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An unwanted hitchhiker: assessment of per- and polyfluoroalkyl substances (PFAS) in vehicle cabin air conditioner and engine filters

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Per- and polyfluoroalkyl substances (PFAS) are highly mobile and widespread chemicals that are associated with an expanding list of adverse health effects. Given their ubiquity and high mobility, dust has become a suitable matrix for assessing potential indoor levels of PFAS. Currently, vehicles represent a largely underexplored source of PFAS contamination in dust. We propose that vehicle cabin air conditioning (AC) filters can be used as opportunistic sampling devices for exploring PFAS levels in dust inherently present within vehicles. This study monitored 47 PFAS in cabin AC filters (n = 10) and engine air filters (as a comparison, n = 10) via high performance liquid chromatography – tandem mass spectrometry (HPLC-MS/MS). Cabin AC filters, which filter air circulated within the passenger compartment, contained higher PFAS concentrations (median  $\sum PFAS = 92 \text{ ng g}^{-1}$ ) than the engine air filters, which filtered outdoor air feeding into the vehicle engine (median  $\Sigma$ PFAS = 2 ng g<sup>-1</sup>). In cabin AC filters, the dominant PFAS were polyfluoroalkyl phosphate esters (PAPs), which accounted for 45% of  $\Sigma$ PFAS by concentration. In engine filters, the dominant PFAS were fluorotelomer sulfonic acids (dominated by one engine filter) and perfluoroalkyl carboxylic acids, which represented 59% and 20% of the  $\sum$ PFAS, respectively. This study demonstrated that we are likely exposed to PFAS inside vehicle cabins and that cabin AC filters are a well-suited sampling matrix worth further exploration.

#### 1. Introduction

Per- and polyfluoroalkyl substances (PFAS) are a class of over 15 000 anthropogenic chemicals that are commonly referred to as "forever chemicals". PFAS are ubiquitous, highly mobile,

bioaccumulative, and have been linked to a myriad of adverse health-related effects.<sup>1,3</sup> A significant thrust of PFAS research has focused on its presence in the natural environment. As of late, there has been a shift toward assessing their presence in built environments. This is due to the fact that people spend roughly 90% of their time indoors, where PFAS are often highly present, leading to an array of daily exposures resulting from the frequent use and close proximity to PFAS-containing products.4 To date, the presence of PFAS in homes and workplaces has been relatively well-studied, yet their existence in several built environments has not been explored, such as the cabin of a vehicle. There are many components inside vehicles that are likely significant sources of PFAS, including car upholstery, carpeting, plastics, varnishes, coatings, and the products we bring into the vehicle cabin during commuting.5-7 For example, carpets, which are found in most car interiors, have been linked to high perfluorocarboxylic acid (PFCA) levels in indoor dust. 6,8 Water resistant sprays, known to contain PFAS, are often used to treat most car surfaces to prevent stains and/or to reduce wear.7 Beyond vehicle-originating sources, the operator of the vehicle can also unknowingly introduce PFAS into the cabin. One such example is the presence of child car seats, which have been found to contain fluorotelomer alcohols (FTOHs) and PFCAs.9

In addition to direct contact with PFAS-containing materials in vehicle cabins, inhalation and/or ingestion of dust could also be a route of exposure. Interestingly, studies have shown that PFAS concentrations measured in human serum were correlated with PFAS concentrations found in household dust.8,10 As such, dust has been shown to be a useful composite matrix for identifying presence of PFAS environments.6,11-15 For larger built environments, such as manufacturing plants, settled dust has been used to assess worker safety and overall PFAS exposure. 16,17 In a study by Zhang et al.,18 dust was used to assess PFAS burden across both urban and industrial settings,19 while several other studies have used dust to assess the infiltration of PFAS into homes neighboring manufacturing facilities.20 More recently, there has been a shift toward focusing on smaller built environments, where there is

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less air volume and square footage for dust to occupy. Childcare environments, non-residential campus spaces (e.g., classrooms), and fire stations are among the smaller indoor environments that have been investigated for PFAS using dust,13-15,21-23 with a high frequency of PFAS detected within these spaces. The potential of using dust in other small built environments, such as inside a vehicle's cabin, has yet to be considered, despite the fact that people in the U.S. spend approximately 392 hours per year in cars, on average.24 Previous research has shown that dust inside car interiors can contain several legacy and emerging contaminants, such as brominated flame retardants,25 organochlorines,26 and other novel chemicals.27,28 Thus, due to the association of dust with PFAS, the sampling of dust within vehicle cabins could offer insight into this underexplored PFAS exposure route.

The purpose of air conditioning (AC) filters is to clean circulating air within indoor spaces, by capturing dust, pollen, and other air-borne particulates. In our previous study,15 we exploited the principal function of AC filters (e.g., collecting dust) to provide a composite dust sample capable of representing our potential indoor PFAS exposure. Other studies have also shown the utility of using AC filters as matrices to assess indoor PFAS levels.29,30 As a sampling strategy, AC filters offer the benefit of collecting a composite dust sample over a set period, while concomitantly taking advantage of the fact that these filters are generally regarded as throw-away materials (i.e., easy to obtain). Here, in this preliminary study, we exploited the use of vehicle cabin AC filters (n = 10) as a new exposure sampling matrix for interrogating the presence of PFAS within vehicle cabins. High performance liquid chromatographytandem mass spectrometry (HPLC-MS/MS) was employed to screen for 47 PFAS within the filters tested. For cabin AC filters, air can be continuously recycled within the vehicle cabin (air recirculation mode) and by using these filters, that capture the dust, these filters can provide an insight into our potential PFAS exposure within vehicles by constantly filtering the same indoor air. Conversely, engine filters, another type of air filter in vehicles, continuously intake outside air that is fed through a snorkel.31 Since engine filters continuously take up new outdoor air, the PFAS profile present in engine filters (n = 10, outdoor environment) were subsequently used in an initial comparison to the PFAS profiles present in cabin AC filters (indoor environment). To help validate the use of this new sampling matrix, we also examined new/unused filters that are commonly used to replace original manufacturer-based filters to assess PFAS background. The potential of using this new car sampling strategy for future studies is discussed.

#### Methods 2.

#### Sample collection and preparation

**2.1.1 Sample collection.** Spent vehicle cabin AC (n = 10)and engine filters (n = 10) were obtained from local auto shops in Gainesville, Florida. To test whether the filters themselves contained PFAS prior to use, new filters, equivalent to those analyzed in this study, were purchased and tested for background PFAS levels alongside the analysis of the used filters

(new filters: n = 5 for cabin AC filters and n = 7 for engine filters). Filter brands, vehicle models (from which filters were removed from) and any additional information provided by the auto shops are included in SI Table S1. Cabin filters, CAF4-1 and CAF4-2, were two filters from the same car. Note that for this preliminary assessment, the collection of filters was opportunistic, i.e., all filters had varying time periods of usage and were not collected from matching vehicles (cabin AC vs. engine). Collected used air filters were removed during vehicle service and were immediately stored in sealed PFAS-free XL Ziploc bags upon collection, while new air filters were kept in their original packaging until analysis.

2.1.2 Sample preparation. A triplicate set of  $2 \times 30$  cm swatches were cut from used filters with the aim to include a set of swatches with a varying range of visible dust saturation on the filter. Duplicate swatches were sampled for each new, unused filter. Samples were handled with gloves over PFAS-free aluminum foil, both of which were replaced between each sample. Stainless-steel scissors were used to cut filter swatches and were cleaned three times with methanol and Kimwipes between each sample to minimize cross contamination. On four occasions, the methanol-cleaned scissors were immersed into 50 mL centrifuge tubes filled with Optima-grade methanol, and this methanol was analyzed for PFAS to test whether the scissors were sufficiently cleaned to prevent cross-contamination between samples; no contamination was observed in the scissor blanks. Each filter swatch was rolled up into a prelabeled Fisherband 50 mL centrifuge tube (Fisher Scientific) and weighed. Samples were kept at room temperature until PFAS extraction.

#### 2.2 Standards and reagents

A mixture of 19 isotopically labeled PFAS standards were purchased from Wellington Laboratories Inc. (MPFAC-24ES, Guelph, ON, Canada) and 47 native PFAS standards were purchased from Wellington Laboratories Inc., Oakwood Products Inc. (Estill, SC, USA), Chiron and Synquest Laboratories Inc. (Alachua, FL, USA). In total, our native screening mix had 13 PFCAs, 10 perfluoroalkyl sulfonic acids (PFSAs), 5 hydrogensubstituted perfluoroalkyl carboxylic acids (H-PFCAs), 6 fluorotelomer carboxylic acid (FTCAs), 3 fluorotelomer unsaturated carboxylic acid (FTUCAs), 4 fluorotelomer sulfonic acids (FTSs), and 6 polyfluoroalkyl phosphate esters (PAPs). Information regarding the specific isotopically labeled and native PFAS standards utilized in this study can be found in SI Tables S2 and S3, respectively. Reagents used for PFAS extraction and instrumental analysis included water, methanol, ammonium hydroxide, ammonium acetate, formic acid (all Optima grade), and were purchased from Fisher Chemical (Bridgewater, NJ, USA).

#### 2.3 PFAS extraction

PFAS extraction was adapted from a previously published method.15 Each sample was spiked with 30 μL of an isotopically labeled internal standard mixture (SI Table S2). Four empty centrifuge tubes were also included, passing through all

extraction and analysis steps, serving as extraction blanks. A total of 15 mL of 0.3% methanolic ammonium hydroxide was added to samples followed by vortexing for 15 s. The samples were then sonicated for 30 min and vortexed for an additional 15 s. Samples were then centrifuged for 5 min at 4000 rpm. Supernatants were transferred to pre-labeled 50 mL polypropylene centrifuge tubes. Extraction was repeated once more, and the two supernatants were combined. The combined extracts were then evaporated to 5-6 mL under a gentle stream of ultra-high purity nitrogen. Partially evaporated extracts were then purified with 50 mg of Supelclean ENVI-Carb graphitized activated carbon (120-400 mesh). After vortexing for 30 s and centrifuging for 10 min at 4000 rpm, purified extracts were transferred to new pre-labeled 15 mL Fisherbrand polypropylene centrifuge tubes (Fisher Scientific) and evaporated to 1 mL. Evaporated extracts were aliquoted into autosampler vials and stored at -20 °C until analysis.

#### 2.4 PFAS analysis

Filter extracts and blanks were analyzed for 47 PFAS via HPLC-MS/MS, with use of a Thermo Vanquish UHPLC system coupled to a Thermo Quantis triple quadrupole mass spectrometer (Waltham, MA, USA), utilizing electrospray ionization in negative mode. Chromatographic separation of PFAS was performed using a Phenomenex Gemini C18 column, using a gradient elution of water and methanol mobile phases (at a flow rate of 0.4 mL min<sup>-1</sup>), both with 5 mM ammonium acetate. A PFAS-free replacement kit was installed on the HPLC-MS/MS system to help reduce background PFAS contamination. Each PFAS was analyzed using scheduled selective reaction monitoring, examining two transitions for each PFAS (when possible). The primary transition was used for quantification, while the secondary transition was used for confirmation. Additional details regarding instrument parameters and analyte scan parameters can be found in SI Tables S4 and S5, respectively.

#### 2.5 Data analysis

Chromatographic peaks were manually integrated using QuanBrowser (Xcalibur v4.1) software. Peaks were reported below the limit of quantitation (<LOQ) if the peak height was <10× the signal-to-noise (S/N) but greater than the limit of detection ( $>3 \times$  the S/N). A 19-level series of calibration solutions were analyzed with the samples, and the resulting linear regression curves were used to calculate PFAS concentrations. If no labeled internal standard was available for a native PFAS, another labeled PFAS, similar in retention time or structure, was used. Final concentrations were normalized to the weight of the extracted filter swatch, reported as ng PFAS per gram of filter. PFAS concentrations for each replicate are provided in SI Table S6, while PFAS concentrations per filter (only when all three replicates had concentrations >LOQ) are found in SI Table S7. When PFAS were detected in filter blanks but were <LOQ, these instances are highlighted in SI Table S6 but were not subtracted from any final concentrations. However, quantifiable concentrations found in cabin filter Blank-CAF2 and

engine filter Blank-EF-5 were subtracted from the final PFAS concentrations for the corresponding used filter.

For data interpretation, summary statistics were generated in two manners. Since all filters collect dust heterogeneously over their lifetimes, (1) detection (LOD < x < LOQ) and quantitation (>LOQ) frequencies were calculated across all filter replicates (10  $\times$  3 = 30 and 10  $\times$  3 = 30, cabin AC and engine filters, respectively). This statistical format, labeled "replicates" allowed for the overall assessment of PFAS frequency regardless of filter type, dust saturation or location on filter. Secondly, (2) a more conservative summary statistical analysis was performed on only those filters which had quantifiable levels (>LOQ) available for all three replicates of a filter. This statistical format, labeled "filters" allowed for the analysis of PFAS that were strongly represented within filter dust, regardless of location on the filter tested, since they were found on all replicates for a filter. A table with all summary statistics is shown in Table 1 (an expanded version of this table is in SI, Table S8). For the calculation of summary statistics (e.g., median and mean), only those extracts with PFAS concentrations above LOQ were used. The goal of this study was to show that car filters can be used as a matrix to assess PFAS levels in vehicle cabins. In this manner, we decided to only show and discuss PFAS that provided concentrations (>LOQ), to emphasize that these were the PFAS most readily quantified.

## 3. Results & discussion

#### 3.1 Are PFAS captured in cabin AC filters?

The first examination was to determine whether PFAS were trapped on cabin AC filters and subsequently, which PFAS were most frequently detected. In this proof-of-concept study, we did not control the length (e.g. days) of filter use, vehicle type that the filter was removed from (e.g., truck or sedan), cabin AC filter model, or brand. Rather the emphasis of this study was to demonstrate the feasibility of sequestering PFAS on randomly donated cabin AC filters - representing a wide variety of combinations of the filter conditions noted above. Dust collection on cabin AC filters was suspected to be a heterogenous process, which was observed. The first comparison performed in this study was to examine the PFAS content across all cabin AC filter "replicates" (10 cabin AC filters × triplicate analysis of each cabin AC filter = 30 filter extracts). First, the number of PFAS detected (>LOD) across all cabin AC filter extracts was performed. Of the 47 PFAS monitored, 34 PFAS were detected in at least one filter replicate, as shown in SI Table S6. A total of 14 PFAS had a detection frequency above 75% across all replicates (with 7 PFAS demonstrated 100%), which included 8 PFCAs: perfluorooctanoic acid (PFOA, 100%), perfluorononanoic acid (PFNA, 100%), perfluorodecanoic acid (PFDA, 100%), perfluoroundecanoic acid (PFUnDA, 100%), perfluorododecanoic acid (PFDoA, 100%), perfluorotridecanoic acid (PFTrDA, 100%), perfluorotetradecanoic acid (PFTeDA, 97%), fluorohexadecanoic acid (PFHxDA, 90%), 1 PFSA: L-PFOS (83%), 3 H-PFCAs: 8 H-perfluorooctanoic acid (H-PFOA, 93%), 9H-hexadecafluorononanoic acid (H-PFNA, 90%), 11H-perfluoroundecanoic acid (H-PFUnDA, 100%) and 2 PAPs: 6:2

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Table 1 Table of" summary statistics for vehicle cabin AC and engine filters

		Cabin AC filters					Engine filters				
Class	Abbreviation	*DF filter "replicates" (%)	# "Filters" >LOQ $(n = 10)$	"Filter" median $(\log g^{-1})$	"Filter" mean $(\log g^{-1})$	"Filter" min/max $(\log g^{-1})$	*DF filter "replicates" (%)	# "Filters" $>$ LOQ $(n=10)$	"Filter" median $(\log g^{-1})$	"Filter" mean $(\log g^{-1})$	"Filter" min/max $(ng g^{-1})$
0	A - certain	4	,	ı		1					
PFCA	PFPeA	10	ī	9.0		2.0	1				
	PFHxA	53	2	2.6		1.5-3.7	I	1			
	PFHpA	70	2	1.8	2.7	9.7-7.0	10	1	1.9	1	1.9
	PFOA	100	6	1.2	4.4	0.2 - 25.5	53	3	0.2	1.9	0.1-5.4
	PFNA	100	6	1.7	1.9	0.3-5.7	09	2	1.9	1	0.5 - 3.4
	PFDA	100	6	2.4	9.9	0.3-40.8	50	21	0.4	2.4	0.1-10.9
	PFUnDA	100	10	6.0	1.2	0.1-2.8	40	4	0.2	0.7	0.0-2.4
	PFDoA	100	10	1.5	3.8	0.1-22.1	47	57	0.4	1.5	0.0 - 6.5
	PFTrDA	100	8	0.7	0.8	0.1-2.5	50	3	0.1	9.0	0.0-1.6
	PFTeDA	97	6	1.7	2.4	0.1-10.4	57	5	0.1	1.0	0.0-4.7
	PFHxDA	06	9	0.4	0.4	0.2-0.6	77	2	0.7	1	0.1-1.2
	PFODA	53	1	0.0	I	0.0	37	1	0.2	1	0.2
PFSA	PFBS	37	2	12.7		7.7-17.6	I	l	l		1
	$\sum$ PFHxS	40	2	1.5	I	1.4-1.6	I	I	I	1	I
	L-PFOS	83	9	3.9	3.8	1.1-7.0	3		1	1	1
	Br-PFOS	53	1	1	1	I	I	I			1
H-PFCA	H-PFHpA	40	1	8.1	1	8.1	27	I	1	1	1
	H-PFOA	93	9	1.3	1.8	0.2-5.1	23	1	0.31	I	0.31
	H-PFNA	06	8	0.7	6.0	0.2-1.9	63	4	0.2	0.3	0.1-0.6
	H-PFUnDA	100	7	9.0	6.0	0.2-1.7	80	2	0.5	1	0.2-0.7
FTCA	6:2 FTCA	10	I	1	I	1	I	I	I	1	I
	8:2 FTCA	3	I	1	I	I	10	I	I	I	I
	5:3 FTCA	57	4	1.2	1.3	0.7-2.2	40	1	2.4	1	2.4
	7:3 FTCA	17	1	1	I	1	I	I	I	I	I
	8:3 FTCA	13	I	1	I	I	10	I	I	I	1
FTUCA	6:2 FTUCA	10	1	14.2	1	14.2	0	I	I	1	1
	8:2 FTUCA	50	3	0.1	8.0	0.1-2.4	20	2	0.14	I	0.03-
	10:2 FTUCA	63	1	3.1	I	3.1	20	1	0.13	1	0.13
FTS	4:2 FTS	1	I	1	1	1	13	I	I	1	1
	6:2 FTS	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	8:2 FTS	73	4	2.0	2.0	1.6-2.5	30	1	1.8	I	1.8
	10:2 FTS	63	2	4.0	4.2	2.1-6.7	13	1	15.8	1	15.8
PAP	6:2 diPAP	93	6	45.9	55.7	15.7-	87	9	1.7	1.9	1.4-2.8
						137.3					
	6:2/8:2 diPAP	08	9	9.6	12.4	6.6 - 26.1	33	Η.	9.0		9.0
	8:2 diPAP	70	4	4.1	5.4	2.9 - 10.5	17	I		1	1
	6:2 triPAP	8		I	I		7		I	I	Ι

<sup>a</sup> For the summary statistical calculations (mean/median), only those extracts with concentrations above LOQ were used. The (\*) indicates that detection (LOD < x < LOQ) frequencies were calculated across all filter replicates (10 filters  $\times$  3 replicates = 30 extracts). This statistical format, labeled "replicates" allowed for the overall assessment of PFAS frequency regardless of filter type, dust saturation or location on filter (data shown in SI Table S6). A more conservative summary statistical analysis was performed on only those filters which had quantifiable levels (>LOQ) available for all three replicates of a filter. This statistical format, labeled "filters" allowed for the analysis of PFAS that were strongly represented in vehicle cabin AC dust, regardless of location on the filter tested, since they were found on all replicates for a filter (data shown in SI Table S7).

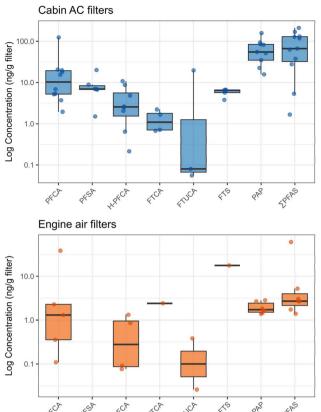


Fig. 1 Boxplots showing the concentrations (ng per g filter, log scale) of different PFAS classes found in cabin AC filters and engine air filters. PFSA were not detected in any of the engine air filters analyzed. Jittered points in the boxes represent individual sample values. The horizontal line indicates the median concentration, while the whiskers represent the range.

fluorotelomer phosphate diester (6:2 diPAP, 93%) and 6:2/8:2 fluorotelomer phosphate diester (6:2/8:2 diPaP, 80%). This initial survey answered the question regarding whether PFAS were present in cabin AC filters and further highlights the capability of cabin AC filters to trap a wide variety of PFAS.

The second evaluation was made across cabin AC filters, now only investigating the PFAS which were quantifiable (>LOQ) in all three replicates of a "filter". More specifically, concentration, reported as both median and mean, were then only reported for each filter only if all three replicates had a concentration above LOQ (Tables S7 and S8). In this manner, of the 10 cabin AC filters tested, 28 different PFAS fit the above criteria and were quantified. As shown in Table 1, there were 16 PFAS quantified in over 50% of the filters tested (e.g., since 10 filters were tested, those PFAS with ≥5 instances where PFAS concentrations >LOQ in all replicates). Among these PFAS (shown in Table 1), the top-5 by median concentration were 6:2 diPAP ( $46 \text{ ng g}^{-1}$ ), 6:2/8:2diPAP (9.6 ng  $g^{-1}$ ), L-PFOS (3.9 ng  $g^{-1}$ ), PFDA (2.4 ng  $g^{-1}$ ) and both PFNA and PFTeDA (1.7 ng  $g^{-1}$ ). Across filters, the median of  $\Sigma PFAS$  was 92  $\pm$  47 ng g<sup>-1</sup> (median absolute deviation, as shown in SI Table S7), while the mean was 95  $\pm$  66 ng  $g^{-1}.$  The data described demonstrates that these filters capture both

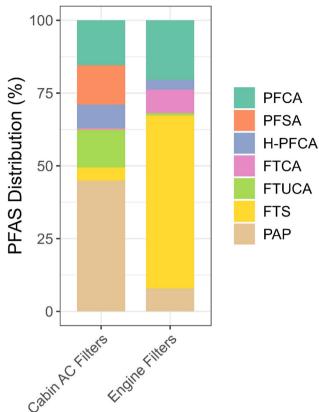


Fig. 2 Comparison of ∑PFAS profiles relative to cabin AC and engine filters. Distribution was represented as the taking the % of ∑PFAS per class over the ∑PFAS quantified for all cabin AC and engine filters. The abbreviated PFAS classes are: perfluoro carboxylic acids (PFCAs), perfluoro sulphonates (PFSAs), fluorotelomer carboxylic acids (FTCA), fluorotelomer unsaturated carboxylic acids (FTUCA), hydrogen substituted PFCA (H-PFCAs), fluorotelomer sulphonates (FTSs), and polyfluoroalkyl phosphate esters (PAPs).

a wide variety of PFAS and at elevated concentrations, highlighting the potential application of using these filters as sampling devices to estimate vehicle cabin PFAS exposures.

In a previous study examining the presence of 92 PFAS on residential and campus building AC filters,15 27 PFAS were observed. Campus building filters (and to a lesser extent, residential filters) clean air from large, confined spaces, thus it was expected that the  $\sum$ PFAS concentration levels would be higher than those reported here in vehicle cabin filters. Timshina et al.¹⁵ found that the median ∑PFAS concentration across filters tested was 104 and 288 ng g<sup>-1</sup> (campus and residential households, respectively), which were higher than the median observed for vehicle cabin AC filters (92 ng g<sup>-1</sup>). However, the ∑PFAS maxima were much higher in Timshina study, 15 at 553 and 2680 ng g<sup>-1</sup> (campus and residential households, respectively), compared to 210 ng g<sup>-1</sup> within vehicle cabin AC filters. A summary table of Timshina et al.15 study's findings compared to this study's findings is found in SI Table S9. Examining the individual PFAS found in Timshina et al.15 revealed that diPAPs were the dominant class of PFAS (by concentration), representing >95% of ∑PFAS. In vehicle cabin AC filters, diPAPs

represented a dominant, yet slightly smaller fraction at 45%. In vehicle cabin filters, PFCAs (including H-PFCAs) dominated the remaining distribution proportion, as similarly shown in Timshina et al.15 Interestingly, in vehicle cabin AC filters, four H-PFCAs were present, with H-PFNA, H-PFOA, H-PFUnDA detected in 90%, 93%, and 100% of filters, respectively, with the latter two H-PFCAs not detected in Timshina et al.15 While relatively new, the environmental presence of H-PFCAs has been attributed to usage as legacy PFAA replacements<sup>32</sup> due to regulations on long chain PFAS and are intended to be less persistent, yet hold most of the same properties with only slight chemical modifications. In Engelhardt et al.33 it was postulated that these hydrogen-substituted analogs may be used for the same applications as legacy PFAS, and found H-PFUnDA was frequently detected in human blood.

Back to PFCAs, the Timshina study15 showed that by frequency and concentration, PFCAs were dominated by longchain species (≥8 carbons). This was also observed in the vehicle cabin AC filters. While long chain PFCAs were also found to be frequently observed in a study by Besis et al.,29 this report contrasted due to the high abundance of short chain PFCAs in some samples. Besis et al.29 found 14 PFAS in trapped dust from AC filters collected from a variety of different indoor spaces, including coffee shops, cafes, and restaurants. These studies highlight that AC filters can effectively trap PFAS (via dust) and that the resultant PFAS profiles likely reflect the varying environments serviced by the AC filters.

#### 3.2 Other considerations for using vehicle cabin AC filters

One essential question about using vehicle cabin AC filters as sampling matrices pertained to whether the cabin filters themselves had background PFAS levels. In our previous study,15 looking at PFAS in residential and campus filters, no PFAS background was observed in blank filters. However, in that study, identical AC filters were readily available. When vehicle cabin AC filters were obtained from the local auto shop in this study, it was difficult to obtain the exact same filter in brand new condition, thus in many cases, we found equivalent replacement filters for blank testing (n = 7, filter matches are shown in SI Table S1). Overall, 9 out of the 10 blank vehicle cabin AC filters (run in duplicate) had no PFAS detected. One filter (Blank-CAF2-A, SI Table S6) had detectable levels of PFAS in one of the duplicates. In that one blank replicate, PFHxA, PFOA, PFDA, PFDoA, PFTeDA, and PFHxDA had quantifiable levels (>LOQ). For engine filter blanks, one filter (Blank-EF-5) had quantifiable levels of PFOA in one of the replicates. To be conservative, the cabin filter background concentrations were directly subtracted from vehicle cabin AC filter CAF4-1 and CAF4-2, likewise, engine filter Blank-EF-5 was directly subtracted from engine filter EF-6, both of which in these cases were direct matches between new and used filters. Due to being detected in multiple unused filters, 5H-perfluoropentanoic acid (5H-PFPeA) and 6:2 FTS were removed from analysis. The conclusion is that when using vehicle cabin AC filters for future studies, blank filters (preferred exact matches) should be tested and examined for

background PFAS, especially as PFAS profiles continue to expand.

The second consideration for use of vehicle cabin AC filters was the heterogeneity of dust collection on the filters. In this study, three separate swatches were cut from each filter, not to test precision, but rather to get an insight into the range of \(\sumething \text{PFAS captured across different regions of the filter. As shown}\) in SI Table S6, the average RSD of \( \sumeq PFAS \) across all 10 filters was 42%  $\pm$  25% (ranging from 13% to 81%). This relatively high RSD (i.e., heterogeneity of PFAS across each filter) highlights the importance of performing multiple replicates per filter or alternatively, scraping off the captured dust, as was done in Besis et al.29 Scraping off the dust allows the collection of dust into a separate container and thus resulting in a sample that is easier to homogenize; however, this approach can suffer from incomplete removal of dust (or loss of dust) from the filter that can be mitigated to a certain extent by extracting directly from the filter, as was performed in this study. More research is needed to better understand the factors that lead to the heterogenous distribution of dust on AC filters.

#### 3.3 Possible sources of PFAS in vehicle cabin AC filters

Vehicle cabin AC filters captured a myriad of PFAS, likely from an array of sources confined within vehicle cabins, plus contributions from outside the vehicle (e.g., particulates and debris entering the vehicle with the windows open) and outside items brought into the vehicle by passengers (e.g., personal care products, food contact materials).34-37 Possible PFAS sources within vehicle cabins include carpeting, upholstery, coatings, and plastic components.38-40 It is common knowledge that dust and other particulates can settle and accumulate in vehicles, with the chance to be recirculated (and captured) in AC filters. It is also worth noting that vehicle cleanliness may be a contributing factor to dust and particulate levels within a vehicle cabin. While PFAS sources may differ across vehicle cabins, a common trend emerged with 6:2 diPAP dominating as the most prevalent PFAS class captured within AC filters. The dominance of diPAPs measured in cabin AC filters was similar to indoor dust and AC filters examined in previous studies.5,15,21 Industrial manufacturing products, which contain PAPs, such as Masurf FS-130 and Masurf FS-240, are used in personal care products and cleaning products, respectively.5 As emerging alternatives to legacy PFAS, diPAPs have been shown to be prevalent in food packaging and other packaging materials,41,42 and as known precursors to the generation of PFCAs, their presence represents a depot of future PFAS release. 43,44 Pertaining to PFCAs, carpets have been shown to contain an abundance of PFAS, with PFDA and PFUnDA being the largest contributors, in comparison to other PFCAs.6 This study similarly found that PFDA was higher in concentration, on average, relative to other PFCAs. Like with carpets, upholstery can also be a source for PFAS. To minimize staining within vehicles, upholstery is often coated with PFAS-containing finishes.45 Furthermore, vehicle maintenance and care may also be a source of PFAS, with a new report emphasizing the levels of PFAS originating from washing and/ or cleaning a vehicle.46

# Communication

3.4 PFAS in engine air filters

Like cabin AC filters, vehicle engine filters also demonstrated the ability to capture PFAS, albeit to a lesser extent, as shown in Table 1. As with the vehicle cabin AC filters, the first examination of the engine filters focused on highlighting the PFAS content across all engine filter "replicates" (10 engine filters × triplicate analysis of each engine filter = 30 filter extracts). The number of PFAS detected (>LOD) across all engine filter replicates was 27 (out of 47 PFAS); however, only three PFAS had a detection frequency above 75% across all replicates and included 6:2 diPAP (87%), H-PFUnDA (80%), and PFHxDA (77%). Next, for the examination of engine filters, only the PFAS which were quantifiable (>LOQ) in all

three replicates of a "filter" (as previously performed for cabin AC

filters, describe above), were investigated (note that one engine

filter EF-7 was only analyzed in duplicate). Across the 10 engine

filters, 20 PFAS were detected in at least one filter (in all three

replicates). For engine filters (shown in Table 1), only four PFAS were present in at least 50% of the filters tested (*e.g.*, since 10 filters were tested, those with  $\geq$ 5 filters fit this criterion) and included PFDA, PFDoA, PFTeDA, and 6:2 diPAP. Among these PFAS found within all replicates of  $\geq$ 5 filters, the PFAS with the highest median concentration was 6:2 diPAP (1.7 ng g<sup>-1</sup>, with a mean of 1.4 ng g<sup>-1</sup>). Across engine filters, the median  $\Sigma$ PFAS concentration was 2

 $\pm$  2 ng g<sup>-1</sup> (median absolute deviation, as shown in SI Table S7).

PFAS are often incorporated into engine lubricants and oils to protect against wear, foaming, and corrosion.<sup>47</sup> For most vehicles, excluding exposed engine filters, the engine air filter is confined within a small box, protected from other areas of the engine, thus possibly limiting its potential to capture local PFAS contamination. Interestingly, of the PFAS analyzed, long-chain PFCAs were the major PFAS in engine specific lubricants in a previous study, 47 which was also observed in this study. Both PFCAs and PFSAs have been detected in ambient outdoor air,48,49 however, no PFSAs were detected in engine air filters, despite their common presence. In contrast to both homes and vehicular cabins, engine bays are subject to more extreme conditions (e.g., heat) that could result in the volatilization of PFAS from the filters (especially highly volatile species); therefore, future research should explore the role of environmental conditions onto PFAS capture on engine filters.

The engine filter blanks were relatively low to nonexistent in PFAS background, similarly to the cabin AC filters. Only one engine filter blank replicate had a PFAS concentration above the LOQ, which was Blank-EF-5 (PFOA, see SI Table S6, this value was subtracted from its matching filter). Also, similarly to the cabin AC filters, the variability within replicates of the same filter was broad, ranging from RSDs of 6% to 173%, perhaps reflecting the heterogeneity of dust captured within the engine filters. Engine filters can be purchased in a variety of shapes and sizes (e.g., round vs. flat), more research is needed to understand the effectiveness of capturing dust (and in turn, PFAS) in engine filters.

# 3.5 Initial comparison of PFAS levels in cabin AC and engine filters

The inclusion of engine filters into this study was to highlight the difference between PFAS on filters that purify used indoor air versus predominantly outdoor air. Concentrations of PFAS measured in cabin AC filters were more abundant (median  $\sum$ PFAS  $\sim$ 45×) than concentrations measured in engine air filters, as highlighted by Fig. 1, thus highlighting the disparity of PFAS burdens between the air purified by both filters. Median concentrations of 6:2 diPAP and ∑PFCAs were ~25-fold and ~200-fold higher in vehicle cabin AC filters than in engine filters, respectively. Both vehicle cabin AC filters and engine filters take in air; however, ambient outdoor air typically has a much lower PFAS concentration when compared to indoor air, 49,50 which was reflected in this study. Indoor spaces often employ re-circulation of air (similarly to options available in newer vehicles), which can, over a period of time and in a prolonged proximity to PFAS sources, exacerbate the levels. 50,51 PFAS were more diverse in cabin AC filters, in comparison to the engine filters tested, as shown in Table 1. The PFAS distribution was also compared between cabin AC and engine filters, as shown in Fig. 2. In this study, diPAPs were observed with the highest distribution in cabin AC filters, at 45%, while FTSs, dominated by one engine filter) and PFCAs had the highest distribution in engine filters (at 59 and 20%, respectively). In addition to the difference in concentration magnitude, PFAS were more abundant, overall, in the cabin AC filters. This study supports the idea that smaller indoor spaces (e.g., vehicle cabins) have the potential to be a source of PFAS exposure, though more studies are required to better elucidate the overall concern.

# 4. Conclusion

The primary objective of this study was to evaluate the feasibility of using vehicle cabin AC filters as a new sampling matrix, one that is readily available and is generally regarded as a throwaway material. The data described herein demonstrates that these filters can provide more information regarding potential PFAS burdens associated with vehicle usage, contrasting PFAS that were different between indoor and outdoor environments, for cabin and engine filters, respectively. In this study, higher concentrations, detection rates, and diversity of PFAS were noted in cabin AC filters, when compared to engine filters. As diPAPs were found frequently within indoor spaces, other PFAS precursors (FTOH, FOSEs) should be included in future analyses. More research is needed to explore these implications, for example, by collecting filters with controlled meta data (filter type, dimensions, duration of use, vehicle type, and passenger behavior) to better define the relationship of sources to PFAS profiles. Regarding passenger behavior, factors such as eating, applying makeup, cleanliness, number and type of passengers, and frequency of AC use, among other behavioral differences, could be studied for PFAS exposure within vehicle cabins using AC filters.

#### **Author contributions**

John Bowden: conceptualization, validation, resources, data curation, writing – review and editing, visualization, project administration, and funding acquisition. Qaim Mehdi: validation, investigation, and formal analysis. Alina Timshina: conceptualization, validation, resources, investigation, data curation, supervision, visualization, software, methodology, and writing – review and editing. Jack Arnold: conceptualization, validation, resources, investigation, data curation, supervision, visualization, software, and writing – original draft.

## Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

# Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information is available. See DOI: https://doi.org/10.1039/d5ay01255d.

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