DEPARTMENT OF ECOLOGY
State of Washington

Quality Assurance Project Plan

Per- and Polyfluoroalkyl Substances (PFAS) in Washington State Products

Product Testing Program

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Each study conducted by the Washington State Department of Ecology (Ecology) must have an approved Quality Assurance Project Plan (QAPP). The plan describes the objectives of the study and the procedures to be followed to achieve those objectives. After completing the study, Ecology will post the final report of the study to the Internet.

This Quality Assurance Project Plan is available on Ecology's website at https://apps.ecology.wa.gov/publications/SummaryPages/1804012.html.

Data for this study will be available on the Product Testing Database (PTDB) website at http://ecyapeem/ptdbpublicreporting. Search Study: PFAS in Washington State Products – 2018

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Per- and Polyfluoroalkyl Substances (PFAS) in Washington State Products

Product Testing Program

June 2018

Approved by:

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HWTR: Hazardous Waste and Toxics Reduction

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2.0 Abstract

Per- and polyfluoroalkyl substances (PFAS) are a group of fluorinated substances with water-, stain-, and oil-resistant properties. They are widely used in consumer products and have been detected in humans, wildlife, and the environment.

Some applications of PFAS include:

- Stain and soil repellents for carpets and clothing textiles.
- Oil- and grease-resistance for food contact paper.
- Surfactants in firefighting foams.

PFAS are highly persistent in the environment and some bioaccumulate in humans. They are linked to health effects in human epidemiological studies, and in animal testing studies, several have proven toxic.

Washington State Department of Ecology will conduct this study to evaluate the presence of the following substances in 140 samples of state-purchased, children's, and general consumer products:

- Perfluorooctane sulfonic acid (PFOS)
- Perfluorooctanoic acid (PFOA)
- Additional PFAS, including related substances and precursors

This study is being carried out to support the:

- Development of Washington State's PFAS Chemical Action Plan.
- Children's Safe Products Reporting Rule, where the use of PFOS and PFOA in children's products are required to be reported to Ecology.
- Executive Order 04-01, which aims to reduce the use and purchase of products containing persistent, bioaccumulative, and toxic compounds by state agencies.

A final report summarizing the study results will be published.

3.0 Background

3.1 Introduction and problem statement

Per- and polyfluoroalkyl substances (PFAS) describe a class of synthetic organic chemicals not found naturally in the environment. Perfluoroalkyl substances contain a chain of fully-fluorinated carbon atoms. When a substance contains both fully-fluorinated carbon atoms and partially- or non-fluorinated carbon atoms, it is called a polyfluoroalkyl substance (Buck et al. 2011). They provide water, stain, and oil resistant properties that are widely used in consumer products and detected in humans, wildlife, and the environment (Buck et al. 2011). PFAS have been manufactured since the 1950s. Major international PFAS manufacturers worked with the EPA to voluntarily phase out two "long-chain" forms of perfluoroalkyl acids (PFAA), perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS), and their precursors and related higher homologues (OECD 2015).

PFAS are extremely persistent and have been detected in drinking water and environmental samples collected in Washington State (Ecology 2010, Dinglasan-Panlilio et al. 2013, Ecology 2017a, Hu et al. 2016). PFAS contamination can occur through direct release of chemicals from industrial use and from use and disposal of consumer products that contain them as impurities or intentional additions (Buck et al. 2011). Some PFAS compounds, referred to as precursors, can break down through degradation pathways into persistent PFAA terminal end products. These PFAA precursors are more commonly used commercially and can be released from consumer products and industrial raw materials (Buck et al. 2011). Some applications of PFAS include textile stain and soil repellents, oil and grease-resistant food contact paper, and as an active ingredient in firefighting foam (also called aqueous film-forming foam or AFFF).

The Washington State Department of Ecology (Ecology) and Department of Health (Health) are currently working on a Chemical Action Plan (CAP) to address PFAS (Ecology 2018a). CAPs identify the potential health and environmental effects of persistent, bioaccumulative, and toxic (PBT) chemicals on Washington's PBT list, including chemical degradation products and available substitutes, and recommend actions to reduce or eliminate those impacts (Chapter 173-333 Washington Administrative Code). PFOS and four PFOS salts were included on the original PBT list in 2006. Governor Gary Locke's Executive Order (EO) 04-01 requires state agencies to reduce the use and purchase of products that contain PBT compounds.

The Washington State Children's Safe Products Act (CSPA) requires manufacturers to annually report the presence of chemicals of high concern to children (CHCC) in children's products to Ecology. The Children's Safe Products Reporting Rule identifies 85 CHCC for which reporting is required when they are present in children's products either intentionally or as a contaminant. PFOS and its salts and PFOA and related substances are included in the list of 85 CHCC (WAC 173-334-130).

This study will evaluate the presence of PFOS, PFOA, and additional PFAS that include related substances and precursors in children's, state-purchased, and general consumer products in Washington State. Product categories in this study may include:

- aqueous film-forming foam (AFFF)
- carpet, rugs, and care and treatment products
- food contact material
- clothing and fabric textiles
- cosmetics and personal care products
- building and exterior-use products
- janitorial, medical, and maintenance products
- industrial use products

A modified version of EPA analytical method 537 version 1.1 will be used for testing product component samples. EPA method 537 version 1.1 has been used to test for a limited suite of PFAS in drinking water and has been used for regulatory purposes (EPA 2009a). Modified versions of EPA 537.1 have been used to test for analytes in other environmental media and also products. Product component samples will be analyzed for 29 PFAS analytes that include perfluoroalkyl sulfonic acids (PFSA), perfluoroalkyl carboxylic acids (PFCA), perfluoroalkane sulfonamides (FASA), and fluorotelomer sulfonic acids (FTS). The total oxidizable precursor (TOP) assay will be performed to estimate the quantity of any unidentified PFAS precursors in the product (Houtz and Sedlak 2012).

3.2 Study area and surroundings

General consumer and children's products available to Washington State residents and businesses either in-store or online will be assessed for inclusion in this study. Products purchased in-store are frequently purchased in the South Puget Sound region. The practice of purchasing products from larger chain stores and online is used to generally reflect merchandise sold and available to customers across Washington State.

State-purchased products will be obtained from contract vendors and contractors administered by Washington State Department of Enterprise Services (DES). In addition, Ecology staff will collect products from individual Washington State agencies that have purchased from contracted vendors.

3.2.1 History of study area

Products purchased during the product collection event are limited to selection of currently available products offered for sale in Washington stores, online, and for use at state agencies at the time of the sampling event.

3.2.2 Summary of previous studies and existing data

PFOS, PFOA and additional PFAS that include related substances and precursors have been detected in consumer products and reported in literature (e.g. EPA 2009c, Herzke et al. 2012, Fujii et al 2013, D'Agostino and Mabury 2014, Barzen-Hanson and Field 2015, Kotthoff et al. 2015, Liu et al. 2015, KEMI 2015, Barzen-Hanson et al. 2017, Robel et al. 2017, Schaider et al. 2017).

Ecology's product testing program has previously supported investigational research with academic labs into the presence of PFAS in consumer products. These academic labs have published independent work that included products from Washington State (Schaider et al. 2017 and Robel et al. 2017).

Schaider et al. (2017)

In a study published in 2017, 407 samples of food packaging from fast food restaurants across the United States were screened for fluorinated chemicals using particle-induced gamma ray emission (PIGE) spectroscopy (Schaider et al. 2017). PIGE screens for total fluorine in the sample and does not differentiate between organic and inorganic fluorine. Samples, including samples from Washington State, were collected from 2014 to 2015 and from 6 product categories: food contact paper like wrappers and bags (248), food contact paperboards (80), paper cups (30), other beverage containers (25), non-contact paper like the outer bags (15), and miscellaneous samples like lids and food containers (9).

In the food contact paper category, 46% of samples had detectable levels of total fluorine (F) concentrations. Twenty percent (20%) of samples in the food contact paperboard category were found to have detectable total F levels. The other beverage containers category were found to have detectable total F concentrations in 16% of the samples. The samples from the non-contact papers, paper cups, and miscellaneous product categories did not have any detectable total F concentrations. Overall, 33% of the 407 samples had detectable total F concentrations and the range was from 16 to 800 nanomoles of F per square centimeter (nmol F/cm^2). Fluorine was more frequently detected in grease-proof products like contact papers than in products that hold liquids or are not intended to come into contact with food.

A subset of 20 samples were analyzed for 89 PFAS compounds by liquid chromatography/time-of-flight mass spectrometry (LC/TOF MS) to provide more information on the specific PFAS present in the food packaging and to validate the PIGE screening results. Samples selected for analysis consisted of 14 samples with a total F concentration over 200 nmol of F/cm^2 and 6 samples with a total F concentration below the limit of detection (LOD). The total peak area for PFAS was higher in 70% of samples (10 of 14) with a total F level greater than 200 nmol/cm^2 by PIGE than peak areas in 6 samples with a total F concentration below the LOD. The most frequently detected PFAS compounds by LC/TOF MS analysis were perfluorohexanoic acid

(PFHxA, 7 out of 20 samples), perfluorobutanoic acid (PFBS, 7 samples), PFOA (6 samples), 2H-perfluoro-2-octenoic acid (FHUEA, 6 samples), and perfluoropentanoic acid (PFPeA, 5 samples).

Robel et al. (2017)

Another study published in 2017 investigated the mass balance of fluorine on paper and textiles by comparing the sum of volatile PFAS, ionic PFAS, unknown PFAS precursors from the TOP assay, and total fluorine after solvent extraction for analysis to initial total fluorine before solvent extraction (Robel et al 2017). Four volatile PFAS were analyzed by GC-MS and 73 ionic PFAS were analyzed by LC-MS/MS. The TOP assay was used to quantify the total concentration of unknown precursors (determined as the net production of PFCAs by oxidative conversion) not identified and measured as individual PFAS precursor analytes. Concentrations were quantified for these three groups of PFAS for each sample, converted to nmol F/cm^2, and summed. Paper and textile samples were screened for total fluorine (nmol F/cm^2) as measured by PIGE before samples were extracted for analysis of volatile, ionic, and unknown precursor PFAS and after solvent extraction to determine the total fluorine that remained associated with the product component sample.

Samples from paper and textile product categories included: food contact paper (5), popcorn bag (1), gift bag (1), copy paper (1), jacket (4), children's fabric (2), hat (1), pillowcase (1), and office upholstery (1). Samples were from products in Washington State purchased in 2015 except the office upholstery sample from a used office chair with a manufacture date of 1988.

In the product component samples, volatile PFAS were found to account for 0-2.2%, ionic PFAS for 0-0.41%, and unknown PFAS precursors for 0.021-14% of the total nmol F/cm^2 determined by PIGE. Paper and textile samples retained 64 ± 28% to 110 ± 30% of the initial total fluorine (nmol F/cm^2) measured by PIGE after solvent extraction for PFAS analysis. The study demonstrated the majority of fluorine remains associated with the papers and textiles and is not readily extracted by the solvents. The fluorine that remains on the product most likely is comprised of polymeric substances since a large volume of manufactured PFAS is for the production of polymers. The solvent-extractable volatile, ionic and unknown precursors PFAS may be a source of human exposure while the fluorine that remains associated on the product may act as a long-term source of persistent PFAS to the environment.

3.2.3 Parameters of interest and potential sources

This study will analyze 29 PFAS analytes for which an analytical method has been established and demonstration of capability provided by the contract laboratory to test a variety of products available in Washington State. Table 5 (Section 7.2.3) shows the PFAS groups and individual analytes in each group.

Perfluoroalkyl acids (PFAA) that include 8 perfluoroalkyl sulfonic acids (PFSA) and 11 perfluoroalkyl carboxylic acids (PFCA) will be analyzed in all product component samples. PFOS is a PFSA with 8 carbons and PFOA is the 8 carbon chain PFCA. Additional PFSA and PFCA for analysis consist of a similar chain structure with the carbon chain length ranging between 4 and 14 carbons. Long-chain PFAA (PFSA with 6 or more carbons and PFCA with 8 or more carbons) are widespread in the environment, highly persistent, bioaccumulative, toxic in animal studies, and detected in humans (EPA 2009b).

Some PFAS break down through biotic (living) and abiotic (non-living) degradation pathways into the terminal persistent PFCA and PFSA end products (Buck et al. 2011). These compounds are referred to as PFAS precursors. Several PFAS precursors will also be analyzed in this study and include 7 perfluoroalkane sulfonamides (FASA) and 3 fluorotelomer sulfonic acids (FTS).

Perfluoroalkane sulfonamides and fluorotelomers are used in products as raw materials for surface protection products and surfactants (Buck et al. 2011). They are used to make polymers that impart soil, stain, grease, and water resistance to coated products like textiles and food contact material. They are also used in product formulations as surfactants in fire-fighting foams, paints, coatings, metal plating applications, and cleaning products.

3.2.4 Regulatory criteria or standards

Information provided in this section focuses on the regulatory environment for Washington State and for products. For more information on regulations outside of Washington and in varying media, the Interstate Technology Regulatory Council (ITRC) has developed several fact sheets on PFAS including a fact sheet on “Regulations, Guidance, and Advisories for Per- and Polyfluoroalkyl Substances (PFAS)” (ITRC 2018).

The Washington State Children’s Safe Products Act (CSPA) requires manufacturers to annually report the presence of chemicals of high concern to children (CHCC) in children’s products to Ecology. The reporting rule identifies 85 CHCC for which reporting is required when they are present in children’s products either intentionally or as a contaminant. PFOS and its salts and PFOA and related substances are included in the list of 85 CHCC (WAC 173-334-130). Ecology has required the reporting of PFOS in children’s products since 2012 and PFOA will be reported starting in January 2019. Ecology’s product testing program tests products for chemicals to ensure manufacturers are reporting accurate information.

PFOS and four PFOS salts are identified in Washington State’s Persistent, Bioaccumulative, and Toxic (PBT) List (WAC 173-333-310). This rule defines the process for developing chemical action plans and identifies the list of PBTs.

Washington State’s Executive Order 04-01 requires state agencies to reduce the use and purchase of products that contain PBTs.

4.0 Project Description

This study focuses on the investigation of PFOS, PFOA, and additional PFAS that include related substances and precursors that may be present in products for children, general consumers, and available for purchase on Washington State contracts.

4.1 Project goals

This study is being conducted with the following goals:

- To identify PFAS in children's, general consumer, and state-purchased products in Washington State with a focus on PFOS and PFOA.
- To evaluate compliance with the Children's Safe Products Reporting Rule for PFOS and PFOA reporting.
- To provide data for the PFAS Chemical Action Plan (Ecology 2018a).

4.2 Project objectives

The following objectives will be carried out to meet study goals:

- Purchase and collect approximately 140 products available for sale in stores and online in Washington State, purchased by Washington State agencies, and available to purchase on Washington State contracts.
- Analyze 140 product components (samples) from selected products for PFOS, PFOA, and additional PFAS.

4.3 Information needed and sources

Products will be identified based on research of manufacturing processes of products, known to be present in similar products, and from sources such as Safety Data Sheets (SDS), ingredient labels, public databases (including the CSPA Manufacturer Reporting Database and Washington State Department of Enterprise Services contracts website), and peer-reviewed journal articles.

4.4 Tasks required

The following tasks will be carried out for this study:

- Conduct research to identify priority products likely to contain PFOS, PFOA, and additional PFAS.
- Work with Ecology's Manchester Environmental Laboratory (MEL) to secure a contract laboratory for analysis of PFAS in products.

- Work with Ecology's Laboratory Accreditation Unit (LAU) to have the contract lab become accredited for analysis of PFAS in a solid matrix using a modification of EPA Method 537 version 1.1 and obtain a waiver from the lab accreditation requirement for the TOP assay.
- Purchase general consumer and children's products directly from retail stores within Washington State and from online stores and vendors.
- Collect state purchased products from Washington government agencies if available (e.g. state, county, city, public schools, and state universities).
- Purchase products available on Washington State contracts directly from the vendors.
- Record product information in Ecology's Product Testing Database (PTDB).
- Separate products into product components and catalog the components in the PTDB.
- Select samples from product components for laboratory analysis.
- Prepare and submit samples for contract laboratory analysis of PFAS using a modified EPA Method 537 version 1.1.
- Review data quality of laboratory results and work with MEL QA Coordinator to resolve any issues.
- Enter PFAS laboratory results in the PTDB.
- Analyze study data and write technical report.

4.5 Systematic planning process used

This Quality Assurance Project Plan (QAPP) and any subsequent Addendum to this QAPP addresses suitable systematic planning for the specific study.

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Table 1. Organization of Study Staff and Responsibilities

Staff	Title	Responsibilities
Kara Steward RTT Unit HWTR Program Phone: 360-407-6250	PFAS CAP Lead and Client	Clarifies scope of the project. Provides internal review of the QAPP and approves the final QAPP.
Kari Trumbull RTT Unit HWTR Program Phone: 360-407-6093	Project Manager and Principal Investigator	Writes QAPP. Oversees and leads product collection, sample processing, chain-of-custody, and transportation of samples to the laboratory. Conducts QA review of data, analyzes and interprets data, and enters data into PTDB. Writes the draft report and final report.
Chrissy Wiseman RTT Unit HWTR Program Phone: 360-407-7672	Product Testing Sampling Assistant	Assists with product purchase and collection, logs product info into PTDB, sample processing, chain-of-custody, and PTDB records review.
Sean Smith RTT Unit HWTR Program Phone: 360-407-7609	Unit Supervisor for the Project Manager	Provides internal review of the QAPP, approves the budget, and approves the final QAPP.
Ken Zarker P2RA Section HWTR Program Phone: 360-407-6724	Section Manager for the Project Manager	Reviews the project scope and budget, tracks progress, reviews the draft QAPP, and approves the final QAPP.
Ginna Grepo-Grove MEL Phone: 360-871-8829	Quality Assurance Coordinator	Management of lab contract. Conducts QA review and validation of data.
Alan Rue MEL Phone: 360-871-8801	Director	Reviews and approves the final QAPP.
Tom Gries Phone: 360-407-6327	Acting Ecology Quality Assurance Officer	Reviews the draft QAPP and approves the final QAPP. Approves 'Request to Waive Requirement to Use Accredited Lab.'

HWTR: Hazardous Waste and Toxics Reduction
RTT: Reducing Toxic Threats
P2RA: Pollution Prevention and Regulatory Assistance
QAPP: Quality Assurance Project Plan
PTDB: Product Testing Database
MEL: Manchester Environmental Laboratory

5.2 Special training and certifications

Ecology staff assisting on product testing studies will have undergone training documented by the completion of the Product Testing Preparation Staff Training Checklist. Training includes reviewing the study-specific QAPP, SDSs, current approved product testing standard operating procedures (SOPs), and the location of personal protective equipment and safety equipment (e.g. first aid kit, eye wash station). Guidance for product testing training is outlined in Ecology's Product Testing SOP for Sample Collection and Processing, Version 1.0 (Ecology 2018b).

Contract laboratory staff and organic chemistry analysts will have appropriate experience using LC-MS/MS to analyze PFAS analytes in environmental and product media.

5.3 Organization chart

Table 1 lists the key individuals and responsibilities.

5.4 Proposed project schedule

Table 2a. Proposed Schedule for Completing Product Collection and Laboratory Work

Product Collection, Processing, and Laboratory Work	**Due Date**	**Lead Staff**
Product purchase completion	August 2018	Kari Trumbull Chrissy Wiseman
Product logging completion	August 2018	Kari Trumbull Chrissy Wiseman
Internal data QA completion	September 2018	Kari Trumbull Chrissy Wiseman
Laboratory analyses completion and receipt: First set 70 samples Second set of 70 samples	 August 2018 October 2018	Contract Laboratory

Table 3b. Proposed Schedule for Completing Data Reviews and Data Entry into the PTDB

Lab Data and PTDB Review	**Due Date**	**Lead Staff**
PTDB Study Name: PFAS in Washington State Products 2018	---	---
Lab data validated	December 2018	MEL QA Coordinator
Lab data QA reviewed	February 2019	Kari Trumbull
Lab data loaded in PTDB	February 2019	Kari Trumbull
PTDB data QA reviewed	February 2019	Chrissy Wiseman
Lab data to CSPA Compliance Coordinator	February 2019	Kari Trumbull

QA: Quality Assurance PTDB: Product Testing Database CSPA: Children's Safe Products Act

Table 4c. Proposed Schedule for Completing Final Report

Final Report Schedule	Due Date	Lead Staff
Final report draft due to supervisor	April 2019	Kari Trumbull
Final report draft due to client/peer reviewer	April 2019	Kari Trumbull
Final report draft due to external reviewer(s)	April 2019	Kari Trumbull
All reviews completed: Final report due to Publications Coordinator	May 2019	Kari Trumbull
Final report due on web	June 2019	Publications Coordinator

5.5 Budget and funding

The estimated total budget for product collection and laboratory analysis (including contracting services) for this study is displayed in Table 3. Product collection and sample processing quality control (QC) samples are included in the number of samples. Some product component samples may be used from previous product testing studies. The cost of these samples are not included in the budget for this study. The number of QC samples includes those that cost a laboratory fee and are not provided free of charge by the laboratory.

Table 5. Study Budget and Funding

Activity/Parameter	Number of Samples	Number of QC Samples[+]	Cost of Sample	Subtotal	Total
Product Collection[#]	140	---	---	$ 3,650	---
				Product Collection Total:	$ 3,650
PFAS Analysis of 29 Analytes (Pre-Oxidation of Top Assay)	140	9	$ 425	$ 63,325	---
TOP Assay with PFAS Analysis of 19 Analytes Post-Oxidation	140	9	$ 495	$ 73,755	---
MEL Contract Fee (25%)	---	---	---	$ 34,270	---
				Laboratory Total:	$ 171,350
				Study Total:	$ 175,000

[+]QC samples in this table include those that are not provided free of charge (an additional duplicate in each batch, sample processing rinsates).

[#]Estimated cost is for purchase of new products, some products may be used from previous product testing studies. One product can result in multiple component samples.

6.0 Quality Objectives

6.1 Data quality objectives (DQOs)

This study will not require data quality objectives (also referred to as decision quality objectives).

6.2 Measurement quality objectives (MQOs)

The measurement quality objectives (MQOs) for analysis of PFAS analytes in this study, expressed in terms of acceptable precision, bias, instrument performance, and sensitivity, are shown in Table 4. MQOs may vary depending on the individual PFAS analyte and type of matrix.

Table 6. Measurement Quality Objectives

Analyte	LCS Duplicates (RPD)^,*	Sample Duplicates (RPD)^,*	LCS (recovery)*	Method Blanks*	Surrogate Standards (recovery)*	Quantitation Limit*
PFAS analytes	Less than 40%	Less than 40%	50–150%	Less than 0.5 ppb–less than 10 ppb	50–150%	1.0 ppb–20 ppb

RPD: relative percent difference.
LCS: laboratory control sample.
ppb: parts per billion.
^RPD for concentrations greater than 5 times reporting limit.
*Method acceptance limits are not well established for product matrices. Values provided represent the preferred maximum limits.

6.2.1 Targets for precision, bias, and sensitivity

6.2.1.1 Precision

Precision is a measure of the variability in the results of replicate measurements due to random error. Laboratory analysis precision will be assessed through laboratory duplicate samples for all matrices and analyses. Table 4 shows MQOs for laboratory control sample duplicates and extracted sample duplicates.

6.2.1.2 Bias

Bias is the difference between the sample mean and the true value. Laboratory analysis bias will be assessed through laboratory control samples. MQOs for percent recoveries are shown in Table 4.

6.2.1.3 Sensitivity

Sensitivity is a measure of the capability of a method to detect a substance. Laboratory analysis sensitivity is defined here as the quantitation limit. See Table 4 for quantitation limit.

6.2.2 Targets for comparability, representativeness, and completeness

6.2.2.1 Comparability

Comparability will be ensured by implementing standardized procedures for sampling and analysis.

Data from this study can be compared to publically available data of similar product types and analyzed using substantially the same analytical methods, if available.

6.2.2.2 Representativeness

Products purchased and collected for this study will be representative of those available to Washington State residents and agencies.

6.2.2.3 Completeness

The project manager will consider the study to have achieved completeness if 95% of the laboratory samples are analyzed acceptably.

6.3 Acceptance criteria for quality of existing data

Not applicable to this study.

6.4 Model quality objectives

Not applicable to this study.

7.0 Study Design

7.1 Study boundaries

Products will be purchased and collected from retail stores, online through internet retailers, and from state agency contracts available in Washington State. Products likely to contain PFAS compounds will be selected from the product categories outlined in section 7.2. Products will include imported products, particularly from countries that may still produce PFOS, PFOA, and other related long-chain PFAS. Some products may be purchased and included in the study that advertise fluorine-free or no PFOA or PFOS in the product.

Some previously purchased products from prior product testing studies may be used in this current study. Product component samples will be collected from a component of the original product stored at Ecology. The use of previously purchased products in this study has been approved by the Ecology QA Officer.

Products purchased and collected will be documented in the product testing database (section 8.5), processed into product component samples (section 9.2), then shipped to the contract laboratory for PFAS analysis.

All 29 PFAS analytes will be tested for their presence after solvent extraction from the product component sample and liquid chromatography with tandem mass spectrometry (LC-MS/MS) analysis at the contract laboratory. The total oxidizable precursor (TOP) assay will be performed to provide a qualitative assessment for the presence of unidentified PFAS precursors in the product component sample (Houtz and Sedlak 2012).

7.2 Sample collection

Products will be purchased or collected from one or more of the following product categories:

- **Aqueous film-forming foam (AFFF):** AFFFs are used primarily in Class B foams to extinguish fires containing flammable liquids. Some formulations contain PFAS as an active ingredient (Herzke et al. 2012, D'Agostino et al. 2014, Barzen-Hanson et al. 2015, Barzen-Hanson and Field 2017). Application of AFFF containing PFAS at a fire releases the foam to the surrounding ground, releasing PFAS to soil, surface water, or groundwater.
- **Carpets and rugs and their care and treatment products:** Carpets and rugs are frequently treated with PFAS to provide soil and stain resistance (EPA 2009c, Herzke et al. 2012, Kotthoff et al. 2015). In addition, after-market products containing PFAS are available to consumers and commercial carpet cleaners to treat carpet, rugs, and upholstery for soil and stain resistance.

- **Food contact material:** PFAS are used in some food contact material to provide oil and grease resistance and have previously been detected in products available in Washington State (Schaider et al. 2017, Robel et al. 2017).
- **Clothing and fabric textiles and their care and treatment products:** PFAS are used to treat clothing and outdoor products such as coats, gloves, umbrellas, tents, and shoes in order to repel water, oil, and dirt (EPA 2009c, Herzke et al. 2012, Kotthoff et al. 2015). PFAS has previously been detected in children's and general consumer clothing from Washington State (Robel et al. 2017). In addition, after-market products containing PFAS are available to consumers to treat clothing, accessories, and upholstery for water, soil, and stain resistance.
- **Cosmetics and personal care products:** PFAS are used in cosmetic and personal care consumer products that are applied directly on the skin, hair, or mouth (Fujii et al 2013, KEMI 2015). PFAS compounds are used in these products for a variety of functions such as anticaking agents, emulsifiers, film forming, surfactants, and solvents.
- **Building and exterior-use products:** PFAS are used in paints, adhesives, and sealants for chemical resistance, to reduce surface tension which improves paint adhesion, and for oil and water repellency (EPA 2009c, Herzke et al. 2012, KEMI 2015).
- **Janitorial, medical, and maintenance products**: PFAS are used in various products like window polish, floor polish, floor waxes, medical garments, ski waxes, and car care products (EPA 2009c, Liu et al. 2015, Kotthoff et al 2015).
- **Industrial use products**: PFAS are used in industrial formulations and applications such as mist suppressants for metal plating and finishing operations, industrial surfactants, resins, molds, plastics, automotive fluids, hydraulic systems in the aviation industry, and engineered coatings in semiconductor production (EPA 2009d, Herzke et al. 2012, KEMI 2015).

7.2.1 Equity considerations in sample collection

Ecology is committed to the principles of equity and environmental justice and shares the EPA's goal "to provide an environment where all people enjoy the same degree of protection from environmental and health hazards and equal access to the decision-making process to maintain a healthy environment in which to live, learn, and work" (Ecology 2017b).

Products purchased and collected in this study will include products that are accessible and/or relevant to diverse ethnic, cultural, and economic groups in Washington State.

7.2.2 Sampling locations and frequency

Products identified for use in this study will be purchased from retail stores, online through internet retailers, and from state agency procurement. Products will also be collected from state agencies which purchased from Washington State contracts. In-store purchases and site collections will be coordinated to minimize the frequency of product collection events. Locations of products purchased and collected will be recorded in the Product Documentation Log (section 8.7) and in the product testing database.

7.2.3 Sample parameters and laboratory analytes to be measured

The PFAS parameters to be analyzed in product component samples for this study are listed in Table 5. For the carboxylate and sulfonate PFAS analytes, the analyte concentrations are reported as the anion form.

Table 7. Target PFAS Groups and Individual PFAS Analytes for Laboratory Analysis

PFAS Group	Individual PFAS Analyte	PFAS Acronym
Perfluoroalkyl Sulfonic Acids/Sulfonates (PFSA)	Perfluorobutane sulfonate*	PFBS
	Perfluoropentane sulfonate *	PFPeS
	Perfluorohexane sulfonate*	PFHxS
	Perfluoroheptane sulfonate*	PFHpS
	Perfluorooctane sulfonate*	PFOS
	Perfluorononane sulfonate*	PFNS
	Perfluorodecane sulfonate*	PFDS
	Perfluorododecane sulfonate*	PFDoS
Perfluoroalkyl Carboxylic Acids/Carboxylates (PFCA)	Perfluorobutanoate*	PFBA
	Perfluoropentanoate*	PFPeA
	Perfluorohexanoate*	PFHxA
	Perfluoroheptanoate*	PFHpA
	Perfluorooctanoate*	PFOA
	Perfluorononanoate*	PFNA
	Perfluorodecanoate*	PFDA
	Perfluoroundecanoate*	PFUnDA
	Perfluorododecanoate*	PFDoDA
	Perfluorotridecanoate*	PFTrDA
	Perfluorotetradecanoate*	PFTeDA
Perfluoroalkane Sulfonamides (FASA)	Perfluorooctane sulfonamide	PFOSA
	N-Methyl perfluorooctane sulfonamide	MeFOSA
	N-Ethyl perfluorooctane sulfonamide	EtFOSA
	N-Methyl perfluorooctane sulfonamidoethanol	MeFOSE
	N-Ethyl perfluorooctane sulfonamidoethanol	EtFOSE
	N-Methyl perfluorooctane sulfonamido acetic acid	MeFOSAA
	N-Ethyl perfluorooctane sulfonamido acetic acid	EtFOSAA
Fluorotelomer Sulfonic Acids/Sulfonates (FTS)	4:2 Fluorotelomer sulfonate	4:2 FTS
	6:2 Fluorotelomer sulfonate	6:2 FTS
	8:2 Fluorotelomer sulfonate	8:2 FTS

*Analytes analyzed post-oxidation of TOP assay.

7.3 Modeling and analysis design

Not applicable to this study.

7.3.1 Analytical framework

Not applicable to this study.

7.3.2 Model setup and data needs

Not applicable to this study.

7.4 Assumptions in relation to objectives and study area

Products used in this study reflect current, on the market products (products purchased between 2017 and 2018), and not previous in-use products that consumers have exposure to. Manufacturing formulations are subject to change in response to changes in the regulatory environment. The profile of PFAS in products has evolved substantially over the last decade due to changes in the regulatory requirements and from voluntary reduction measures.

The analytical techniques in this study address a limited suite of PFAS analytes. As a result, the absence of a particular PFAS compound in a particular product component sample does not necessarily mean that fluorinated compounds are not used in the product. Some PFAS precursors in product component samples simply cannot be detected and quantified by EPA method 537.1 and modified versions. In an effort to quantify the contribution of PFAS precursors, this study includes the total oxidizable precursor (TOP) assay. The TOP assay oxidation can convert some PFAS precursors, which would otherwise go undetected, to quantifiable analytes (i.e., those in Table 5).

7.5 Possible challenges and contingencies

A limited suite of PFAS analytes tested provides limited information on PFAS presence and use in products. Analytical challenges for testing PFAS analytes may occur due to the complex matrices of products.

7.5.1 Logistical problems

Limits on the selection of products available during product purchase and collection event may require adding additional sampling events.

7.5.2 Practical constraints

See sections 7.5.1 logistical problems and 7.5.3 schedule limitations.

7.5.3 Schedule limitations

Limitations of product testing sampling and processing staff and laboratory analysis of complex product matrices may impact the proposed study schedule.

8.0 Sampling Procedures

8.1 Invasive species evaluation

Not applicable to this study.

8.2 Measurement and sampling procedures

Guidance for product collection, sample processing, and recording product and sample component data in the product testing database is provided in Ecology's Product Testing SOPs:

- Ecology's Product Testing SOP for Sample Collection and Processing, Version 1.0 (Ecology 2018b)
- Ecology's Product Testing SOP for Data Entry and Database, Version 1.0 (Ecology 2018c)

Ecology product testing staff will take a product collection tote containing the necessary tools and equipment to be used for the sample collection event. Tote will include Product Documentation Log, large, medium, and small plastic bags to store products (instead of paper bags the store may provide), pens, camera, decontaminated tools for collecting aliquots of product component samples (if applicable), and gloves. Clothing, footwear, and accessories that may contain PFAS chemicals will not be worn by product testing personnel during product collection events and sample processing. It is also recommended that cosmetics and personal care products not be used on days of product collection and sample processing.

Field blanks and trip blanks will be collected during product collection. Field blanks consist of a piece of copy paper (no recycled content) placed into a new quart-sized resealable plastic bag and the bag will never be opened during product collection. Trip blanks consist of a piece of copy paper (no recycled content) placed into a new quart-sized resealable plastic bag and the bag will be opened during product collection. One field blank and one trip blank will be collected for each product collection day which could include multiple site locations. These blanks will be handled exactly the same as other product samples, e.g., during product processing, and will be submitted to the contract lab for analysis.

Products purchased in stores will be brought back to the Ecology product testing processing room. Purchase or collection event will be documented and recorded in the product testing database. Products will be photographed, recorded into the product testing database, placed in individual plastic bags with product testing ID (see section 8.5), and stored in totes in locked cabinets. Gloves will be worn (and a new pair replaced between individual products) when

separating, recording, and photographing products. Products will be placed on a new piece of plain aluminum foil[1] (dull side up) when not stored in individual plastic bags.

The processing and preparation of products into product component samples for laboratory analysis is discussed in the sample preparation methods section (section 9.2) of this QAPP.

8.3 Containers, preservation methods, holding times

Hand-reduced and aliquoted laboratory samples will be stored in certified clean brown wide-mouth high density polyethylene (HDPE) jars. Laboratory samples consisting of homogenous mixtures and oils will be stored in original product containers (if not aliquoted). All samples will be stored in the dark and kept at ambient temperatures.

Table 8. Sample Containers, Preservation, and Holding Times

Parameter	Matrix	Minimum Quantity Required	Container	Sample Receipt and Preservative*	Sample Storage	Estimated Holding Time*
PFAS analytes	paper and textile solids; powder homogeneous mixtures	5 grams	HDPE jar or original unopened product container	minimize exposure to light, keep at ambient temperature	stored in dark location at ambient temperature	1 year
PFAS analytes	liquid, gel, and cream homogeneous mixtures	100 milliliters	HDPE jar or original unopened product container	minimize exposure to light, keep at ambient temperature	stored in dark location at ambient temperature	1 year
PFAS analytes	oils	5 grams	HDPE jar or original unopened product container	minimize exposure to light, keep at ambient temperature	stored in dark location at ambient temperature	1 year

*No demonstrated maximum holding times or preservation methods have been established for product matrices.

8.4 Equipment decontamination

All tools used in the preparation of solid product component samples and collection of aliquots of samples will be decontaminated following a low-level tool cleaning procedure specific to testing for PFAS analytes. The procedure is outlined below.

[1] Aluminum foil is listed as a sampling and lab supply that should not be used for PFAS collection and analysis. PFAS analytes can be potentially transferred from the aluminum foil if contaminated with PFAS (EPA 2009a). Aluminum foil used in this study will be tested as a QC processing sample (see Section 9.2).

Product Testing Tool Cleaning and Decontamination for PFAS analysis:

- Wear appropriate PPE (e.g. gloves, eyewear, and lab coat) while cleaning tools. Nitrile gloves are required for all tool decontamination processes and silver shield gloves can be worn in addition to nitrile gloves when handling methanol.
- Transport cleaning supplies and tools to the sink using a cart if needed.
- Set up a drying location for clean tools by placing a large piece of aluminum foil (see Footnote 1, Section 8.2) covered with KimWipes®, or other pretested absorbent towels, on the countertop.
- Replace wipes, gloves, and aluminum foil as necessary throughout the process to prevent cross contamination.
- Wash the prep room clean tool bin with Liquinox® and potable water and rinse with ethanol prior to use and line with aluminum foil (dull side up). Use this bin to transport and store clean tools.
- Prepare a diluted 1% solution of 10 ml cleaning agent (Liquinox®) to 1-Liter potable water in pre-labeled product testing soap wash bottle.
- Follow steps 1 through 5 to clean one tool at a time:
    - Step 1: Squirt a small amount of the cleaning solution onto a clean scrub brush and scrub each tool thoroughly for at least 30 seconds, or longer if there are visible pieces of product on the tool.
    - Step 2: Rinse the tool at least three times using potable water to remove the cleaning solution.
    - Step 3: Place tools on aluminum foil covered with KimWipes®, or other pretested absorbent towels, to air dry until cleaning process is complete. Pat tool dry with new KimWipe® and place on new foil dull side up without layer of KimWipes®.
        - Thoroughly rinse the scrub brush prior to cleaning the next tool.
    - Step 4 performed in fume hood: Transfer appropriate volume of methanol (Certified ACS Reagent Grade ≥ 99.8%) from original storage container to clean methanol rinse bottle. Use the methanol rinse bottle to rinse surface of processing tool with methanol. Collect methanol rinse into a HDPE sample bottle setting in a stainless steel collection bowl. Place tool on clean aluminum foil (dull side up) inside the fume hood.
        - Allow tools to air-dry in the fume hood.
        - Collect used rinse solvent as a processing rinse sample.
        - Methanol and methanol rinse bottle are located in the flammable cabinets in the Hazardous Material Storage room.
    - Step 5: For individual wrapping of each tool, lay out aluminum foil dull side up (dull side in contact with tool) large enough to encapsulate the item.
        - Do not use KimWipes® on tool after methanol rinse.

        - Wrap tools in aluminum foil after all solvent has evaporated and place in the clean tool bin.
        - Place all tools in the same direction (e.g. handles and cutting edges).
- Continue with the above process until all tools are clean. Use tool-cleaning process on any tools and equipment that will be used to process solid samples or collect aliquots of samples.

8.5 Sample ID

For product testing product component samples, individual product component IDs are auto-generated by the product testing database during product and component login (described in Ecology 2018c). Product component IDs combine information from store of purchase or location of collection, purchase or collection event, product, and component of product (e.g. "CA-1-1-2" = Cabela's, purchase event 1, product 1, 2nd component of the product processed).

Product component samples sent for analysis to the contract lab will include a MEL ID number generated from a seven-digit work order number for the study sample set followed by a dash and a two-digit number specific for each sample in the set (e.g. 1234567-01).

The product testing sample ID and MEL sample ID number will be recorded on both the sample containers and the chain of custody form.

8.6 Chain-of-custody

Chain of custody will be maintained for all samples throughout the study. Products collected for the study will be stored in locked cabinets in Ecology's product testing processing room for the duration of the study. Samples will be stored in locked cabinets in Ecology's product testing processing room until shipped to the contract lab. Ecology staff will use the contract laboratory's chain of custody form (or MEL's chain of custody form if one is not provided by contract lab) for shipment of product component samples to the laboratory.

8.7 Field log requirements

Product purchasing and collection events will be recorded in a bound notebook with pre-numbered pages. A permanent ink pen will be used to record all entries and corrections will be made with single line strikethrough, initials, and date. The Product Documentation Log includes the following information:

- Study QAPP Name
- Project Manager (PM) Name
- Collector/Sampler Name
- Collection Date

- Store or Site Name and Address
- Purpose of Product Collection (optional)
- Explanation of Marketing (if applicable)
- Arrival Time at the Product Collection Location
- Number of Products Purchased/Collected
- Location Contact Name, Phone Number and Email Address
- Miscellaneous/Comments
- Return Time to Ecology

Advertisements, photos of product marketing, and other information gathered during the purchasing and collection event for this study will be recorded and uploaded or scanned into the product testing database.

8.8 Other activities

Necessary activities are detailed in other sections of this QAPP.

9.0 Laboratory Procedures

9.1 Lab procedures table

Ecology will post a solicitation for bid seeking a laboratory to carry out the analyses described in Table 7. The contract will be managed through MEL. The contract laboratory will be expected to meet or exceed the quantitation limits outlined below and have established methods for the target analytes (Table 7) using the outlined instrumentation.

Table 9. Lab Procedures

Analyte	Sample Matrix	Estimated Sample Number	Estimated Arrival Date	Expected Range of Results	Quantitation Limit	Method
29 PFAS analytes*	paper, textiles, homogenous mixtures, oils	140	June 2018 through September 2018	< 1.0 ppb - 1,000 ppb	1.0 ppb - 20 ppb	LC-MS/MS; isotopic dilution
19 PFAS analytes* after TOP assay	paper, textiles, homogenous mixtures, oils	140	June 2018 through September 2018	< 1.0 ppb - 1,000 ppb	1.0 ppb - 20 ppb	LC-MS/MS; isotopic dilution

ppb: parts per billion
LC-MS/MS: liquid chromatography-tandem mass spectrometry
*PFAS analysis includes 29 PFAS analytes analyzed pre-oxidation followed by total oxidizable precursor (TOP) assay and 19 PFAS analytes analyzed post-oxidation.

9.2 Sample preparation method(s)

The procedure for processing solid-type samples (i.e. paper and textiles) and collecting aliquots of liquid-type samples (i.e. cream, gel, and liquid homogenous mixtures and oils) for PFAS analysis is outlined below. Solid-type products are deconstructed into individual components that comprise the product. For example, an outdoor jacket can be separated into three component samples: the outside fabric, inside fabric, and interior filling. The product components that are of interest for lab analysis are processed into samples. Multiple product component samples from one product may be submitted to the laboratory for testing. Liquid-type samples are collected as an aliquot of the original product component.

Processing blanks will be collected and consist of a piece of copy paper (no recycled content) placed in the center of the table where solid products are processed into samples. One process blank will be collected for each sample processing day. An additional processing blank will include a surface process blank which consists of a piece of clean aluminum foil (see footnote 1, Section 8.2) used to process samples on.

Product Testing Sample Processing for PFAS analysis:

- Processing Solid Samples
    - Line a clean table with aluminum foil, dull side up.
    - Place a new piece of aluminum foil, large enough to place under the product that will be processed for a sample, on the table with dull side up.
    - Weigh the empty labeled sample jar and place jar on processing surface with lid set loosely on top of the jar.
    - Use a clean tool (i.e. scissors) that is wrapped in aluminum foil (dull side towards blades) and cleaned following the tool cleaning procedure (section 8.4). Remove aluminum foil around tool and place on clean aluminum foil processing area.
    - Set the product that will be processed for a sample next to the clean aluminum foil processing area. Open the bag that contains the product.
    - Change gloves (change gloves before processing every sample) and remove the product from open bag and place the product on clean processing area (aluminum foil). Minimize contact of the selected product component sample with the surface of the aluminum foil during processing of sample.
    - Hand-reduce the sample of product to approximately 2 centimeter by 2 centimeter (cm) into the sample jar using a clean tool.
    - Weigh the jar and record the weight of final processed sample on label.
- Collecting Aliquots of Liquid Samples
    - Liquid, gel, and cream homogeneous mixtures and oil samples will be sent to the lab in original unopened container, if possible.
    - If product container is too large to ship to the lab (e.g. 5 gallon AFFF) then:
        - Wear appropriate PPE (e.g. gloves, eyewear, and lab coat) while collecting product component samples.
        - Vigorously mix product in unopened original container. Open lid of product container.
        - Open lid of clean sample jar with clean gloves and pour a well-mixed aliquot of the original product into clean jar.
        - Replace lid of jar without touching inside of jar or lid.
        - Ensure that the aliquot poured from original container does not run down side of container but is a clean pour directly from original container into clean sample jar.
        - Collect aliquot in a fume hood, if possible, or a well-ventilated area.
    - Collecting aliquots of product component samples at site locations where product cannot be vigorously mixed (e.g. 55 gallon drum of AFFF) then:
        - Wear appropriate PPE (e.g. gloves, eyewear, and protective clothing) while collecting product component samples.

- Use a single-use (new precleaned disposable with no Teflon) HDPE coliwasa [2] or glass with glass plug (composite liquid sampler) to collect a minimum of 100 ml of a representative sample from large container or drum.
- Open lid of clean sample jar with clean gloves and discharge collected sample from the coliwasa sampler into clean jar.
- Replace lid of jar without touching inside of jar or lid.
- Ensure that the outside of coliwasa sampler containing sample does not touch any surface before releasing sample collected from original container into clean sample jar.
- Replace used coliwasa sampler in original packaging (if individually packaged) or wipe outside of sampler and collect used wipes in waste bag and dispose of coliwasa and waste in appropriately designated waste container.

Product component samples sent to the contract laboratory will undergo solvent extraction followed by analysis of 29 PFAS analytes using liquid chromatography with tandem mass spectrometry (LC-MS/MS). The TOP assay converts oxidizable PFAS into terminal PFCA through the use of persulfate oxidation on an aliquot of the product component sample extract and samples are subsequently analyzed for the suite of 19 PFSA and PFCA. The results of the product component samples for the suite of PFSA and PFCA analyzed pre-oxidation (before persulfate oxidation) and post-oxidation (after persulfate oxidation) will be compared. The increase in concentration of the terminal PFCA following oxidation represents the precursor potential of the product component sample (Houtz and Sedlak 2012).

9.3 Special method requirements

The PFAS method required for this project are newly developed for product matrices. The project manager will need to work closely with the contract laboratory and the MEL QA Coordinator to ensure that the method used meet the needs of this study.

9.4 Laboratories accredited for methods

A laboratory accreditation waiver will be obtained for this study as the PFAS analytes and total oxidizable precursor assay are non-standard and no accreditation exists at this time for environmental samples and consumer products.

[2] Review Ecology's Hazardous Waste and Toxics Reduction Program QAPP for use of coliwasa, internal document.

10.0 Quality Control Procedures

10.1 Table of field and laboratory quality control

Collection and processing quality control (QC) samples will follow procedures outlined in section 8.2 (field and trip blanks), section 8.4 (methanol processing rinsates), and section 9.2 (sample processing blanks) and included in the batch of product component samples sent to the laboratory for analysis. Table 8 displays the laboratory QC procedures required for this study.

Table 10. Laboratory Quality Control Procedures

Parameter	Matrix	Collection and Process Blanks^	Laboratory				
			LCS	LCS Duplicates	Sample Duplicates	Method Blanks	Surrogate
PFAS analytes	paper, textiles, homogeneous mixtures, oils	as collected	1/batch	1/batch	1/batch	1/batch	every sample
PFAS analytes	methanol	2	---	---	---	---	---

batch: 20 samples or fewer
LCS: laboratory control sample
^Includes field, trip, and processing blanks and processing rinsate blanks.

10.2 Corrective action processes

Deviations from the project plan when conducting product purchase and collection, processing, and sample preparation will be discussed in the final report. Substantial deviations will need to be described in an addendum pre-approved by the QA Officer.

The project manager will work closely with the contract laboratory and the MEL QA Coordinator conducting the data review to examine data that fall outside of QC criteria. The project manager will determine whether samples should be re-sampled, re-analyzed, rejected, or used with appropriate qualification.

11.0 Data Management Procedures

11.1 Data recording and reporting requirements

Documentation of purchased and collected products will be recorded in the Product Documentation Log. Study data will be stored in Ecology's internal PTDB (Ecology 2018c). Data collected in the PTDB includes purchase receipts, products purchased (in store and online) and collected, product descriptions, product photos, description of product components, methods used to process component samples, laboratory results, and case narratives.

Laboratory data will be sent to the project manager as an electronic data deliverable (EDD) package from MEL after data validation is performed. The project manager will conduct a QA review of the data and assess results for usability (see Sections 13 and 14). The project manager will upload the final data to the internal PTDB.

11.2 Laboratory data package requirements

The contract laboratory will deliver a Tier 4 Level data package to MEL after completing laboratory analysis. The contract laboratory will submit laboratory data as a fully paginated and bookmarked comprehensive PDF format file with all contract specific content, along with data in EDD format as .CSV files. MEL will review the data package and provide case narratives to the project manager with the final qualified results and a description of the quality of the contract laboratory data. Case narratives will include any problems encountered with the analyses, corrective action taken, minor modifications to the requested analytical method, and definition of data qualifiers.

11.3 Electronic transfer requirements

MEL will deliver case narratives in PDF format and electronic data deliverables in Excel spreadsheet format (as .CSV files) to the project manager by email.

11.4 EIM/STORET data upload procedures

Not applicable to this study. Section 11.1 describes the database where data will be stored for this study.

11.5 Model information management

Not applicable to this study.

12.0 Audits and Reports

12.1 Field, laboratory, and other audits

Laboratories must participate in performance and system audits of their routine procedures. No audits are planned for this study.

12.2 Responsible personnel

No audits are planned for this study.

12.3 Frequency and distribution of reports

A final published report summarizing the data and findings will include at a minimum:

- An overview of the study.
- Goals and objectives of the study.
- General description of products purchased.
- Discussion of data quality and the significance of any problems encountered.
- Summary tables and graphs of laboratory data.
- Discussion of laboratory results.
- Conclusions and Recommendations.

12.4 Responsibility for reports

The project manager/principal investigator will be the lead responsible for the final report.

13.0 Data Verification

13.1 Field data verification, requirements, and responsibilities

The project manager will conduct a final review of product purchases and collections, product components, component samples shipped to the laboratory, and additional product data entered into the PTDB by the project sampling assistant or additional product testing staff.

13.2 Laboratory data verification

Data verification involves examining the data for errors, omissions, and compliance with QC acceptance criteria. MEL SOPs for data reduction, review, and reporting will meet the needs of the project. Contract laboratory data packages will be assessed by MEL's QA Coordinator following MEL SOPs and the EPA Functional Guidelines for Organic Data Review (EPA 2017).

MEL's QA Coordinator will provide a written report of their data review which will include a discussion of whether (1) MQOs were met, (2) proper analytical methods and protocols were followed, (3) calibrations and controls were within limits, and (4) data were consistent, correct, and complete, without errors or omissions.

The project manager/principal investigator is responsible for the final acceptance of the project data. The complete data package, along with MEL's written report, will be assessed for completeness and reasonableness. Based on these assessments, the data will either be accepted, accepted with qualifications, or rejected and re-analysis considered.

13.3 Validation requirements, if necessary

Independent data validation will not be required for this project.

13.4 Model quality assessment

This study does not involve modeling or analysis of existing data.

14.0 Data Quality (Usability) Assessment

14.1 Process for determining project objectives were met

Upon completion of the data verification process, Data Quality (Usability) Assessment will be conducted (Ecology 2004). Data from all field and laboratory procedures will be examined to determine whether they were measured with the proper procedures, fall into the expected range of results, and meet reporting limits as described in Sections 8 and 9, above. They will also be examined to determine whether all MQOs and QC procedures described in Sections 6 and 10, respectively, have been met.

If all specifications are met, the quality of the data should be usable for meeting project objectives. If the MQOs have not all been met, the project manager/principal investigator will examine the data to determine whether they are still usable and whether the data quantity and quality are sufficient to meet project objectives. Data that do not meet the criteria detailed in this QAPP will be qualified appropriately. The project manager will be responsible for analyzing the data and determining how the results will be summarized and documented in each report.

14.2 Treatment of non-detects

Laboratory data will be reported down to the method detection limit, with an associated "U" or "UJ" qualifier for non-detects.

14.3 Data analysis and presentation methods

A summary of the data will be presented in the final report. Summary statistics will be displayed in tables and results in graphs or charts.

14.4 Sampling design evaluation

Analysis of 140 children's, general consumer, and state-purchased product component samples is sufficient for an investigation of the presence of PFOS, PFOA, and other PFAS in Washington State products. The results of this investigation may lead to additional testing of a wider variety of products and/or larger sample size.

14.5 Documentation of assessment

Documentation of assessment will occur in the final report (see Section 12).

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16.0 Appendices

Appendix A. Glossaries, Acronyms, and Abbreviations

Glossary of General Terms

Ambient: Background or away from point sources of contamination. Surrounding environmental condition.

Anthropogenic: Human-caused.

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.

Acronyms and Abbreviations

Term	Description
e.g.	For example
Ecology	Washington State Department of Ecology
EPA	U.S. Environmental Protection Agency
et al.	And others
i.e.	In other words
MEL	Manchester Environmental Laboratory
MQO	Measurement quality objective
PBT	persistent, bioaccumulative, and toxic substance
QA	Quality assurance
QC	Quality control
RPD	Relative percent difference
SOP	Standard operating procedures
WAC	Washington Administrative Code

Units of Measurement

Unit	Measurement
g	gram, a unit of mass
kg	kilograms, a unit of mass equal to 1,000 grams
m	meter
mm	millimeter
mg	milligram
mg/Kg	milligrams per kilogram (parts per million)
mg/L	milligrams per liter (parts per million)

Unit	Measurement
mL	milliliter
ng/g	nanograms per gram (parts per billion)
ng/Kg	nanograms per kilogram (parts per trillion)
ng/L	nanograms per liter (parts per trillion)
pg/g	picograms per gram (parts per trillion)
pg/L	picograms per liter (parts per quadrillion)
s.u.	standard units
ug/g	micrograms per gram (parts per million)
ug/Kg	micrograms per kilogram (parts per billion)
ug/L	micrograms per liter (parts per billion)
ww	wet weight

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 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)

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