance of an aspect of a measurement system. (Kammin, 2010; Ecology 2004)

Control limits: Statistical warning and action limits calculated based on control charts. Warning limits are generally set at +/- 2 standard deviations from the mean, action limits at +/- 3 standard deviations from the mean. (Kammin, 2010)

Data integrity: A qualitative DQI that evaluates the extent to which a data set contains data that is misrepresented, falsified, or deliberately misleading. (Kammin, 2010)

Data quality indicators (DQI): Commonly used measures of acceptability for environmental data. The principal DQIs are precision, bias, representativeness, comparability, completeness, sensitivity, and integrity. (USEPA, 2006)

Data quality objectives (DQO): Qualitative and quantitative statements derived from systematic planning processes that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.
(USEPA, 2006)

Data set: A grouping of samples organized by date, time, analyte, etc. (Kammin, 2010)

Data validation: An analyte-specific and sample-specific process that extends the evaluation of data beyond data verification to determine the usability of a specific data set. It involves a detailed examination of the data package, using both professional judgment and objective criteria, to determine whether the MQOs for precision, bias, and sensitivity have been met. It may also include an assessment of completeness, representativeness, comparability, and integrity, as these criteria relate to the usability of the data set. Ecology considers four key criteria to determine if data validation has actually occurred. These are:

- Use of raw or instrument data for evaluation.
- Use of third-party assessors.
- Data set is complex.
- Use of EPA Functional Guidelines or equivalent for review.

Examples of data types commonly validated would be:

- Gas Chromatography (GC).
- Gas Chromatography-Mass Spectrometry (GC-MS).
- Inductively Coupled Plasma (ICP).

The end result of a formal validation process is a determination of usability that assigns qualifiers to indicate usability status for every measurement result. These qualifiers include:

- No qualifier – data are usable for intended purposes.
- J (or a J variant) – data are estimated, may be usable, may be biased high or low.

- REJ – data are rejected, cannot be used for intended purposes.
  (Kammin, 2010; Ecology, 2004).

Data verification: Examination of a data set for errors or omissions, and assessment of the Data Quality Indicators related to that data set for compliance with acceptance criteria (MQOs). Verification is a detailed quality review of a data set. (Ecology, 2004)

Detection limit (limit of detection)**:** The concentration or amount of an analyte which can be determined to a specified level of certainty to be greater than zero. (Ecology, 2004)

Duplicate samples: Two samples taken from and representative of the same population, and carried through and steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variability of all method activities including sampling and analysis. (USEPA, 1997)

Field blank: A blank used to obtain information on contamination introduced during sample collection, storage, and transport. (Ecology, 2004)

Initial Calibration Verification Standard (ICV): A QC sample prepared independently of calibration standards and analyzed along with the samples to check for acceptable bias in the measurement system. The ICV is analyzed prior to the analysis of any samples. (Kammin, 2010)

Laboratory Control Sample (LCS): A sample of known composition prepared using contaminant-free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is prepared and analyzed in the same batch of regular samples using the same sample preparation method, reagents, and analytical methods employed for regular samples. (USEPA, 1997)

Matrix spike: A QC sample prepared by adding a known amount of the target analyte(s) to an aliquot of a sample to check for bias due to interference or matrix effects. (Ecology, 2004)

Measurement Quality Objectives (MQOs)**:** Performance or acceptance criteria for individual data quality indicators, usually including precision, bias, sensitivity, completeness, comparability, and representativeness. (USEPA, 2006)

Measurement result: A value obtained by performing the procedure described in a method. (Ecology, 2004)

Method: A formalized group of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, data analysis), systematically presented in the order in which they are to be executed. (EPA, 1997)

Method blank: A blank prepared to represent the sample matrix, prepared and analyzed with a batch of samples. A method blank will contain all reagents used in the preparation of a sample, and the same preparation process is used for the method blank and samples. (Ecology, 2004; Kammin, 2010)

Method Detection Limit (MDL): This definition for detection was first formally advanced in 40CFR 136, October 26, 1984 edition. MDL is defined there as the minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, and reported to be greater than zero. (Federal Register, October 26, 1984)

Percent Relative Standard Deviation (%RSD): A statistic used to evaluate precision in environmental analysis. It is determined in the following manner:

$$\%RSD = (100 * s)/x$$

where s is the sample standard deviation and x is the mean of results from more than two replicate samples. (Kammin, 2010)

Parameter: A specified characteristic of a population or sample. Also, an analyte or grouping of analytes. Benzene and nitrate + nitrite are all "parameters." (Kammin, 2010; Ecology, 2004)

Population: The hypothetical set of all possible observations of the type being investigated. (Ecology, 2004)

Precision: The extent of random variability among replicate measurements of the same property; a data quality indicator. (USGS, 1998)

Quality assurance (QA): A set of activities designed to establish and document the reliability and usability of measurement data. (Kammin, 2010)

Quality Assurance Project Plan (QAPP): A document that describes the objectives of a project, and the processes and activities necessary to develop data that will support those objectives. (Kammin, 2010; Ecology, 2004)

Quality control (QC): The routine application of measurement and statistical procedures to assess the accuracy of measurement data. (Ecology, 2004)

Relative Percent Difference (RPD): RPD is commonly used to evaluate precision. The following formula is used:

$$[Abs(a-b)/((a + b)/2)] * 100$$

where "Abs()" is absolute value and a and b are results for the two replicate samples. RPD can be used only with 2 values. Percent Relative Standard Deviation is (%RSD) is used if there are results for more than 2 replicate samples (Ecology, 2004).

Replicate samples: Two or more samples taken from the environment at the same time and place, using the same protocols. Replicates are used to estimate the random variability of the material sampled. (USGS, 1998)

Representativeness: The degree to which a sample reflects the population from which it is taken; a data quality indicator. (USGS, 1998)

Sample (field): A portion of a population (environmental entity) that is measured and assumed to represent the entire population. (USGS, 1998)

Sample (statistical): A finite part or subset of a statistical population. (USEPA, 1997)

Sensitivity: In general, denotes the rate at which the analytical response (e.g., absorbance, volume, meter reading) varies with the concentration of the parameter being determined. In a specialized sense, it has the same meaning as the detection limit. (Ecology, 2004)

Spiked blank: A specified amount of reagent blank fortified with a known mass of the target analyte(s); usually used to assess the recovery efficiency of the method. (USEPA, 1997)

Spiked sample: A sample prepared by adding a known mass of target analyte(s) to a specified amount of matrix sample for which an independent estimate of target analyte(s) concentration is available. Spiked samples can be used to determine the effect of the matrix on a method's recovery efficiency. (USEPA, 1997)

Split sample: A discrete sample subdivided into portions, usually duplicates (Kammin, 2010)

Standard Operating Procedure (SOP): A document which describes in detail a reproducible and repeatable organized activity. (Kammin, 2010)

Surrogate: For environmental chemistry, a surrogate is a substance with properties similar to those of the target analyte(s). Surrogates are unlikely to be native to environmental samples. They are added to environmental samples for quality control purposes, to track extraction efficiency and/or measure analyte recovery. Deuterated organic compounds are examples of surrogates commonly used in organic compound analysis. (Kammin, 2010)

Systematic planning: A step-wise process which develops a clear description of the goals and objectives of a project, and produces decisions on the type, quantity, and quality of data that will be needed to meet those goals and objectives. The DQO process is a specialized type of systematic planning. (USEPA, 2006)

References for QA Glossary

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