Broad Spectrum PFAS – Detecting Precursors

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Overview

'Forever pollution': Explore the map of Europe's PFAS contamination

- Characteristics of PFAS •
- Perfluoroalkyl Substances Perfluoroalkyl ۲ Acids (PFAAs)
- Polyfluoroalkyl Substances PFAA precursors
- PFAS Manufacture
- PFAS in Products
- PFAS Chemical Analysis
 - Target Methods (e.g. USEPA 1633)
 - Non Target Approaches
 - Combustion Ion Chromatography
 - TOP assay
- Summary ۲





Projected fluorotelomer production in 2019 of 42,500 tonnes.

After Global Market Insights, 2016. Projected compound annual growth rate of 12.5% from 26,500 tonnes in 2015.

https://ipen.org/sites/default/files/documents/the_global_pfa s problem-v1 5 final 18 april.pdf



Characteristics of PFAS

Extreme Persistence

PFAS show no sign of biodegradation and have been termed "forever chemicals" –due to multiple robust C-F bonds which also protect C-C bonds from attack

Mobility

PFAS tend to be very mobile in the environment as they are soluble in water (unlike most other POPs)

Bioaccumulation

Some PFAS bioaccumulate and biomagnify - long chain PFAS concentrate humans via renal reabsorption, other mammals can excrete at much faster rates

Toxicity

There are very low (<10 ng/L) and diminishing regulatory acceptance criteria (drinking water standards) as more is known about the toxicity of specific PFAS

Surfactants

Amphiphilic PFAS stick on surfaces / interfaces when at higher concentrations





"Treatment of contaminated drinking water, are estimated to range between 17 and 170 billion EUR over the next 20 years"



Per- & Polyfluoroalkyl Substances





Perfluoroalkyl Acids (PFAAs)

- Were termed "chemical rocks" with stone arrowhead analogy –extreme persistence
- Previously called Perfluorinated Compounds (PFCs)
- Terminal transformation products of polyfluoroalkyl substances
- Long chain vs short chain defined by bioaccumulation potential
- Generally, C1 C20+ PFAAs
- Lipo- and hydrophobic, usually anionic
- Higher water solubility as chain shortens
- Long chain more regulated, but short chain are focus of increasing regulation
- Amphiphiles ≥4 perfluoroalkyl carbons
- Also includes perfluroalkylphosphinic (PFPiS) and phosphonic acids (PFPAs) (PFPiAs) plus perfluoroalkyl ethers (e.g. GenX)

arrows represent long chain & bioaccumulative PFAAs





Polyfluoroalkyl Substances -PFAA Precursors

- Polyfluorinated precursors will biotransform to make PFAA's as persistent "dead end" daughter products, some are anionic
- Fluorotelomer precursors form PFCAs i.e. PFOA, PFHxA
- Some precursors are cationic (positively charged) or zwitterionic (mixed charges)
- Environmental fate and transport will be complex as PFAS in AFFF comprise multiple chain lengths and charges
- Proprietary polyfluorinated precursors to PFAAs dominate most firefighting foams –there are a very limited number of analytical standards, so they largey go undetected using conventional analyses





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"chemical rocks", like stone arrowheads



Manufacture of PFAS

PRODUCTION Electrochemical Fluorination (ECF) PROCESS Fluorotelomerization (FT) Oligomerization⁽¹⁾ CHARACTERISTIC Linear + branched isomers, mixtures of homologues Linear isomers only, mixture of homologues Linear and branched perfluorinated alkyl or alkylene groups that may be connected by a fluorinated and byproducts (including C4-C14 PFCAs) and C4-C14 PFCA as by-products polyether(2, 3, 4, 5) $CTFE^{*(4)}$ STARTING PASF PACF **PFAIs** $TFE^{(2,3)}$ HFPO⁽⁵⁾ MATERIAL (e.g., POSF, etc.) (e.g., POCF, etc.) INTERMEDIATES HFPO-DA Highly Tetramer pentamer fluorinated fluoride, other FTIs X-FASA/Es hexamer, and vinvl sulfonvl perfluoropolyheptamer halide ether carbonyl fluorides oligomers FTSAs, FTOHs FTOs **PFSAs** Non-polymers Side-chain PFCAs (e.g., PFNA Non-polymers Side-chain Fluoropolymers, Fluoropoly Fluoropoly PRODUCTS ethers, HFPO-(e.g., PFOS, (e.g., EtFOSA, fluorinated PFOA, (e.g. FTOHs, fluorinated amine ethers, ADONA PFHxS, polymers (e.g PFHxA) FTSs, n:2 polymers, (e.g., sulfonamide DA, HFPO-TA **EtFOSE** SAmPAPs) diPAPs) acrylates) etc.) acrylates) MAJOR USES SURFACTANT SURFACE PROCESS AID PROCESS AID AFFFs, AFFF SURFACE HYDRAULIC SURFACTANT. SURFACTANT TREATMENT FOR PTFE, FOR PVDF SURFACE TREATMENT. FLUIDS. EMMULSION SURFACE EMMULSION TREATMENT, ETC. FEP, PFA, TREATMENT, ETC REFRIGERANTS. SURFACE TRANSFORMER PVDF INSECTICIDE etc TREATMENTS ETC. FLUIDS TURBINE IMPELLENTS

Acronyms not previously defined: PASF = perfluoroalkane sulfonyl fluoride; PACF = perfluoroalkyl carbonyl fluoride; PFAI = perfluoroalkyl iodide; FTOs = fluorotelomer olefin; TFE =

tetrafluoroetheylene; CTFE* = chlorotrifluoroethylene and other unsaturated fluoromonomers; X-FASA/Es = (N-methyl/ethyl) perfluoroalkyl sulfonamide/ sulfonamidoethanol

<u>Three</u> main methods of manufacture:

1. Electrochemical Fluorination

- Used to manufacture branched and straight chain PFOS (~30/70%) and PFOA (~78/22%) PFOA
- perfluorosulfonates (PFSAs) e.g. PFOS, PFBS and perfluorocarboxylates (PFCAs) e.g. PFOA
- perfluoroalkylsulfonamides (e.g. FASAs, FASEs, FOSAAs, SAmPAP, diSAmPAP, PFASaAm, PFASaAm)

2. Fluorotelomerization

Used to manufacture even carbon numbered PFCA or PFPA/PFPiA

and multiple precursors, generally linear PFAS, but also isopropyl isomers

- Perfluorocarboxylates (PFCAs) e.g. PFOA, TFA
- Fluorotelomer alcohols (FTOHs) e.g. 6:2 FTOH,
- Fluorotelomer sulfonic acids (FTSs) e.g. 6:2 FTS, 8:2 FTS
- Fluorotelomer carboxylic acids (FTCAs) Fluorotelomer betaines (FTBs),
- Polyfluoroalkyl phosphates (PAPs) such as the diPAPs etc. etc.

3. Oligomerisation

 Used to manufacture perfluoroalkylethers (PFECAs) and perfluoroalkyl ether sulfonate (PFESAs) such as: GenX hexafluoropropylene oxide dimer acid (HFPO-DA), HFPO-TA, ADONA



e.g., Polyfluorinated – 6:2 fluorotelomer sulfonic acid (6:2 FTS)





3H-perfluoro-3-(3-methoxy-propoxy) propanoic acid (ADONA)



Hexafluoropropylene oxide dimer acid (HFPO-DA or "GenX")





PFSaAm Perfluoroalkyl sulfonamido amine PFSaAmA Perfluoroalkyl sulfonamide amino carboxylate

Field & Sedlak 2017. SERDP.

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Fluorotelomer Foams



Fluorotelomer Precursor Partial Defluorination - Chain Shortening

				Product Descentage Polative to Initial DEAA Dresurger Concentration						
				PFBA	PFPeA	PFHxA		PFOA	PFNA	Total
Type of PFAA Precursor	Inoculum	Reference	Duration	(C4)	(C5)	(C6)	(C7)	(C8)	(C9)	PFCAs
	Ethanol-degrading mixed microbial	Dinglasan et al.						201		201
8:2 FtOH	enrichment	2004	90 days					3%		3%
8:2 FtOH	Activated sludge	al. 2005	90 days			1%		6%	<0.2%	7%
6:2 mono-PAP	Wastewater mixed liquor	Lee et al. 2010	90 days		<1%	2%	8%			10%
6:2 di-PAP	Wastewater mixed liquor	Lee et al. 2010	90 days		<1%	6%	7%			13% (6.5%)
6:2 FtOH	Soil	Liu et al. 2010	84 days	0.8%	4.2%	4.5%				9.5%
6:2 FtS	Activated sludge	Wang et al. 2011	90 days	0.14%	1.5%	1.1%				2.7%
8:2 Ft stearate monoester	Soil	Dasu et al. 2012	80 days			0.16%	0.4%	1.7%		2.3%
6:2 FtOH	Anaerobic digester sludge	Zhang et al. 2013	200 days			<0.4%				<0.4%
8:2 FtOH	Anaerobic digester sludge	Zhang et al. 2013	200 days					0.3%		0.3%

Precursors slowly form PFAA's –some are shorter chained PFAA's vs parent

PFAS Biotransformation Funnel

"Dark Matter

Proprietary PFAS molecules that are specific to certain products

Parent **polyfluorinated** precursors in products e.g. in AFFF

Polyfluorinated daughter biotransformation intermediates

Common semi-stable polyfluorinated daughter intermediates accumulating (especially under anaerobic conditions)

Terminal common **perfluorinated** daughter products (PFAAs) (formation favored under Adapted from Ross et al., 2016 aerobic conditions) 13

e.g. 8:2 FTSAS, PFHxSaAm

e.g. 8:2 FTSAS-SO e.g. 8:2 FTS e.g. PFOA, PFHxA, PFOS

Persist Indefinitely



Polyfluoroalkyl substances under scrutiny

C&en GLOBAL ENTERPRISE	Enter search terms	6:2 FTAB in UK waters - post				
PERSISTENT POLLUTANTS For the first time, US EPA orde	rs testing of a PFAS chemical	sewage treatment				
Cheryl Hogue C&EN, 2022, 100 (21), p 14 June 13, 2022	Cite this: C&EN 100, 21, 14	Canwick STW Canwick STW Canwick STW Countess Wear STW Canwick STW Canwick STW				
Abstract F ₃ C F	$F F F F$ $F F F$ H_{3C} H_{3C}	Bowerhill STW Canwick STW Millbrook Hull STW Avonmouth STW Southend STW Southend STW Avonmouth STW	F			
	6:2 FTAB	Southend STW Avonmouth STW				
Four companies must conduct inhalation toxic FTAB) under a June 6 order from the US Envir , 6:2 FTAB may present an unreasonable risk of insoluble solid, the manufacture, processing,	city tests on the firefighting foam ingredient 6:2 fluorotelomer sulfonamide alkylbetaine (6:2 onmental Protection Agency . Part of the family of per- and polyfluoroalkyl substances (PFAS of injury to human health or the environment, the EPA says. Because the chemical is an or use of 6:2 FTAB may lead to the formation of particles that workers could inhale, the agence	5:2 Canwick STW Canwick STW Canwick STW Canwick STW gency 0 1000 2000 3000 4000 5000 6000	7000			
https://pubs.cos.org/doi/f	ull/10.1021/con 10021 poleon1	Concentration (ppt)	Concentration (ppt)			

https://pubs.acs.org/doi/full/10.1021/cen-10021-polcon1





Broad Spectrum of PFAS

PFAS in Products



Paper and Food Packaging

- Side-chain fluoropolymers
- PAPs/diPAPs

 NEtFOSE, NEtFOSAA, PFBS, PFOA, PFHxA



Textile and Leather	Firefighting	WWTPs and Landfills	Metal Plating
 Polymers Polymer raw materials PFOA, FTOHs 	 PFOA, PFOS, PFHxS, PFHxSaAm C8/C6 fluorotelomers e.g 8:2FTAoS; 6:2FTAB PFECHS 	 n:2 FTUCA n:3 FTCA (5:3 FTCA) n:2 FTSA EtFOSA 	 PFOS 6:2 FTS, 8:2 FTS F53B PFECHS
	A CONTRACT		



Chemical Analysis



Laboratory Analysis

Targeted Analysis

- USEPA Method 537.1
 - 18 PFAS (12 PFAAs + 6 Other PFAS Including GenX)
- USEPA Method 533
 - 25 PFAS (16 PFAAs + 9 Other PFAS Including GenX)
 - Focuses on Short Chain

USEPA Method 1633

- Final Method January 2024
- 40 PFAS
- Required by EPA for all non-drinking water samples (e.g., groundwater, surface water, wastewater, soils, sediments, biosolids)
- DWI 47 PFAS
- Europe 20 / 24 PFAS



Non-Target Laboratory Analysis

Methods to Detect Non-Target Polyfluoroalkyl Substances

- Total Oxidizable Precursor (TOP) Assay
 - Converts Precursors to PFAAs
- Total/ Adsorbable/ Extractable Organic Fluorine by combustion ion chromatography (USEPA 1621)
 - All organofluorine compounds (~0.4 ppb detection limits)
- High Resolution Mass Spectrometry
 - Identifies Chemical Formula and Structure of Polyfluoroalkyl PFAS in addition to PFAAs
- Nuclear Magnetic Resonance (NMR)

Organofluorine Detection via Combustion Ion Chromatography

- Potential to detect all organofluorine compounds
- Rapid screening for PFAS, but higher detection limits vs LCMSMS
- No speciation of PFAS chain length, reports total fluorine in sample
- Burn either samples, adsorbent or extractant and measure fluoride released using ion chromatography
 - Total Organic Fluorine (TOF)
 - Adsorbable Organic Fluorine (AOF) ~0.4 ppb detection limits
 - Extractable Organic Fluorine (EOF) ~0.2 ppb detection limit
- AOF with USEPA 1621 will not detect ultrashort PFAS as not extracted
- Mixed mode extraction cartridges (WAX, WAX, HLB. WAX/GCB) more comprehensive for PFAS extraction

https://www.metrohm.com/en_us/discover/blog/20-21/history-of-metrohm-ic--part-6.html

TOP Assay

- All PFAS-containing firefighting foams (AFFF, FFFP, FP) contain proprietary polyfluorinated PFAA precursors for which analytical standards are not available 'Dark matter'
- Converts 'Dark Matter' PFAS into detectable PFAAs –to provide some quantification of precursors
- Used to identify source areas of AFFF, FFFP, FP impacts and essential to find PFAS in firefighting foams

Persistence of Perfluoroalkyl Acid Precursors in AFFF-Impacted Groundwater and Soil

Erika F. Houtz[†], Christopher P. Higgins[‡], Jennifer A. Field[§], and David L. Sedlak^{†*}

Figure 1. Average concentrations of perfluorinated sulfonates and carboxylates in AFFF formulations analyzed before (a) and after oxidation (b). Dates represent the years of manufacture of AFFF formulations analyzed in each category.

https://pubs.acs.org/doi/10.1021/es4018877

TOP Assay Chemical Oxidation – Analogous to Camp Fire

TOP Assay Output

Oxidation of Precursors to PFAAs with OH•

TOP Assay Applied to Surface Water from Recent C6 Fluorotelomer Foam Loss

PFAS Pre-Oxidation

Analysis

PFAS Post-Oxidation

(Oxidation)

Post Oxidation

Analysis

Data Quality Objectives

- Don't <u>Overcook</u> or <u>Undercook</u> TOP assay
 - Measured PFAA precursors have been (i.e., 6:2 FtS, FOSA, etc. <0) have been oxidized <u>–undercook test</u>
 - If Σ[Perfluoroalkyl carboxylates] post-TOP < Σ[Perfluoroalkyl carboxylates] pre-TOP, this may suggest that the digest conditions have become acidic, and carboxylates were destroyed - overcook test
 - [Perfluoroalkyl sulphonates] post-TOP ≈ [Perfluoroalkyl sulphonates] pre-TOP; concentrations should not go down, but they may rise slightly.

Guidance on Use and Interpretation of the TOP assay Erika Houtz PhD and Ian Ross PhD

Edited by Caitlin H. Bell • Margaret Gentile • Erica Kalve Ian Ross • John Horst • Suthan Suthersan With foreword by Thomas K. G. Mohr

TOP Assay Interpretation

Ross I. Houtz E., B.J., McDonough J. and Quinnan J., *Characterization and Treatment of Poly- and Perfluoroalkyl Substances (PFASs)*, in *Advances in Remediation, A Better Way Forward*. 2018, Arcadis. p. 63-76.

TOP Assay Advances

Science of The Total Environment Volume 909, 20 January 2024, 168145

Science of The Total Environment Volume 946, 10 October 2024, 174079

Advancing PFAS characterization: Enhancing the total oxidizable precursor assay with improved sample processing and UV activation

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Advancing PFAS characterization: Development and optimization of a UV- H_2O_2 -TOP assay for improved PFCA chain length preservation and organic matter tolerance

David Patch ^a, <u>Natalia O'Connor ^a, Ellie Ahmed ^a, Erika Houtz ^c, Michael Bentel ^b, Ian Ross ^d,</u> Jennifer Scott ^a, Iris Koch ^a, Kela Weber ^a $\stackrel{R}{\sim}$ 🖾

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https://doi.org/10.1016/j.scitotenv.2024.174079 🛪

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Highlights

- UV-activated H_2O_2 mineralizes up to ~1000 mg/L of total organic carbon.
- Optimal parameters identified include pH (5–9) and oxidant concentration (500 mM $\rm H_{2}O_{2}).$
- Sodium persulfate identified as an effective alternative to potassium persulfate for the UV-TOP assay.
- Tandem UV-H₂O₂-TOP assay demonstrates better preservation of longer PFCA chain lengths compared to thermal and UV-TOP.

Katerina Tsou, Edmund Antell, Yanghua Duan, Christopher I. Olivares, Shan Yi, Lisa Alvarez-Cohen,* and David L. Sedlak*

between branched and linear PFCAs was also integrated into the assay to enhance its ability to differentiate between PFASs produced by electrochemical fluorination and those produced by fluorotelomerization. In addition to its potential application in forensic studies, the improved TOP assay reduces uncertainties associated with incomplete oxidation of precursors or formation of ultrashort PFCAs that are not detected by standard analysis methods.

Data Quality Objectives

- Polyfluoroalkyl substances are the dominate PFAS in most commercial products
- These go undetected using conventional target analysis for PFAS
- Polyfluoroalkyl substances are precursors to PFAAs
- Precursor transformation rates are variable
- There are multiple chemical analytical methods available to assess PFAS more comprehensively
- Grouping PFAS may need to account for precursors

Implications of grouping per- and polyfluoroalkyl substances for contaminated site regulation

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Abstract

Per- and polyfluoroalkyl substances (PFAS) are a broad group of persistent organic compounds with vastly differing physicochemical and toxicological properties. Some jurisdictions have proposed to regulate PFAS as a single class to overcome the limitations of regulating such a diverse group on a chemical-by-chemical basis. Implications of regulating PFAS as a single class have been discussed for PFAS production and use, but equivalent discussion of implications for managing contaminated sites is largely lacking. This opinion piece summarizes the views of a group of environmental consultants, environmental regulators, land managers, and academics with significant experience in researching or managing PFAS. The group considered that neither a single PFAS class approach nor a chemical-by-chemical approach is well suited to managing risks from PFAS in a contaminated site setting,

approach is well suited to managing risks from PFAS in a contaminated site setting, and defining PFAS subgroups would have value. Second, some but not all in the group, hypothesize that PFAS properties that drive fate and transport are those that influence toxicity and bioaccumulation in animals. This may be a valuable observation for future discussions on dividing PFAS into subclasses for contaminated site regulation based on physicochemical properties rather than purely structural definitions.

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R YI: technical bulletin

CL:AIRE technical bulletins describe specific techniques, practices and methodologies relevant to sites in the UK. This bulletin describes per- and polyfluoroalkyl substances (PFAS) uses and identifies industries and activities which could potentially cause environmental impacts.

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An overview of the uses of PFAS to assist with identification of sites of concern

INTRODUCTION 1.

precursors) and perfluoroalkyl ethers.

Per- and polyfluoroalkyl substances (PFAS) are a broad group of over 12,000 synthetic chemicals [1, 2], widely applied for bulk industrial uses and in consumer applications since the 1930s [3, 4]. As a result of their detection in drinking water supplies, PFAS are the subject of accelerating environmental regulatory concern globally.

The initial regulatory focus was on two perfluoroalkyl acids (PFAAs), perfluorooctane sulfonic acid (PFOS) and perfluorooctanoic acid (PFOA), but attention has now widened to an array of additional PEAS, including short chain PEAS, polyfluorinated PEAS (PEAA

TB 22

(March 2023)

CL:AIRE Technical Bulletin TB19 Managing Risks and Liability associated with Per- and Polyfluoroalkyl Substances (PFAS) should be used as a reference document to this bulletin. It provides summary of PFAS-contaminated soil and groundwater.

pose a risk to environmental or human health receptors.

the environment, when released, a significant mass of amphiphilic or cationic/zwitterionic PFAS can remain at release sites and continue to represent a source of PFAS [10, 11, 16].

Some PFAS may be regarded as being ubiguitous at very low concentrations. The presence of PFAS in rain and the spreading of Class B firefighting foams continue to be stored and used for fire biosolids wastes from wastewater treatment plants to land as a fertiliser, can result in widespread (low level) detections of PFAS in soils [17-21].

This bulletin aims to identify industries and activities which could cause significant losses of PFAS to the environment, which may information on PFAS chemistry; their origin; their properties, fate and continue to act as a source of PFAS to potentially impact receptors. A behaviour: their regulation; risk assessment of PFAS; and remediation brief description of the types of PFAS that are associated with differing activities is then provided.

This technical bulletin, describing PFAS uses and potential sites of As a result of the widespread use of PFAS in multiple applications, a concern, aims to provide an overview of some of the bulk, legacy comprehensive guide to all potential source areas is not possible. A review, cataloguing which PFAS may be used in multiple applications [23-31], which provide a detailed overview.

Firefighting foams containing PFAS termed fluorosurfactants have been used for extinguishment of flammable liquid (Class B) fires since 1962 [32], and continue to be widely used.

- Aqueous Film Forming Foams (AFFF) (Figure 1) and alcohol resistant derivatives (AR-AFFF):
- Film Forming Fluoroprotein Foams (FFFP) and alcohol resistant derivatives (AR-FFFP): and
- Fluoroprotein Foams (FP) and alcohol resistant derivatives (AR-FP)

suppression, fire training, equipment testing and flammable vapour suppression at multiple sites where there is a fire risk from holding flammable liquids such as hydrocarbons (e.g. gasoline, kerosene, diesel, greases, tars, oils), alcohols, solvents and speciality chemicals, If a flammable liquid was or is handled or stored at any location, the

https://www.claire.co.uk/home/news/1782-pfas-bulletin

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and/or dispersive uses of PFAS, to act as a guide to site investigations of land by assisting with identification of activities and by differing industries has recently been published [22, 23]. The which may lead to PFAS being present at concentrations which could multitude of PFAS uses has been described in various publications High concentrations of PFAS can remain at locations where PFAS 2. FIREFIGHTING FOAMS have been lost to ground, acting as ongoing sources to groundwater or surface waters [5-15]. PFAS may remain in soils and be associated with low permeability deposits [6] or concrete surfaces [8, 9]. Activities where the ongoing and legacy releases of liquid forms of PFAS (in solution) have likely occurred, have been prioritised, as The types of firefighting foams that contain PFAS include: these uses could result in a significant mass of PEAS release to the environment over time. Although many PFAS can be highly mobile in