March 9, 2023

Director Megan Patterson
Maine Board of Pesticides Control
28 State House Station
Augusta, ME 04333-0028

Dear Director Patterson,

We would appreciate it if the attached article from the Journal Environmental Science and Technology, "Directly Fluorinated Containers as a Source of Perfluoroalkyl Carboxylic Acids," by Heather D. Whitehead and Graham F. Peaslee of the University of Notre Dame, could be shared with the Board of Pesticides Control in advance of the meeting on March 15, 2023.

This brand-new article is directly relevant to the Board's deliberations about implementing the Legislature's directive to regulate PFAS contamination of pesticides from contact with fluorinated containers. The article confirms previous studies including by the Environmental Protection Agency finding that toxicologically significant levels of various PFAS can and do migrate from fluorinated HDPE containers into liquids stored within, including pesticides. The Whitehead/Peaslee study additionally reviewed the impact of conditions that mimic actual storage conditions for pesticides in a farm setting in a shed or other farm building, where the temperature is not controlled.

The Notre Dame scientists found a large number of PFAS compounds leaching into samples in concentrations from 45.12 to 94.81 ng/g plastic with an average and standard deviation of 63.75 ± 13.12 ng/g plastic. Analytes detected in fluorinated containers included PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoDA, PFTrDA, and PFTDA, all detected in 100% of samples. PFHxDA and PFODA were detected in 14% and 29% of samples. When samples in fluorinated containers were exposed to high temperatures not inconsistent with summer heat wave conditions inside a farm shed, "sums of PFAS concentrations were 27 times higher in samples exposed at 50 °C than those exposed at room temperature, demonstrating that exposure to elevated temperatures significantly increased the migration of PFAS from the containers into the water."

While another takeaway from the article is that PFAS are also likely migrating from plastic containers into common foods such as ketchup and mayonnaise, this is no reason not to limit as much as possible human worker and consumer exposure from PFAS as an ingredient

and/or contaminant in pesticides. Indeed, the fact that people are being exposed to PFAS through multiple pathways - including from their clothing, water and food - is all the more reason to shut down additional exposure routes, including contamination from fluorinated containers. There are alternatives to these containers and the Board has the clear authority and responsibility to regulate under these circumstances.

Thank you for your consideration.

Sincerely,

Heather Spalding
Deputy Director, MOFGA

Letter

Directly Fluorinated Containers as a Source of Perfluoroalkyl Carboxylic Acids

Heather D. Whitehead* and Graham F. Peaslee

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ABSTRACT: Direct fluorination of plastics is performed to impart chemical resistance via exposure of polyethylene to fluorine gas to produce a fluorine-modified surface layer. Leaching experiments were performed on a directly fluorinated container under various conditions and with different matrices, including foodstuffs. The average sum of per- and polyfluoroalkyl substances (PFAS) concentrations measured from extraction of a fluorinated container was 63.75 ± 13.2 ng/g plastic. Seven-day leaching experiments of fluorinated containers with water, methanol, and acetone produced sums of PFAS concentrations that ranged from 0.99 to 66.92 ng/g plastic. Leaching experiments with food matrices produced sums of PFAS concentrations ranging from 2.66 to 7.19 ng/g plastic. A subset of samples subjected to leaching at elevated temperatures generated sums of PFAS concentrations up to 830% higher. In all experiments, short-chain perfluoroalkyl carboxylic acids (PFCAs) were detected in the highest frequencies and concentrations with analyte concentration decreasing as chain length increased. An estimate for PFAS released into food ranged from 0.77 to 2.68 ng/kg body weight per week, showing ingestion of food stored in these containers could be a significant source of exposure. Based on the large number of applications where directly fluorinated containers find use, the observation of PFAS migration suggests use regulations are warranted, and future studies should explore their fate when disposed or recycled.

KEYWORDS: PFAS, fluorinated containers, LC-MS/MS, plastics

■ INTRODUCTION

Per- and polyfluoroalkyl substances (PFAS) are a class of more than 12,000 anthropogenic chemicals with physical properties that are desirable for use in a variety of industrial processes and consumer products. Significant uses of these compounds range from use in firefighting foams and nonstick cookware to those in cosmetics, textiles, and more.[1−4] Research in both animal models and human exposure studies has increasingly shown that exposure to these compounds is hazardous to health, leading to efforts to limit and monitor their use.[5−7] Recently, the United States Environmental Protection Agency (U.S. EPA) lowered the interim health advisory limits of perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS), two of the most historically used PFAS, to 0.004 and 0.02 parts-per-trillion in drinking water, 17,500 times and 3,500 times lower than previous values set in 2016.[8,9] Industrial processes account for the largest use of PFAS, and a 2020 report from Europe highlighted the largest single-use category was in the production of plastics and rubbers.[2]

Uses of PFAS in plastics and rubbers include direct application of polymeric and nonpolymeric PFAS on other plastics or the manufacturing of fluorinated plastics and rubbers. Specific uses include the application of fluorinated polymer processing aids as extrusion agents, nonpolymeric PFAS as mold release agents for plastics and resins, and the production of fluoropolymer plastics and rubbers.[2] Higher costs to produce fluoropolymer plastics encouraged the development of alternative approaches for imparting chemical resistance by direct fluorination of less-expensive hydrocarbon-based plastics.[10,11] Direct fluorination involves the exposure of plastic to a fluorine-inert gas mixture to encourage the replacement of hydrogens with fluorine on the plastic surface, forming a $0.01-10$ μm fluorine-modified layer.[12] This process allows for cheaper and more efficient production of plastics that contain desirable physical properties, primarily increased barrier properties.[13]

The economic advantages of direct fluorination, however, do not come without disadvantages, as detailed by Rand and Mabury (2011).[14] They note that both the intentional and

unintentional introduction of oxygen during direct fluorination leads to the production of perfluoroalkyl carboxylic acids (PFCAs) in these containers. This work found total PFAS up to 113 ng/cm^2, with concentrations dominated by short-chain (C3−C6) carboxylic acids, although measurable concentrations of C7−C10 PFCAs were also reported. Their results also demonstrated the capacity for PFAS to leach from the containers when water was stored in the containers for a year. More recently, the U.S. EPA reported that parts-per-billion concentrations of total PFAS could be leached using water and methanol from samples of directly fluorinated containers in as little as 1 week of exposure.[15−17] Of concern are the known health risks and environmental concerns associated with exposure to and release of PFCAs, especially for longer-chain PFCAs such as PFOA.[18] There are more limited toxicology data concerning long-term effects of exposure to short-chain PFCAs, though early data suggest they show similar toxicological profiles to their long-chain equivalents.[19] Compounding this concern is the growing number of applications where directly fluorinated containers are used, including food packaging, pesticides, indoor household cleaners, containers for personal care products, and containers for Toxic Substance Control Act (TSCA)-regulated cleaners, solvents, and fuels as noted in various literature and industrial reports.[12,20−24] A 2022 notice from the U.S. Food and Drug Administration (U.S. FDA) noted the potential risk that fluorinated containers pose when used for food and requested additional information to address the potential for PFAS to migrate from fluorinated containers to food.[25]

To help address these concerns and to build upon earlier studies, this study was designed to measure the mobility of PFCAs from containers into the products they can contain. Segments of a directly fluorinated high density polyethylene (HDPE) container were exposed to solvents and foodstuffs to quantify the amount of PFCAs that migrate from the containers to the products. The effect of heat on the mobility of PFCAs was also measured to determine how environmental conditions might affect the total PFAS that can be released by directly fluorinated containers.

■ MATERIALS AND METHODS

LC-MS/MS grade methanol (A456-4) and acetonitrile (A955-4) were purchased from Fisher Scientific (Waltham, MA). Ultrapure water was used for sample preparation and instrumental analysis. Ammonium acetate (5438340100) and acetone (34850-1L) were purchased from Sigma-Aldrich (St. Louis, MO). Twenty-one native standards (PFAC-MXC) and 13 isotopically labeled standards (MPFAC-C-ES) were purchased from Wellington Laboratories (Guelph, ON, CA). QuEChERS extraction salts (5982-6555) and dispersive SPE (5982-5258) materials were purchased from Agilent Technologies (Santa Clara, CA) as were polypropylene HPLC vials (5191-8150) and caps (5191-8151). Poly(ether sulfone) syringe filters (76479-022) and 3 mL polypropylene syringes (53548-017) were purchased from VWR (Radnor, PA).

Twelve containers of fluorinated HDPE (EW-62500-10) and nonfluorinated HDPE (EW-62150-20) bottle containers were purchased from Cole-Parmer (Vernon Hills, IL). Fluorinated and nonfluorinated HDPE containers were stored in their original, separate shipping packaging to ensure minimal contact between containers. Food samples were purchased in-store from a national grocery chain. Each of the food samples purchased were labeled as "organic" and were purchased in

glass containers to minimize the potential presence of PFAS in the food due to their original packaging. Food samples were transported unopened into the laboratory where they were labeled and stored at 4 °C before and after opening.

Sample Preparation and Extraction. Detailed descriptions of the preparation of samples are given in the Supporting Information. Briefly, 86 samples were prepared for extraction and instrumental analysis, including extracts of the fluorinated and nonfluorinated containers alone ($n = 30$ samples), extracts from the food samples with and without exposure to containers ($n = 16$), samples of various solvents with and without exposure to containers ($n = 21$), and samples of select solvent and food matrices with exposure to containers at elevated temperatures ($n = 19$). In all tests where containers were used, small, 1 cm × 1 cm, segments were cut from one of the 12 containers of that type (fluorinated or nonfluorinated HDPE). In all tests where a container was measured alone, the sample was prepared for analysis without additional preparation using the modified-QuEChERS protocol described below. In all tests where food or solvent was exposed to a container, the matrix was exposed for a total of 7 days by placing the plastic segment into approximately 2 mL of solvent or 1 g of food material. In food and solvent samples, a subset was exposed at 50 °C for 7 days to compare the effects of temperature on migration. A seven-day exposure period was selected based on previous studies from the U.S. EPA that demonstrated significant migration of PFAS to water and methanol within a one-week time period.[15,16] Over the exposure period, samples were left out of direct sunlight in their respective storage conditions without mixing and were checked daily to ensure the container segments remained in contact with each matrix.

Extraction of the containers and of food matrices was performed using a modified-QuEChERS extraction method. First developed for application to pesticides in agricultural samples, QuEChERS was recently employed for the measurement of PFAS in food by the U.S. FDA.[26] The exact volumes and masses used during extraction varied depending on the sample matrix with specific details given in the Supporting Information. All extractions began with the addition of 10 ng of isotopically labeled standards to either 0.15 g of container segments or 1 g of food material in 15 mL centrifuge tubes. From there, equal parts of water and acetonitrile were added, followed by QuEChERS salts. Tubes were briefly vortexed before sonication and centrifugation. After centrifugation, a phase partition between the bottom salt−water mixture and the top acetonitrile layer allowed the transfer of the acetonitrile layer to a fresh centrifuge tube. To this centrifuge tube a small amount of dispersive SPE material was added. The tube briefly vortexed, and the sonication and centrifugation process repeated. The resulting supernatant was transferred to a new tube and was concentrated to dryness under a stream of N$_2$ before reconstitution with 1 mL of an 80:20 methanol−water mixture. Samples were filtered using a 0.45 μM PES syringe filter, and 250 μL was transferred to a polypropylene HPLC vial for analysis.

LC-MS/MS Analysis and QA/QC. A targeted analysis method was developed and validated for a total of 20 analytes. Detailed information on the LC-MS/MS analysis is given in the Supporting Information. Analysis was performed on an Agilent 1290 Infinity II UHPLC system coupled to an Agilent 6470B triple quadrupole mass spectrometer. A complete list of all analytes measured are given in Table S1. Detailed information on the chromatographic conditions and ion source

parameters is given in Table S2 with a complete MRM transition table given in Table S3. Chromatography and ion source parameters were adapted from an existing Agilent Technologies method for PFAS.[27] Limits of detection and quantification for each analyte are given in Table S4.

Detailed information on QA/QC is given in the Supporting Information. Continuing calibration checks had precision and accuracy values that fell within 77%−135% of the expected values, and interday relative standard deviations ranged from 1.7%−14.6% (median 6.1%). Average recoveries of isotopically labeled internal standards in the container extracts and solvent exposure samples ranged from 77%−122% (median 93%) for all analytes. Average recoveries of isotopically labeled internal standards in the food exposure samples ranged from 39%−92% (median 67%) for all analytes. To ensure accurate quantification in food matrices, one sample of each food type was spiked with 10 ng/mL of each native analyte and the recovered concentration measured. The average native recovery ranged from 68%−129% (median 93%) for all analytes except PFHxDA and PFODA which had average recoveries of 55% and 48%, respectively. As the average recoveries of isotopically labeled standards were reasonable across all sample types, individual analyte concentrations were not corrected using recovery data.

■ RESULTS AND DISCUSSION

Extraction of Fluorinated and Nonfluorinated Containers. A total of 30 samples were prepared to measure extractable PFAS from both fluorinated and nonfluorinated containers. Sums of PFAS concentrations measured in nonfluorinated containers ranged from 0.01 to 0.88 ng/g plastic with an average and standard deviation of 0.29 ± 0.30 ng/g plastic with only PFOA being measured above its limit of quantification in these samples. While these concentrations of PFAS in these nonfluorinated HDPE containers are notable, the occurrences of PFAS in plastics that have not undergone direct fluorination are outside of scope of this work, though they warrant future study. In comparison, the sums of PFAS concentrations measured in the fluorinated containers ranged from 45.12 to 94.81 ng/g plastic with an average and standard deviation of 63.75 ± 13.12 ng/g plastic. Analytes detected in fluorinated containers included PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoDA, PFTrDA, and PFTDA, all detected in 100% of samples. PFHxDA and PFODA were detected in 14% and 29% of samples. The comparison of the average analyte concentration measured in each container type is given in Figure S1. These data illustrate that short-chain PFCAs, namely, PFBA and PFPeA, are measured in the highest concentrations at 16.12 ± 4.88 ng/g of plastic and 18.85 ± 4.23 ng/g of plastic in fluorinated containers, respectively. Analyte concentration was observed to decrease as chain length increases in these fluorinated containers, with PFTrDA and PFTDA at 0.29 ± 0.07 and 0.22 ± 0.05 ng/g of plastic, respectively. This relationship is similar to what was observed in Rand and Mabury (2011) where concentrations of PFCAs from Soxhlet extraction ranged from 8.5−113 ng/cm^2, depending on the level of fluorination of the container.[14] The concentrations of PFCAs measured here ranged from 5.6−12.2 ng/cm^2, suggesting these containers are similar to levels reported for a lower-level fluorination described in Rand and Mabury (2011).

Leaching of PFAS from Fluorinated Containers Exposed to Solvents. A total of 21 samples were prepared

for PFAS migration from both the fluorinated and non-fluorinated containers when exposed to different solvents that might be found in containers used for TSCA-regulated products. Table S5 summarizes the sum of PFAS concentrations measured, which were consistently low for non-fluorinated containers in each exposure solvent. For fluorinated containers, water exposure samples had an average sum of PFAS concentrations of 0.99 ± 0.46 ng/g plastic. Methanol exposure samples had a significantly higher average at 69.72 ± 7.75 ng/g plastic, and acetone fell in between with an average of 50.13 ± 4.41 ng/g plastic.

A breakdown of the individual analyte concentrations measured in these samples is given in Figure S2, which demonstrates a similar relationship between analyte concentration and chain length. The concentrations of PFAS measured in these exposure solvents after 1 week of leaching with fluorinated containers can be compared to those of a 2022 report from the U.S. EPA where sums of PFAS concentrations measured in fluorinated containers exposed to water and methanol for 1 week ranged from 0.13−0.39 and 0.61−6.07 ng/mL, respectively.[16] Here, average sums of PFAS concentrations measured in fluorinated containers exposed to water and methanol for 1 week were 0.16 ± 0.09 and 11.28 ± 0.27 ng/mL, respectively, demonstrating results generated here are comparable to those found by the U.S. EPA.

Leaching of PFAS from Fluorinated Containers Exposed to Food. A total of 26 samples were prepared to measure the amount of PFAS that would leach from both fluorinated and nonfluorinated HDPE when exposed to three different food matrices, including olive oil, ketchup, and mayonnaise. These matrices were chosen as they represent food types that are sold in HDPE containers and are typically stored in containers for extended periods of time. Extractions of the food matrices alone found sums of PFAS concentrations of 0.24, 2.65, and 3.31 ng/g of food in olive oil, ketchup, and mayonnaise, respectively. Individual analytes detected in each matrix varied. Individual analyte concentrations observed in each food matrix were subtracted from the concentrations measured in samples of that food matrix with exposure to containers to account for PFAS already present in the matrix. Each food matrix was then subjected to 1 week of exposure to fluorinated and nonfluorinated containers. Sums of PFAS concentrations for each food matrix and container type are given in Table S6. Sums of PFAS concentrations measured 2.66 ± 0.82, 5.95 ± 1.59, and 7.19 ± 3.39 ng/g plastic on the olive oil, ketchup, and mayonnaise samples, respectively. The large standard deviation of these samples is likely due to small sample sizes (n = 3 for each sample set). These concentrations are notably higher than those measured during water leaching experiments but are still significantly lower than leaching experiments performed in both methanol and acetone for the same exposure period. Figure S3 summarizes the distribution of individual analytes measured in each of the food matrices after exposure to fluorinated containers. Similar to previous observations, PFBA and PFPeA had the greatest concentrations measured. Analyte concentrations measured in food matrices demonstrated a similar, but less defined, relationship between analyte concentration and chain length.

Effect of Heat on PFAS Leaching in Solvents and Food. To measure the effect of heat on PFAS migration from the containers, replicate sets of water exposure and food exposure samples were prepared and placed in a 50 °C oven for the same exposure time of 7 days. A comparison of sum of
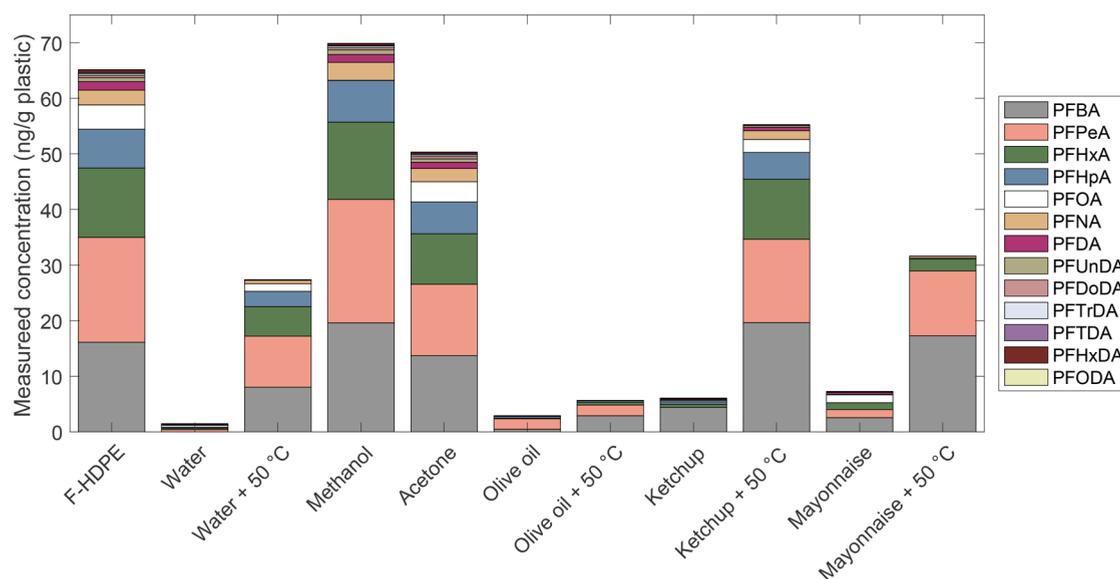
Figure 1. Combined results of extraction of containers alone (F-HDPE), solvent exposures (water, water + 50 °C, methanol, and acetone), and food exposure (olive oil, olive oil + 50 °C, ketchup, ketchup + 50 °C, mayonnaise, and mayonnaise + 50 °C) experiments performed with fluorinated containers. Concentrations of each analyte are reported in ng/g plastic and are the average of three replicates for each experiment, excluding F-HDPE which is the average of 14 replicates.

PFAS concentrations for the water samples is given in Table S7. These results highlight that concentrations of PFAS in the samples exposed to 50 °C were 0.31 ± 0.22 and 26.88 ± 4.21 ng/g plastic in the nonfluorinated and fluorinated containers, respectively. In fluorinated containers, sums of PFAS concentrations were 27 times higher in samples exposed at 50 °C than those exposed at room temperature, demonstrating that exposure to elevated temperatures significantly increased the migration of PFAS from the containers into the water. Trends within individual analyte concentrations again showed that concentration increased as chain length decreased.

Results of the food exposure samples at 50 °C showed a similar increase in PFAS concentrations relative to the samples that were stored at room temperature and are shown in Table S8. The distributions of individual analyte concentrations measured in each food matrix after exposure to fluorinated containers at elevated temperatures are shown in Figure S4. These data demonstrate significantly higher sums of PFAS concentrations in food matrices exposed to fluorinated containers at elevated temperatures at 5.63 ± 0.42, 55.25 ± 11.87, and 31.52 ± 4.62 ng/g plastic for the olive oil, ketchup, and mayonnaise samples, respectively, and a direct comparison is given in Figure S5. From this we can see concentrations increased between 110%−830% in the food matrices when exposed at elevated temperatures. We anticipate differences in PFAS migration into each food type are factors of the differences in chemical properties of each food matrix. For example, ketchup and mayonnaise both represent water-in-oil emulsions with chemical properties that might promote greater migration of PFAS from the containers into the surrounding food. The increase in concentrations between ketchup and mayonnaise is potentially a measure of differences in their ionic strength. Comparatively, olive oil likely showed lower levels of PFAS migration as the solubility of PFAS is expected to be lower in olive oil.

PFAS Exposure Assessment. Tables S8 and S9 show complete LC-MS/MS results for all samples. Figure 1 shows the combined results of each set of fluorinated container

results. Parts-per-billion PFCAs concentrations measured through both the extraction of the containers alone and measurement of migration from fluorinated containers into various matrices are of concern for both direct and indirect routes of exposure. The median sums of PFAS concentrations measured in the food matrices exposed to the fluorinated containers for 7 days (1.75 ng/cm^2) were divided by 2 to represent the concentrations present on just the inside of the container. Using a 14 fluid ounce cylindrical container with an internal surface area of 376 cm^2, the total amount of leachable PFAS from the inside of the container is about 329 ng, or 0.83 ng/g of food within the container assuming 14 oz of food. Using a serving size of 15 g, the amount of PFAS present in each serving is 12.43 ng. This estimate increases when using the median sums of PFAS concentrations measured in the food matrices exposed to the fluorinated containers at 50 °C for 7 days (6.10 ng/cm^2, or 3.05 ng/cm^2 when accounting for only the inside) to 43.33 ng per serving. Using an estimated five servings per week and the average body weight of a North American adult (80.7 kg),[28] the weekly intake of PFAS from these containers in just one food container would range between 0.77−2.68 ng/kg body weight per week. In 2020, the European Food Safety Authority set the tolerable weekly intake of PFOA, PFNA, PFOS, and PFHxS to 4.4 ng/kg body weight per week.[29] For direct comparison, the weekly intake from just these four analytes in these data would be driven by PFOA and PFNA and would range between 0.00 and 0.18 ng/kg body weight per week. The concentrations of PFAS that can be leached from these containers could then represent significant contributions to this PFAS intake and would add to PFAS exposures from drinking water, consumer goods, and other sources. Title 21 of the Code of Federal Regulations Part 177.1615 (21 CFR 177.1615) prohibits the use of direct fluorination processes where oxygen, water, or other compounds outside of nitrogen are introduced. Whether this limitation is being effectively implemented by the industry and how widely fluorinated containers are used in food packaging requires further investigation.[30] Similarly, the detection of

PFAS where water or other solvents are present in fluorinated containers raises concern about the potential for exposure from packaging applications for consumer, commercial, and industrial products subject to TSCA.

The end of the life cycle of many fluorinated containers is disposal at a landfill, where their PFCA concentrations are likely to contribute to PFAS concentrations measured in landfill leachates, which are discharged to wastewater treatment plants. The high concentrations and detection frequencies of short-chain PFCAs measured from these fluorinated containers is of concern based on research that highlights most remediation technologies employed at wastewater treatment plants are not effective at removing short-chain PFAS.[31−34] This represents a second, indirect route of exposure as wastewater effluents are ultimately discharged to aquatic environments where PFAS exposures continue. Finally, given that some fraction of HDPE is recycled, fluorinated HDPE entering the recycling stream will lead to additional routes of exposure. Considering the number of fluorinated containers in use for various applications, their contribution to overall PFAS exposure through multiple pathways may be significant, warranting regulatory action to limit this form of packaging, and future studies should concentrate on the total release of PFAS to the environment from these containers.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.estlett.3c00083.

> Additional details on sample collection and preparation, as well as parameters used for the instrumental analysis. Full results of LC-MS/MS analysis, as well tables and figures examining trends and relationships observed in samples. (PDF)

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

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