PFAS in Washington State Products, 2018

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Abstract

In 2018, the Washington State Department of Ecology (Ecology) conducted a study to assess the levels of per- and polyfluoroalkyl substances (PFAS) in consumer products. A total of 142 samples, consisting of 130 product component samples and 12 collection and processing quality control samples, were tested for 29 PFAS analytes and 19 total oxidizable precursors.

Products in the following categories were tested:

- Automotive, Building, and Cleaning Products
- Children's Clothing and Textiles
- Cosmetics and Personal Care Products
- Food Contact Material

This short summary provides details of the testing methods, including product collection and data quality, for the PFAS 2018 product testing study. This information was developed to accompany the PFAS 2018 product testing data available in Ecology's product testing database by searching *PFAS in Washington State Products – 2018*.

Publication Information

This report is available on the Department of Ecology's website at:
https://apps.ecology.wa.gov/publications/SummaryPages/2303007.html.

Data for this project are available in Ecology's Product Testing Database at:
https://apps.ecology.wa.gov/ptdbreporting/

Study: *PFAS in Washington State Products – 2018*

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Background

Per- and polyfluoroalkyl substances (PFAS) are a group of synthetic 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. PFAS are highly persistent in the environment and some bioaccumulate in humans. Epidemiological studies link PFAS to health effects in humans (Ecology and Health, 2021).

The Quality Assurance Project Plan (QAPP) for this study provides a detailed discussion on the background including a description of the project goals and specific PFAS analytes and total oxidizable precursors tested (Trumbull, 2018).

Methods

The project plan for this study is *Quality Assurance Project Plan: Per- and Polyfluoroalkyl Substances (PFAS) in Washington State Products* (Trumbull, 2018).

Product Collection

Ecology's product testing team purchased products from June 20 through August 27, 2018. Products in this study were primarily purchased in a store. In-store retail purchasing occurred on four days and included 27 retail stores located in Thurston and King counties. Products were also purchased online in five online retail store purchases. Product information and photos were recorded in Ecology's product testing database (PTDB).

Product component samples were labeled with a unique Ecology identification number (ECY ID). For example, the ECY ID product component sample, TG-38-2-1, corresponds to: TG for Target, the 38 indicates the 38th time Ecology purchased products from Target, the 2 refers to a unique product from TG in that purchase, and the 1 indicates the component from that product.

Products were purchased, processed into samples, and tested for PFAS analytes and total oxidizable precursors in the following product categories:

- Automotive, Building, and Cleaning Products (40 samples)
  - Automotive Care (7 samples)
  - Building Maintenance (10 samples)
  - Cleaning Products (23 samples)
- Children's Clothing and Textiles (15 samples)
  - Activity Gyms (4 samples)
  - Floor rugs (4 samples from 2 products)
  - Pillows (2 samples)
  - Clothing (5 samples)
- Cosmetics and Personal Care Products (40 samples)
  - Sun Protection (10 samples)
  - Skin Moisturizer (5 samples)
  - Complexion Cosmetics (8 samples)
  - Lip Cosmetics (7 samples)
  - Antiperspirants and Deodorants (10 samples)

- Food Contact Material (35 samples)
  - Disposable Containers (8 samples)
  - Disposable Plates (20 samples)
  - Disposable Wraps (7 samples)

Sample Processing

A total of 69 liquid, gel, cream, powder, and wax product component samples from smaller-sized product containers were sent to the lab in the original and unopened container. Eleven liquid product component samples from large-sized containers were subsampled with a decontaminated stainless-steel ladle or by decanting into a 250 milliliter (mL) wide-mouth high density polyethylene (HDPE) sample container.

A total of 50 paper and textile product component samples were hand-reduced in size to approximately 2 millimeters (mm) by 2 mm size pieces with decontaminated stainless-steel scissors into a 250 milliliter (mL) wide-mouth HDPE sample container.

A total of 12 quality control (QC) samples were collected during product collection and sample processing and outlined below.

- One tool cleaning methanol rinse sample was collected as a blank QC sample during decontamination of the stainless-steel scissors and ladles.
- Two sample processing water blank samples were collected as blank QC samples, one during processing of the paper samples and one during processing of the textile samples.
  - The contract laboratory provided Canadian Springs water in 250 mL wide-mouth HDPE sample containers. The open containers were placed in the center of the table where paper and textile products were processed into samples.
- One processing QC sample consisting of the brand of nitrile gloves (unused) that were used by the product testing team during product handling and sample processing of the product components into samples.
  - The unused nitrile glove was hand-reduced in size following the same procedures for processing products into samples.
  - The brand of nitrile gloves is Kimberly-Clark purple nitrile powder-free exam gloves.
- Two processing QC samples consisting of two brands of foil (unused) that were used as surface cover on the table to process paper and textile products into samples.
  - The two pieces of foil were hand-reduced in size following the same procedures for processing products into samples.
  - The two brands of foil are Handi-Foil and Reynolds Foodservice Foil.
- Three product collection field blank QC samples consisting of copy paper (no recycled content) were placed into a new quart-sized resealable plastic bag and the bag was not opened during product collection.
  - Each field blank QC sample corresponds to a single product collection day comprising of multiple store locations.
  - The paper QC sample was hand-reduced in size following the same procedures for processing products into samples.

- Three product collection trip blank QC samples consisting of copy paper (no recycled content) were placed into a new quart-sized resealable plastic bag and the bag was opened during product collection.
    - Each trip blank QC sample corresponds to a single product collection day comprising of multiple store locations.
    - The paper QC sample was hand-reduced in size following the same procedures for processing products into samples.

Laboratory Analysis

SGS Axys Analytical Services, Ltd. (SGS Axys) in Sidney, British Columbia, Canada performed the PFAS analyte and total oxidizable precursors analyses for 142 samples consisting of 130 product component samples and 12 collection and processing QC samples.

Sample preparation, extraction, instrumental analysis, and analyte quantification procedures were in accordance with SGS AXYS Methods:

- MLA-110: *Analytical Procedure for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples, Solids and Solvent Extracts by LC-MS/MS.*
- MLA-111: *Analytical Procedure for the Analysis of Total Oxidizable Precursors (TOP) in Aqueous and Solid Matrices by LC-MS/MS.*

For the analysis of 29 PFAS analytes by SGS AXYS Method MLA-110, approximately 0.01 to 0.5 grams of each sample was accurately weighed, spiked with isotopically labeled quantification standards and extracted and cleaned up by solid phase extraction (SPE) using a disposable cartridge. After spiking with labeled recovery (internal) standards, the extract was analyzed by high performance liquid chromatography/tandem mass spectrometry (LC-MS/MS). Analyte concentrations were determined by isotope dilution/internal standard method, comparing the area of the quantification ion to that of the isotopically labeled standard.

For the analysis of 19 total oxidizable precursors by SGS AXYS Method MLA-111, approximately 0.01 to 0.5 grams of each sample was accurately weighed, spiked with isotope labeled extraction standards and an isotope labeled oxidation monitoring standard, and oxidized using base and heat activated persulfate. After cooling and pH adjustment, the reaction mixture was spiked with isotope labeled quantification standards, extracted, and cleaned up using weak anion exchange SPE cartridge. The cartridge was eluted using basic methanol, and the resulting extract was then spiked with labeled recovery (internal) standards and analyzed using a HPLC reversed phase C18 column using a solvent gradient. The column was coupled to a triple quadrupole mass spectrometer run at unit mass resolution in the Multiple Reaction Monitoring (MRM) mode with negative electrospray ionization. Target analyte concentrations were determined by isotope dilution/internal standard method.

Data Quality

Stage 4 validation was performed by Ecology's Manchester Environmental Laboratory Acting Quality Assurance Coordinator to verify that the data were generated following the analytical method with no omissions or errors. Data validation reports were provided to the project manager. All data were also reviewed by the project manager. The project manager made revisions as needed and documented in a data review narrative. Data were deemed usable as qualified.

- No qualifier: the analyte was positively identified in the sample.
- "J" qualifier: the analyte was positively identified, and the reported result is an approximate concentration of the analyte in the sample.
- "U" qualifier: analyte was not detected at or above the reported quantitation limits.
- "UJ" qualifier: analyte was not detected at or above the reported quantitation limits, and the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to measure the analyte accurately and precisely in the sample.
- "REJ" qualifier: the sample result was rejected due to serious deficiencies in the ability to analyze the sample, meet quality control criteria or other technical reason. The presence or absence of the analyte cannot be verified.

PFAS analyte concentrations at levels less than five times the concentrations found in the associated lab method blank, if present, were qualified as non-detects, "U," for this study. Data were evaluated on the technical acceptance criteria outlined in the contract laboratory's methods. Measurement quality objectives (MQOs) were met with the following exceptions and documented under "Additional Comment Description" column in Ecology's PTDB for each sample result qualified.

- A significant dilution was conducted for a sample and re-analyzed. The analyte concentrations were not recovery corrected and should be considered an estimated possible minimum value. The results were qualified as "J."
- For sample and sample duplicate RPD exceeding method QC limits, the associated target compounds were detected in the sample and qualified as "J."
- For LCS recoveries, LCS duplicate recoveries or both above method QC limits, the associated target compound was not detected in the sample and not further qualified. The qualifier remained as "U."
- For LCS recoveries, LCS duplicate recoveries or both below method QC limits, the associated target compounds were not detected in the sample and qualified as "UJ."
- For LCS and LCS duplicate RPD exceeding method QC limits, the associated target compound was not detected in the sample and qualified as "UJ."
- For surrogate recoveries above method QC limits, when
  - the associated target compound was not detected in the sample, the result was not further qualified. The qualifier remained as "U."
  - the associated target compound was detected in the sample, the result was qualified as "J."
- For surrogate recoveries below method QC limits, when
  - the associated target compound was not detected in the sample, the result was qualified as "UJ."
  - the associated target compound was detected in the sample, the result was qualified as "J."
- For surrogate recovery below method QC limit and below 10% recovery, when
  - the associated target compound was not detected in the sample, the result was qualified as "REJ."
  - the associated target compound was detected in the sample, the result was qualified as "J."

- For internal standard recoveries below method QC limits, the associated target compounds were not detected in the sample and qualified as "UJ."
- For reaction monitoring surrogate recoveries exceeding method QC limits, the associated target compounds were not detected in the sample and qualified as "UJ."
- For extraction standard recoveries below method QC limit, when
  - the associated target compound was not detected in the sample, the result was qualified as "UJ."
  - the associated target compound was detected in the sample, the result was qualified as "J."
- For extraction standard recoveries below method QC limit and below 10% recovery, when
  - the associated target compound was not detected in the sample, the result was qualified as "REJ."
  - the associated target compound was detected in the sample, the result was qualified as "J."

Results

PFAS and total oxidizable precursors data for product component samples and collection and processing QC samples are available for download in Ecology's PTDB[1] by searching: *PFAS in Washington State Products - 2018.* All PFAS analyte concentrations are reported on an as received (wet weight) basis in concentration units of nanogram per gram (ng/g).

References

Ecology and Health, 2021. Per- and Polyfluoroalkyl Substances Chemical Action Plan. Publication 21-04-048, 636 pages. Washington State Department of Ecology and Washington State Department of Health, Olympia. https://apps.ecology.wa.gov/publications/summarypages/2104048.html

Trumbull, K. Quality Assurance Project Plan: Per- and Polyfluoroalkyl Substances (PFAS) in Washington State Products. Publication 18-04-012. Washington State Department of Ecology, Olympia. https://apps.ecology.wa.gov/publications/SummaryPages/1804012.html.

[1] https://apps.ecology.wa.gov/ptdbreporting/