arpav Agenzia Regionale per la Prevenzione e Protezione Ambientale del Veneto

IRSA CNR

DETERMINAZIONE DI PFAAs IN MATRICI AMBIENTALI: ESPERIENZE E CRITICITA'.

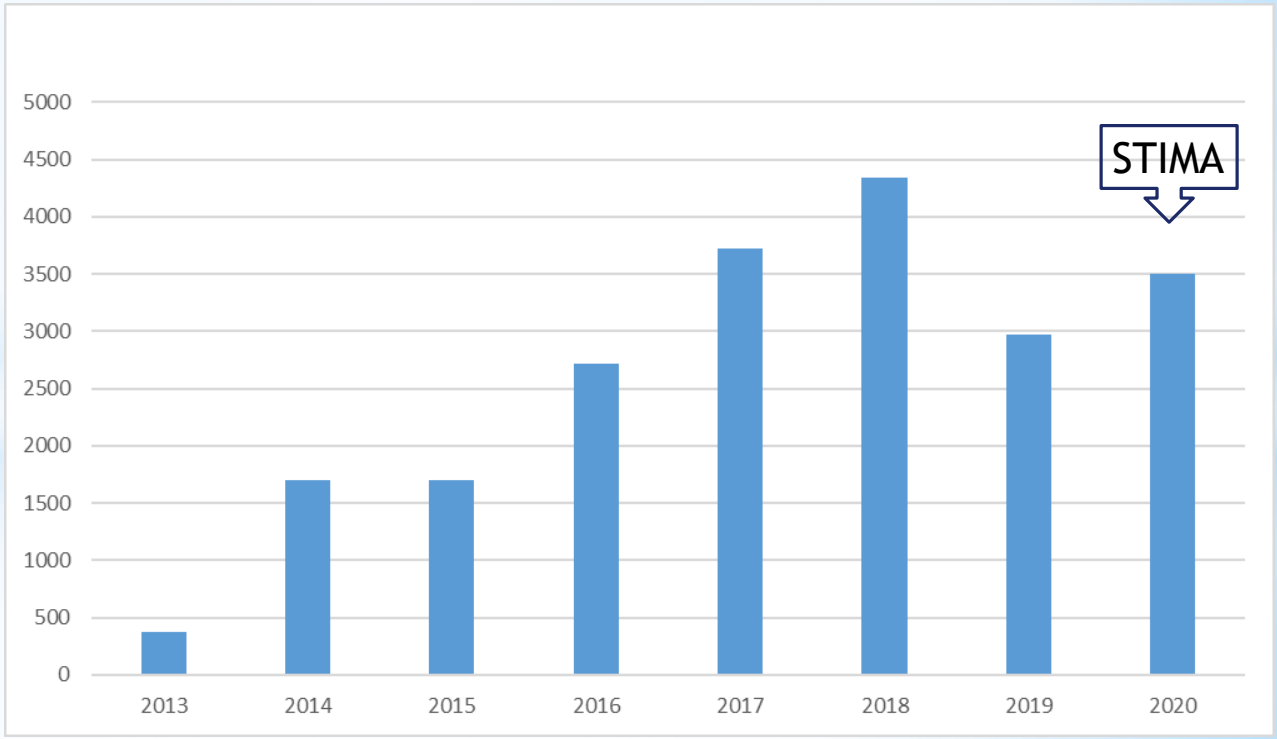
Acque del Chiampo s.p.a.
Servizio Idrico Integrato

CRONISTORIA ACCREDITAMENTO PFAAs in AdC: Metodiche e matrici

- 2015: EPA 537 «*Acque destinate al consumo umano*»: 14 isomeri lineari + 4 isomeri ramificati
- 2016: EPA 537 «*Acque destinate al consumo umano*»: 5 isomeri ramificati di PFOS e PFOA
- 2017: ASTM D 7979 «*Acque di scarico trattate*»: isomeri lineari
- 2018: ASTM D 7979 «*Acque superficiali*»: isomeri lineari
- 2019: EPA 537.1 «*Acque destinate al consumo umano e di falda*»: isomeri lineari + ramificati
- 2020: ASTM D 7979 «*Acque destinate al consumo umano*»: isomeri lineari

ANALISI PFAAs EFFETTUATE IN AdC A PARTIRE DA AGOSTO 2013:

- 2013: 373
- 2014: 1702
- 2015: 1704
- 2016: 2717
- 2017: 3725
- 2018: 4343
- 2019: 2974
- 2020: 1909



METODI ADOTTATI DA AdC:

- EPA 537-2009 «Determination of selected perfluorinated alkyl acids in drinking water by solid phase extraction and liquid chromatography/tandem mass spectrometry (LC/MS/MS)»
- EPA 537.1-2018 «Determination of selected per and polyfluorinated alkyl acids in drinking water by solid phase extraction and liquid chromatography/tandem mass spectrometry (LC/MS/MS)»
- EPA 8327-2019 «Per and polyfluoroalkyl substances (PFAS) using external standard calibration and multiple reaction monitoring (MRM) liquid chromatography/tandem mass spectrometry (LC/MS/MS)»
- ASTM D 7968-17a «Determination of Polyfluorinated compounds in soil by liquid chromatography tandem mass spectrometry (LC/MS/MS)»

METODI ADOTTATI DA AdC:

- ASTM D 7979-19 «Determination of per and polyfluoroalkyl substances in water, sludge, influent, effluent, and wastewater by liquid chromatography tandem mass spectrometry (LC/MS/MS)»
- ISO 25101-2009 «Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) – Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry»
- UNI CEN/TS 15968-2010 «Determinazione del perfluoro ottano sulfonato (PFOS) estraibile in articoli solidi, rivestiti e impregnati, nei liquidi e nelle schiume antincendio- Metodo per il campionamento, l'estrazione e l'analisi per mezzo di LC-QMS o LC-MS»
- RAPPORTI ISTISAN 19/7 «Metodi analitici per il controllo delle acque da destinare e destinate al consumo umano ai sensi del DL.vo 31/2001 e s.m.i. – ISS.CBA.051/052.REV00»

MATRICI ANALIZZATE E RISPETTIVI LoQ:

- Acque destinate al consumo umano: < 5 ng/l
- Acque di scarico trattate + acque superficiali: PFOS < 5ng/l, altri< 10ng/l
- Acque di scarico non trattate: PFOS < 5ng/l, altri < 10ng/l
- Acque sotterranee/falda: < 5 ng/l
- Rifiuti liquidi/fanghi: PFOS < 20ng/l, altri < 50ng/l
- Rifiuti solidi/fanghi: < 0,5 µg/Kg
- Percolati discarica: PFOS < 5ng/l, altri < 10ng/l
- Prodotti chimici: 50 ng/l ÷ 0,5 µg/Kg
- Suoli/terreni: < 0,5 µg/Kg
- Eluato/test di cessione: PFOS < 5ng/l, altri < 10ng/l
- Pelli/tessuti: < 0,1 µg/Kg

CIRCUITI INTERLABORATORIO:

- 2013: n.d.
- 2014: n.d.
- 2015: 4 (acque potabili) di cui 3 z-score ok, 1 warning + JRC
- 2016: n.d.
- 2017: 2 (acque naturali), 1 z-score ok, 1 warning
- 2018: 2 (acque scarico/superficiali), z-score ok
- 2019: 11 (acque scarico/superficiali), z-score ok
- 2020: 6 (acque scarico/superficiali) + 8 (lisciviato), z-score tutti ok

ALTRI PFAAs IMPLEMENTATI:

- PFPA, PFHpS, PFNS, PFDS, PFDoS, PFTrA, PFTeA, PFPeS, P35DMHxS, P4MHpA, P5MHpA, P6MHpA, P55DMHxA, P1MHpS, P3MHpS, P4MHpS, P5MHpS, P6MHpS
- Gen x ≡ HFPO-DA
- C6O4
- 4:2 FTS, 6:2 FTS, 8:2 FTS (Fluorinated Telomer Sulfonic acid)

INTERVENTI SOSTENUTI DA ADC:

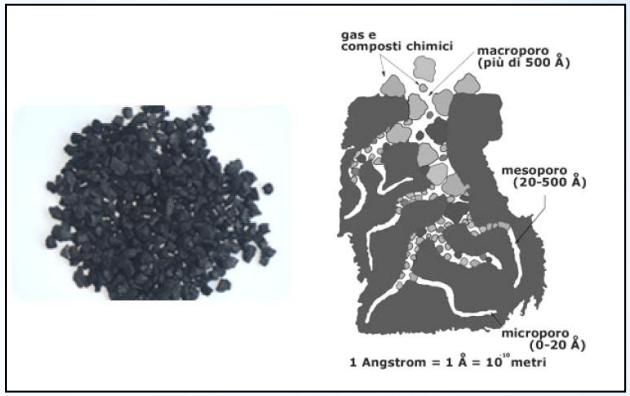
Analisi acque potabili/pozzi per cittadini comuni soci
Interventi su reti e impianti
Predisposizione punti di distribuzione acqua non contaminata (fontanelle pubbliche)
Interventi di interconnessione
Completamento sistemi idrici
Estensione rete idrica per utenti non serviti da acquedotto
Investimenti attuali e futuri

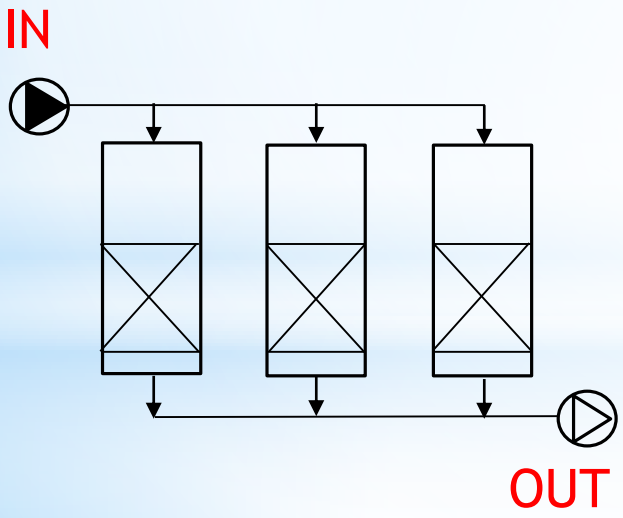
INSTALLAZIONE SISTEMI FILTRAZIONE A CARBONI ATTIVI «GAC»

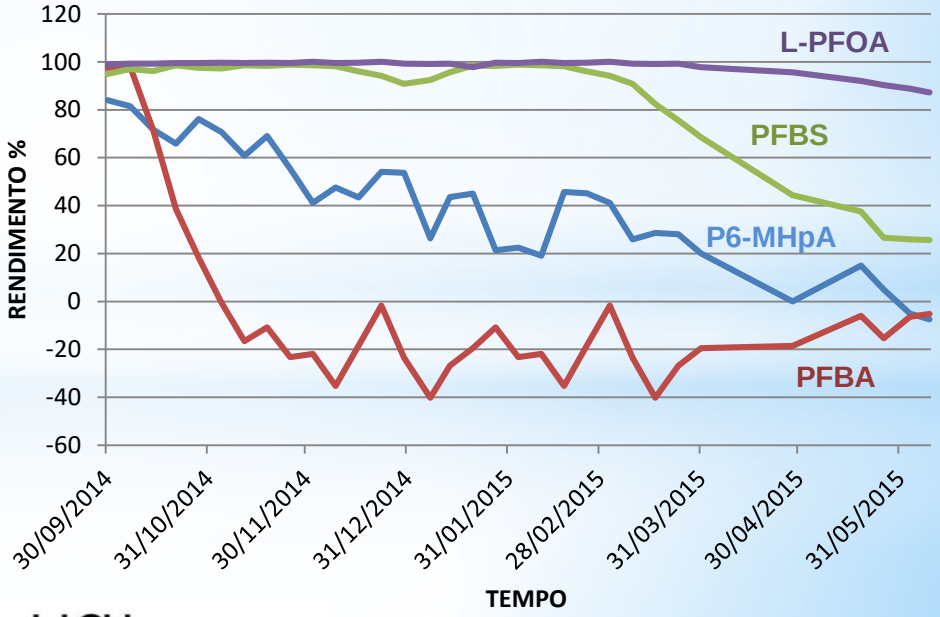
- 2013 (Agosto): Centro Idrico Madonna dei Prati-Brendola
- 2014 (Marzo): Impianto filtrazione carboni attivi Lonigo
- 2019: Centro Idrico Canove (predisposto per futura filtrazione GAC)
- 2020: Centrale di filtrazione di Montorso

ACIDI PERFLUOROALCHILICI (PFAAs):

RENDIMENTO FILTRI A CARBONE ATTIVO







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Identification and quantification of linear and branched isomers of perfluorooctanoic and perfluorooctane sulfonic acids in contaminated groundwater in the veneto region

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ABSTRACT

Perfluoroalkylated acids (PFAAs) are ubiquitous xenobiotic substances characterized by high persistency, bioaccumulation potential and toxicity. They have generated global concern because of their widespread presence both in water and biota compartments. In the past four years, alarming levels of these pollutants have been found in both surface and groundwater collected in an area covering more than 150 square kilometers in the south-western part of the province of Vicenza (Veneto region, Italy). One of the sources of the contamination recognized by local authorities is a fluorochemicals production plant that produced PFAAs since late sixties by electrochemical fluorination involving the ehtainment of a complex mixture of linear and branched isomers. Branched isomers account for a significant part of total long chain homologues (22%–35%). Because of the potential threat to public health and the absence of specific limits set for these pollutants by Directive 98/83/EC, local authorities have established the following performance limits for drinking water: 90 ng L^{-1} for PFOA+PFOS, (reduced to 40 ng L^{-1} in the most contaminated municipalities), 30 ng L^{-1} for PFOS, and 300 ng L^{-1} for the sum of all other PFAAs. Given the non-negligible incidence of branched isomers, it appears very important to correctly identify and quantify their contribution to total PFAAs. A liquid chromatography-electrospray ionization tandem spectrometry LC-MS/MS method, coupled with solid phase extraction, was developed to identify and quantify 25 PFAAs including six branched isomers of PFOS and four branched isomers of PFOA. Expanded uncertainty, recovery and precision were determined and found to agree with the reference EPA method 537.2006. The quantification limit is comprised in the 1–5 ng L^{-1} range.

1. Introduction

In the past four years, high levels of PFAAs have been detected in both surface and underground water sampled in an area covering approximately 150 square kilometers in the south-western part of the province of Vicenza (Veneto region, Italy) [1]. Because of the potential threat to public health and the absence of specific limits set for these pollutants by Directive 98/83/EC, local Authorities have established the following performance limits for drinking water: 90 ng L^{-1} for PFOA+PFOS, (reduced to 40 ng L^{-1} in the most contaminated municipalities), 30 ng L^{-1} for PFOS, and 300 ng L^{-1} for the sum of all other PFAAs.

Surveys conducted by competent authorities identified one of the sources of contamination in a fluorochemical production plant that manufactured PFAAs since late sixties by electrochemical fluorination (ECF). Further investigations on both surface and groundwater sampled in the polluted area revealed the presence of significant quantities of branched and linear isomers for both PFOA and PFOS.

The PFAA production process must be considered when seeking to explain the presence of not negligible quantities of isomers. Electrochemical fluorination and telomerization are the major manufacturing methods used to produce both long and short chain PFAAs [2]. While telomerization produces primarily straight chain PFAAs, electrochemical fluorination produces a complex mixture of linear and branched isomers.

Electrochemical fluorination is an electrochemical reaction that replaces hydrogen atoms with fluorine atoms in an organic substrate (usually a fluoride or a chloride of an organic acid) dissolved

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OBIETTIVO SU Acque sotterranee e superficiali

Sviluppo e messa a punto di una metodica di determinazione di sostanze perfluoroalchiliche

Viene presentato lo sviluppo e la messa a punto di una metodica per la determinazione di sostanze perfluoroalchiliche (PFASs), composta dalla fase di estrazione in fase solida (SPE extraction) e dalla fase di analisi mediante cromatografia liquida ad alte prestazioni con rivelatore triplo quadrupolo (UHPLC-MS/MS). Oltre agli isomeri lineari, è possibile anche determinare gli isomeri ramificati mono e di-sostituiti del PFOS e del PFOA.

a cura di Massimo Fant, Diego Cracco, Alessandro Pellizzaro
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Figura 1- Il sistema analitico UHPLC-QQQ

I PFASs (perfluoro-alkyl substances) sono composti costituiti da catene di atomi di carbonio lineari o ramificate, a lunghezza variabile, che in genere va da C4 a C14. La loro forte stabilità è dovuta al legame C-F, il più forte tra i legami covalenti, con un' energia di dissociazione pari a ~ 490 KJ mol^{-1} che li rende particolarmente resistenti all'idrolisi, alla fotolisi e alla degradazione microbica. Presentano, inoltre, una debole tensione superficiale associata a caratteristiche antifiliche (presenza di un gruppo idrofilo e di uno idrofobo) che conferisce loro una discreta tensioattività (Minoia et al., 2008). Sono prodotti ottenuti per via sintetica attraverso reazioni di fluorurazione elettrochimica, o mediante successiva degradazione, anche metabolica, di altri prodotti fluoroderivati (Russo et al., 2014).

Come categoria, i PFASs sono costituiti da migliaia di sostanze chimiche, ma gli studi disponibili si sono concentrati principalmente sugli acidi perfluoroalchisolfonici (es. acido perfluoroftansolfonico - PFOS) e gli acidi perfluoroalchilcarbossilici (es. acido perfluoroottanoico - PFOA).
L'utilizzo dei PFASs, fin dal primo dopoguerra, è stato variegato e molteplice: emulsionanti e surfactanti in prodotti per la pulizia di svariati materiali, nonché componenti inerti di fitofarmaci.
Tali composti sono stati impiegati anche per produrre contenitori ad uso alimentare per fast-food, shampoo, schiume antincendio, dentifrici, pellicole fotografiche.

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Use of strong anion exchange resins for the removal of perfluoroalkylated substances from contaminated drinking water in batch and continuous pilot plants

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Anion exchange resin
Granular activated carbon

ABSTRACT

In recent years abnormally high levels of perfluoroalkylated substances (PFAS) have been detected both in surface and underground water sampled in an area covering approximately 150 square kilometers in the Veneto region (Italy) indicating the presence of a pollution point source (fluorochemicals production plant). Adsorption on granular activated carbon is an emergency measure which is poorly effective requiring frequent replacement. This work focuses on the application of three strong anion exchange resins (Purolite® A520E, A600E and A532E) for the removal of traces of PFOA, PFOS, PFBA and PFBS (concentration of hundreds of ng L^{-1}) from drinking water. This technology is attractive for the possibility of reusing resins after an in-situ regeneration step.

A strong relationship between the hydrophobicity of the exchange functional group of the resin and its capacity in removing PFAS exists. A600E (non hydrophobic) and A520E (fairly hydrophobic) show a reduced sorption capacity compared to A532E (highly hydrophobic). While A600E and A520E can be regenerated with solvent-less dilute solutions of non-toxic NH_4Cl and NH_4OH, A532E requires concentrated solutions of methanol or ethanol and 1% NH_4Cl and for the sake of this work it was regarded as non-regenerable. The volume of regeneration effluents requiring incineration can be efficiently reduced by more than 96.5% by using reverse osmosis coupled with under-vacuum evaporation.

Transmission electron analysis on saturated resins showed that large molecular macro-aggregates of PFAS can form in the intraparticle pores of resin indicating that ion exchange is not the only mechanism involved in PFAS removal.

1. Introduction

The term "PerFluoroAlkylated Substances" or PFAS indicates a broad group of anthropogenic compounds comprising a per-fluoroalkyl backbone and a terminal functional group.

In the last six decades these substances have been incorporated into a wide range of industrial and commercial products used in more than 200 applications: surfactants (Kissa, 2001), medical applications (Krafft and Reiss, 2007), surface protecting agents (Kostov et al., 2009), fire fighting foams (Magrabi, 2002), mist suppressor (Nishiyama and Ishikawa, 1979). Because of their past and present widespread use, PFAS are ubiquitous micro pollutants found both in underground and surface water with concentration ranging from detection limits (<1 ng L^{-1}) to several tens of ng L^{-1}.

Perfluoroalkylated substances have unique physico-chemical properties such as simultaneous hydro and oleophobicity, chemical, radiation and biological stability and extremely low surface tension.

All these properties are attributable to fluorine atoms which shield carbon–carbon bonds of the molecular backbone from physical and chemical attacks. Further, fluorination stiffens alkyl chains which self assemble at interface packing close together. This characteristic structure called "molecular-brush" forms a dense layer of trifluoromethyl groups which repels both water and oil. The number of completely fluorinated carbon atoms is the key factor in determining the degree of molecular packing; the longer the

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MASTER UNIVERSITARIO DI SECONDO LIVELLO IN CARATTERIZZAZIONE E RISANAMENTO DI SITI CONTAMINATI

ELABORATO FINALE

SVILUPPO DI UN METODO UPLC-MS/MS PER LA DETERMINAZIONE DI ACIDI PERFLUOROALCHILICI: CONTROLLI E MONITORAGGIO DI UNA CONTAMINAZIONE DI ACQUE SOTTERRANEE DESTINATE AL CONSUMO UMANO

ALESSANDRO PELLIZZARO

Numero di matricola: 965336

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Università Ca'Foscari Venezia

Ca' Foscari Challenge School

A.A. 2014-2015

Acque del Chiampo s.p.a.
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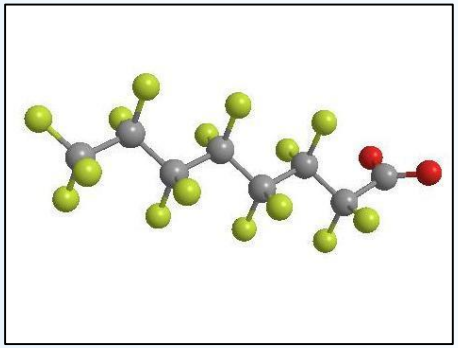
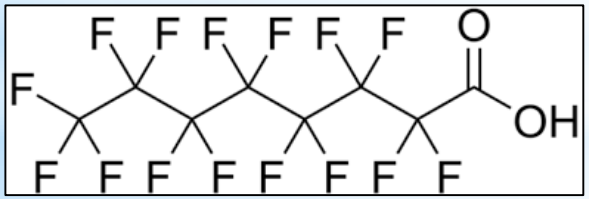
PERFLUOROALKYL ACIDS

WHAT DID WE LEARN?

PFOA

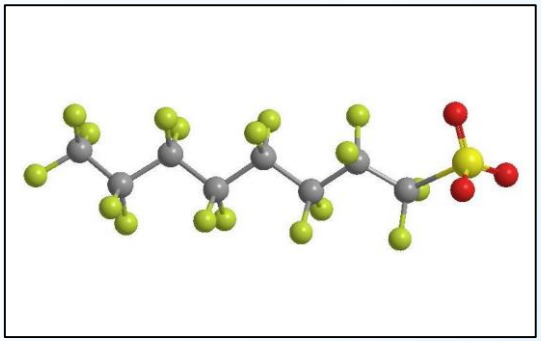
PERFLUOROOCTANOIC ACID

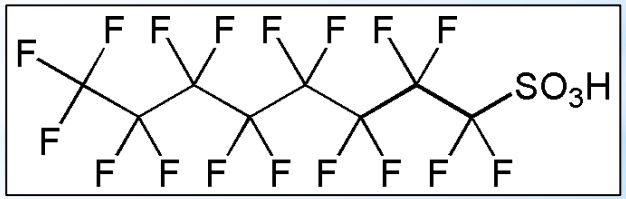




PFOS

PERFLUOROOCTANSOLFONIC ACID

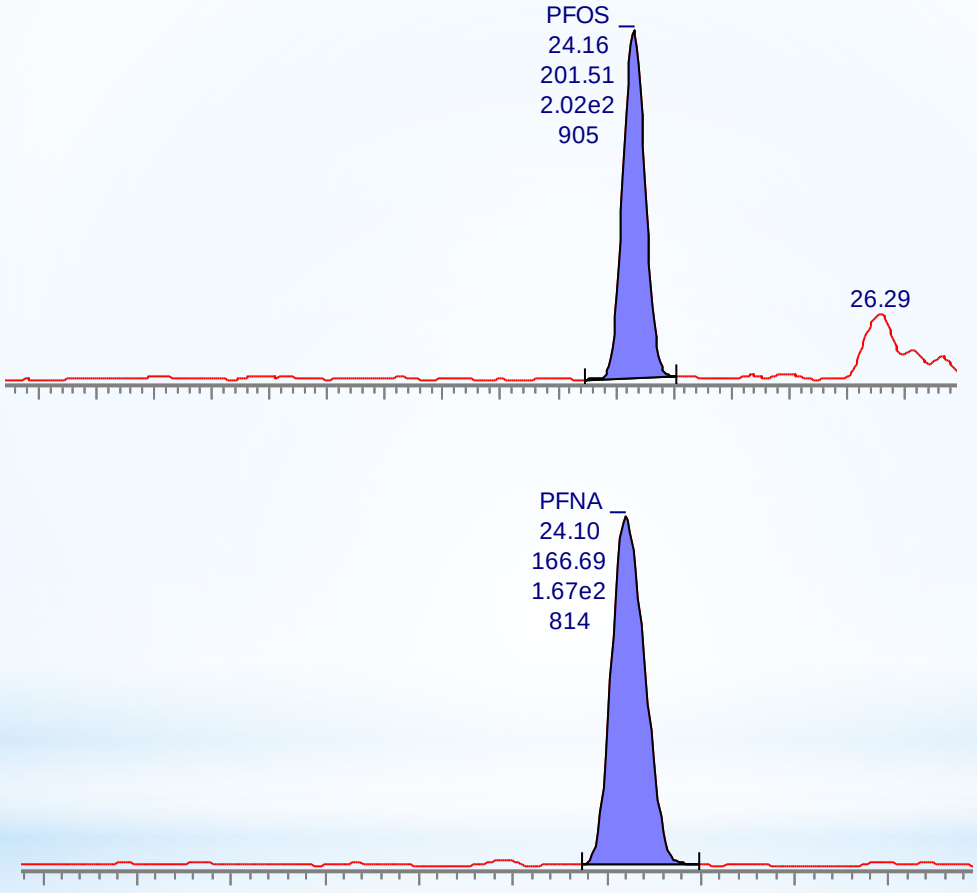




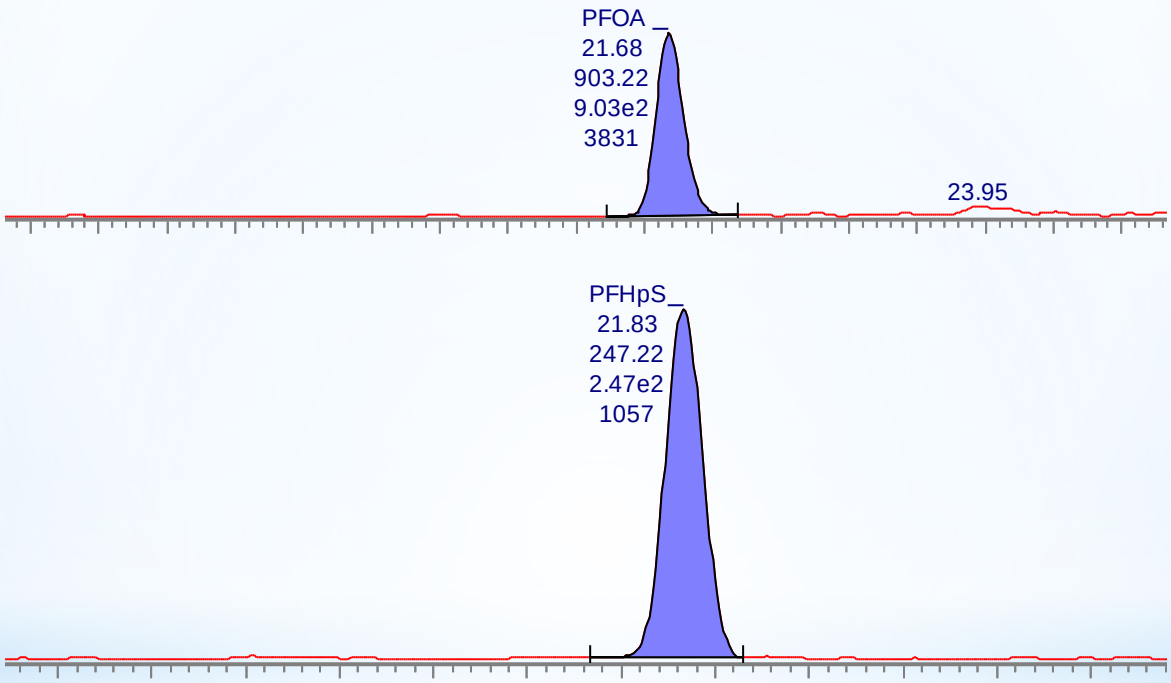
…some chemical and physical data

Abbreviation	log K_{AW}	log $K_{OW,\,dry}$	log S_W (mol L^{-1})
Perfluorinated carboxylate acids (PFCAs)			
PFBA	–3.23	2.82	0.42
PFPeA	–2.90	3.43	–0.37
PFHxA	–2.58	4.06	–1.16
PFHpA	–2.25	4.67	–1.94
PFOA	–1.93 (–3.0)	5.30	–2.73
PFNA	–1.58	5.92	–3.55
PFDA	–1.27	6.50	–4.31
PFUnDA	–0.92	7.15	–5.13
PFDoDA	–0.58	7.77	–5.94
PFTrDA	–0.38	8.25	–6.59
PFTeDA	0.03	8.90	–7.42
Perfluorinated sulfonic acids (PFSAs)			
PFBS	–2.59	3.90	–1.00
PFHxS	–2.38	5.17	–2.24
PFOS	–1.65	6.43	–3.92

…chromatographic retention time…



…chromatographic retention time…



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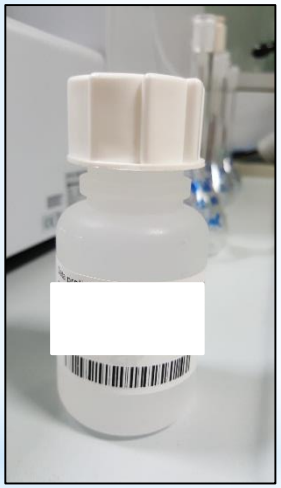
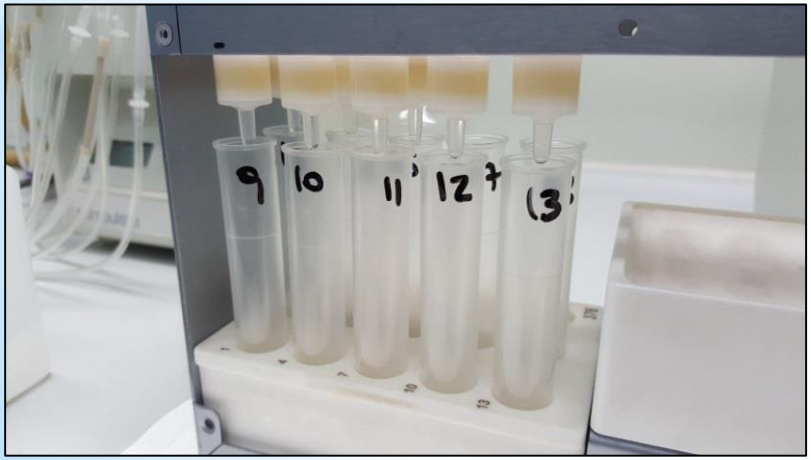
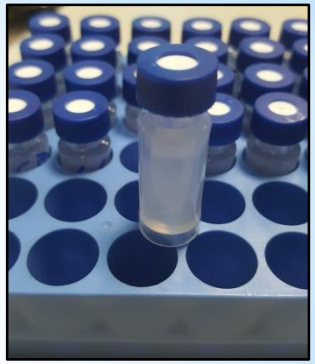
Solid Phase Extraction (SPE):







T_R – RECOVERY - PRECISION

Analyte	Retention time	RECOVERY	RSD%	INTERVAL CONCENTRATION
		n = 4 - 7		
PFBA	5,7	95 – 101%	4 - 10%	200 - 50000
PFBS	11	85 – 122 %	3 - 7%	200 - 50000
PFPeA	10,2	97 – 115%	3 - 8%	200 - 50000
PFHxA	15	99 – 128 %	2 - 7%	200 - 50000
PFHpA	18,8	97 – 127 %	4 - 14%	200 - 50000
PFHxS	19	92 – 129 %	2 - 13%	200 - 50000
PFOA	21,7	91 – 102 %	2 - 14%	200 - 50000
PFNA	24,1	84 – 106 %	7 - 19%	200 - 50000
PFOS	24,2	75 – 123 %	6 - 20%	200 - 50000
PFDA	26,1	76 – 125 %	10 - 20%	200 - 2000
PFUnA	27,8	76 – 129 %	3 - 14%	200 - 2000
PFDoA	29,3	97 – 121 %	5 - 13%	200 - 2000

SURROGATES MIX

$^{13}C_4$-PFBA
$^{13}C_4$-PFOA
$^{13}C_4$-PFOS
$^{13}C_3$-PFHxS
$^{13}C_2$-PFDA

INTERNAL STANDARD MIX

$^{13}C_2$-PFHxA
$^{13}C_3$-PFBA
$^{13}C_8$-PFOA
$^{13}C_8$-PFOS
$^{13}C_3$-PFBS
$^{13}C_2$-PFDoA

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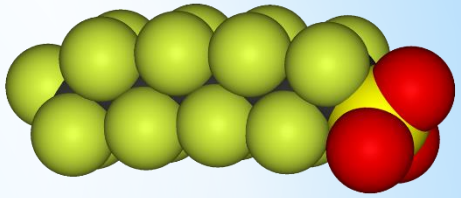
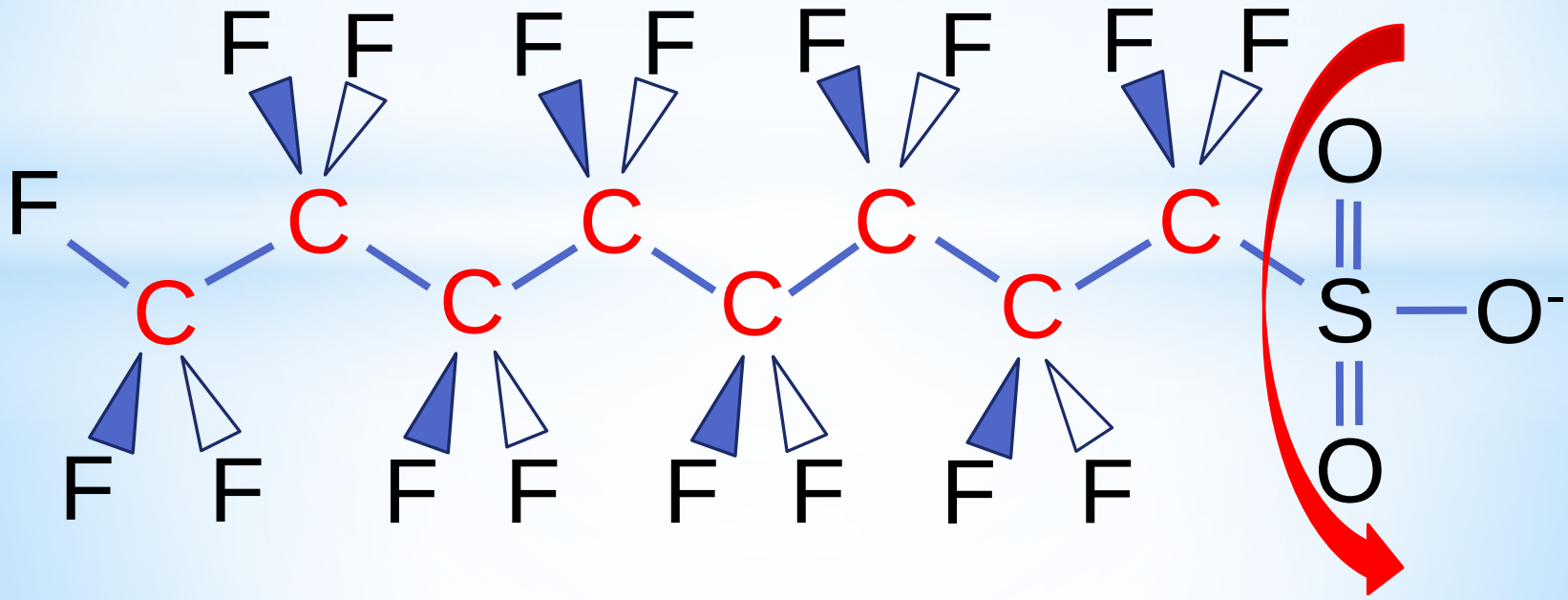
L-PFOS: 499 > 80 (SO3)

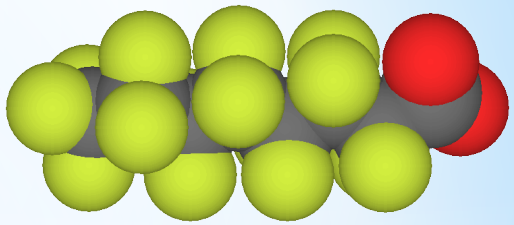
Voltaggi: cono = 60V; cc = 39V

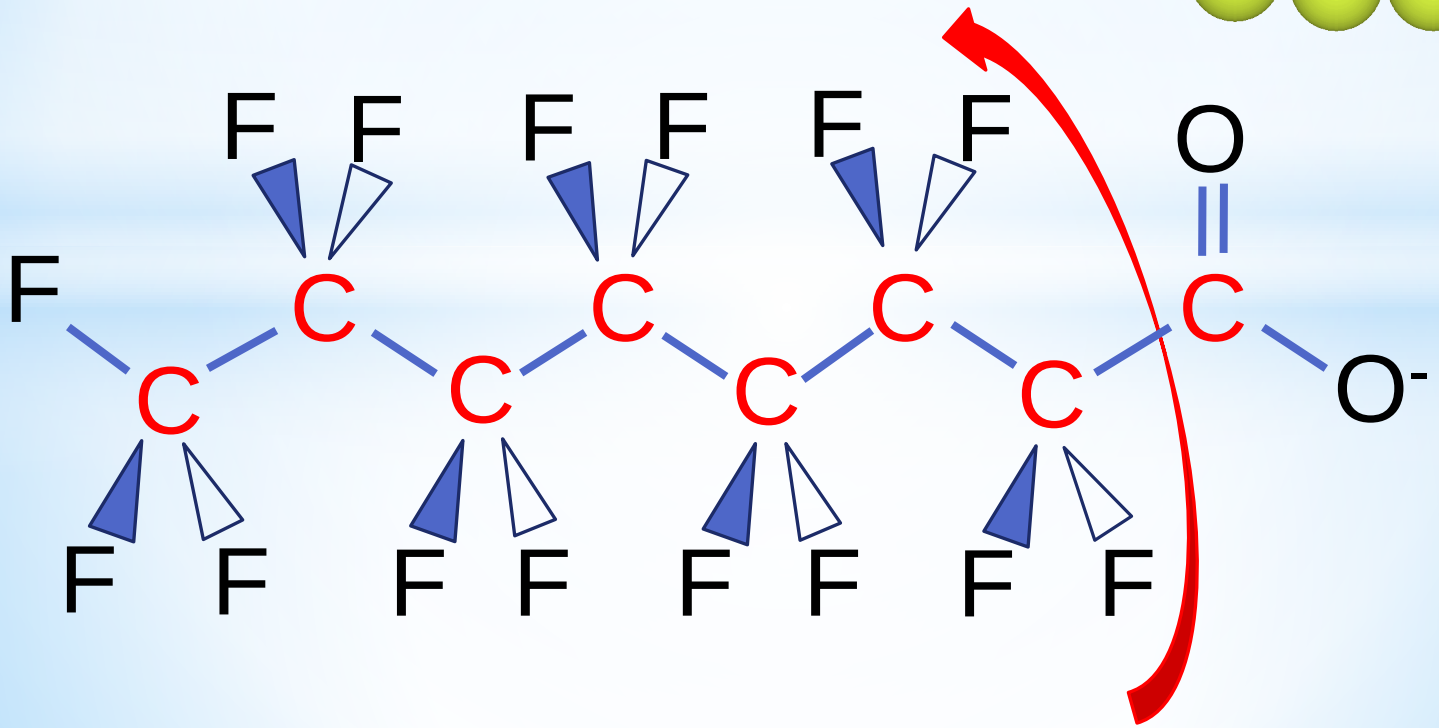




L-PFOA: 413 > 369

Voltaggi: cono = 16V; cc = 10V





Acque del Chiampo s.p.a.
Servizio Idrico Integrato

PERFLUOROALKYL ACIDS

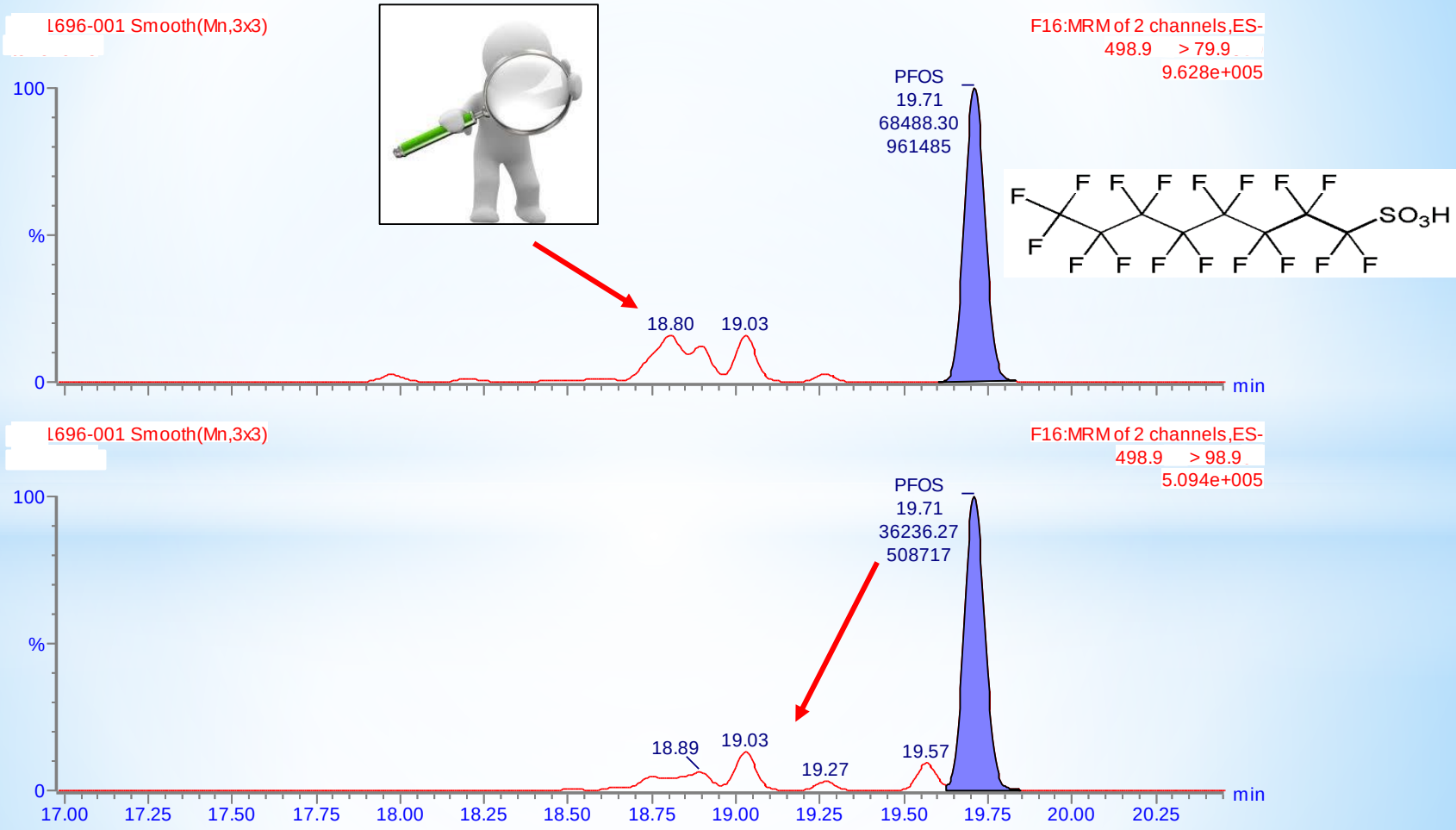
WHAT
CRITICAL ISSUES
?

Acque del Chiampo s.p.a.
Servizio Idrico Integrato

TRANSITIONS AND VOLTAGES

MS file			
Analyte	Transition	Cone voltage (V)	Collision cell voltage (V)
PFBA	213 > 169	18	10
PFPeA	263 > 219	15	9
PFOS	499 > 80	60	39
	499 > 99	60	39
PFBS	299 > 80	54	30
	299 > 99	54	28
PFDA	513 > 219	20	16
	513 > 269	20	16
PFNA	163 > 219	20	18
	163 > 169	20	16
PFHpA	363 > 119	16	20
	363 > 169	16	16
PFHxA	313 > 119	16	17
	313 > 269	16	10
PFUnA	563 > 219	20	20
	563 > 269	20	18
PFOA	413 > 369	16	16
	413 > 169	16	10
PFHxS	399 > 80	40	40
	399 > 99	40	40
PFDoA	613 > 169	25	15
	613 > 319	25	15
	613 > 569	25	15

PFOS ISOMERS: CHROMATOGRAFIC PROFILE



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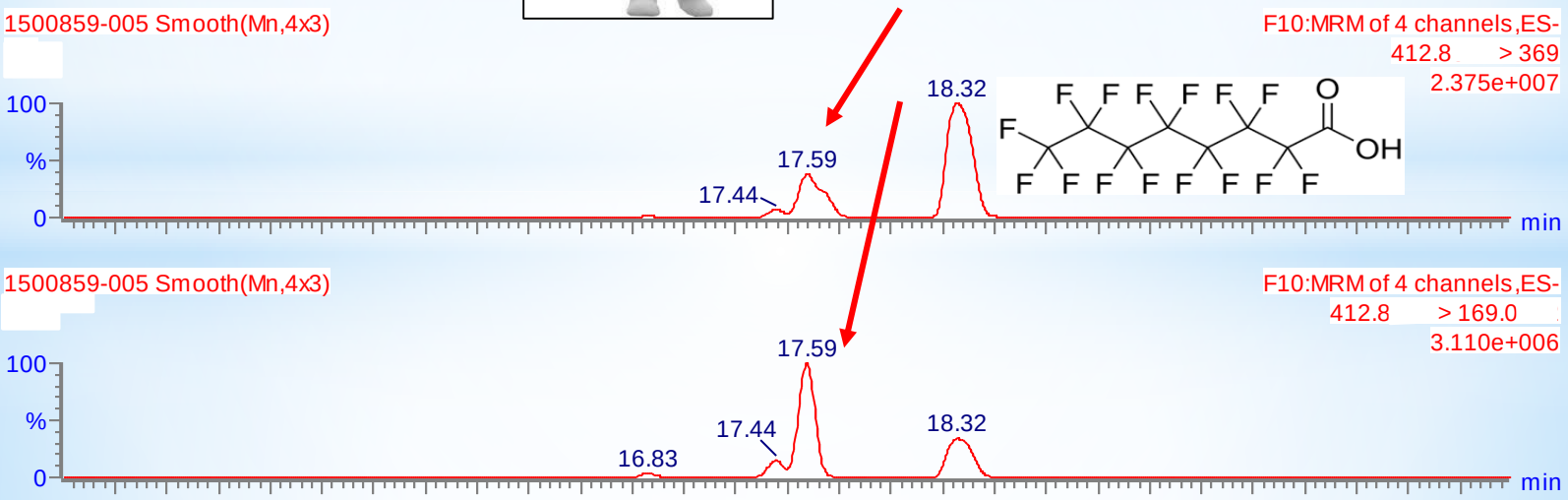
PFOA ISOMERS: CHROMATOGRAFIC PROFILE



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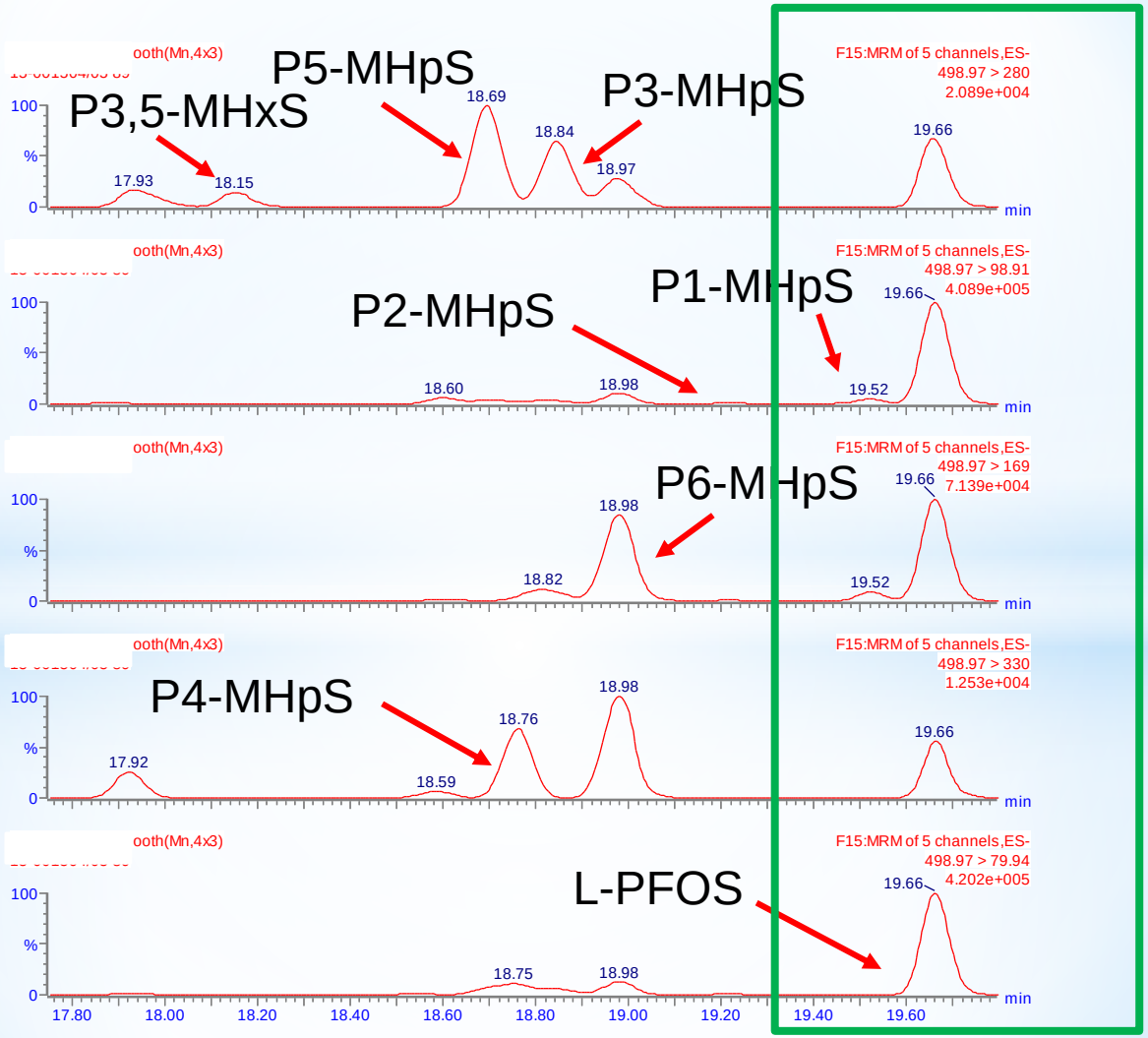
MRM TRANSITIONS: QUALIFICATION AND QUANTIFICATION

PFOA ISOMERS	PFOS ISOMERS
413 > 369	499 > 80
413 > 169	499 > 99
+	+
413 > 119	499 > 169
413 > 219	499 > 330
	499 > 280

PFOS TRANSITIONS



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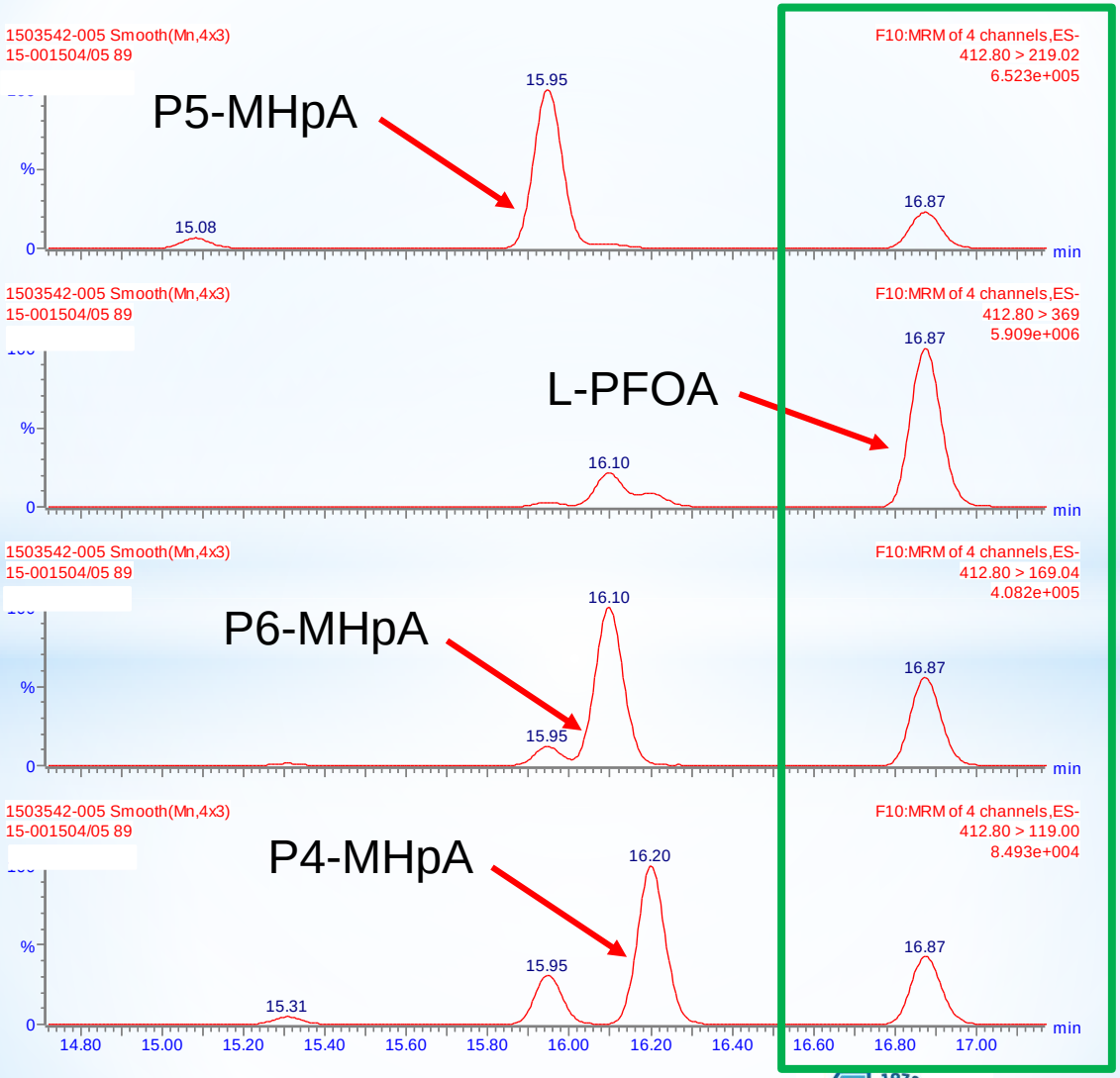
1978-2018
40 ANNI DI DEPURAZIONE

PFOA TRANSITIONS



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1978-2018
40 ANNI DI DEPURAZIONE

PFAAs

AQUEOUS SAMPLE 100%

SOLUTION?

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AMPHIPHILIC MOLECULES

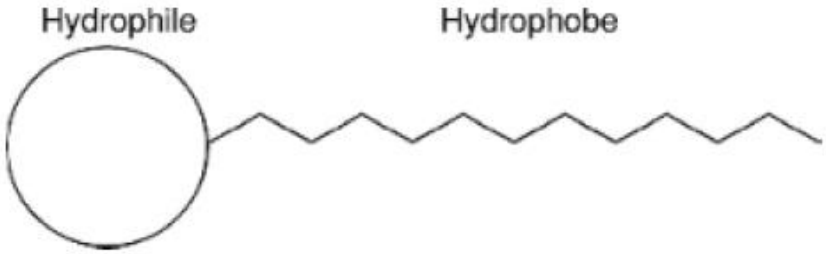
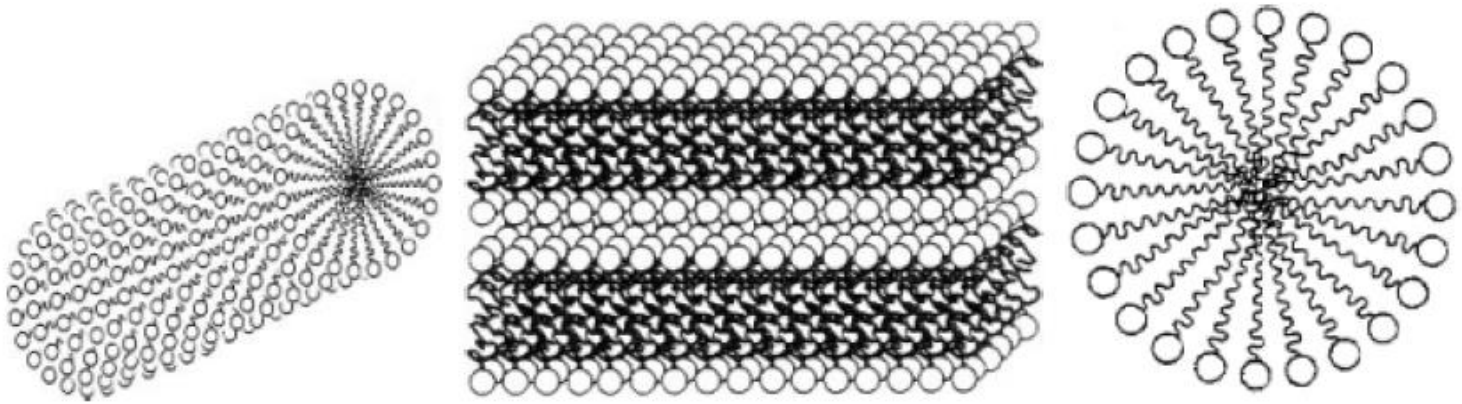


Figure 1.1 Simplified surfactant structure.



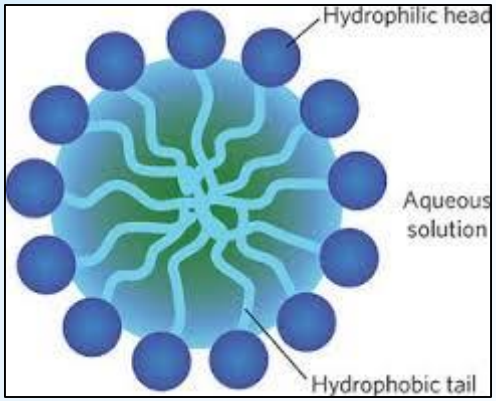
(a) Cylindrical (b) Lamellar (c) Spherical

Figure 1.2 Typical micelle configurations.

PFAAs → AMPHIPHILIC MOLECULES

↓

AGGREGATES



CONCENTRATION

PERFLUOROALKYL CHAIN LENGHT

TEMPERATURE

Na^+ CONCENTRATION (ANIONIC T.)

TURBIDITY

SURFACE ACTIVE AGENTS

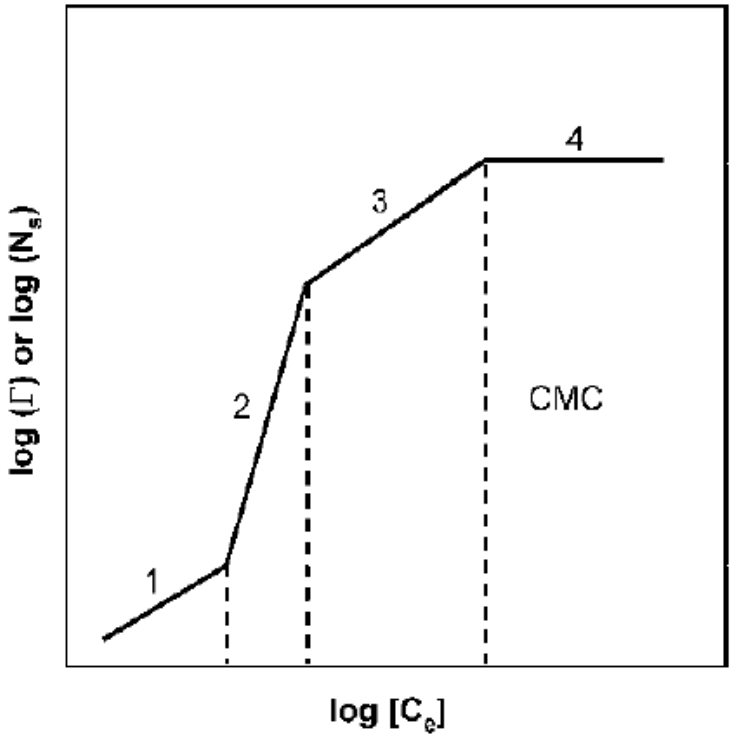


Figure 2.4 Four region isotherms of surfactant adsorption.

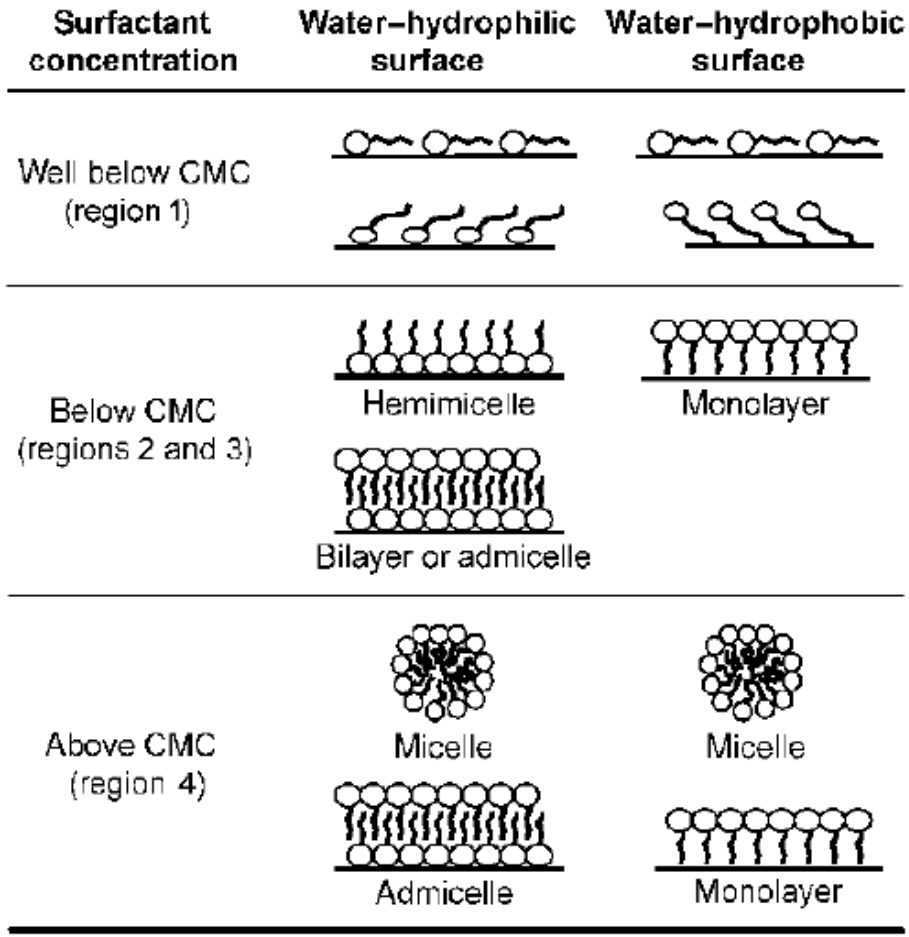


Figure 2.5 Surfactant adsorption on solid surfaces.

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Use of strong anion exchange resins for the removal of perfluoroalkylated substances from contaminated drinking water in batch and continuous pilot plants

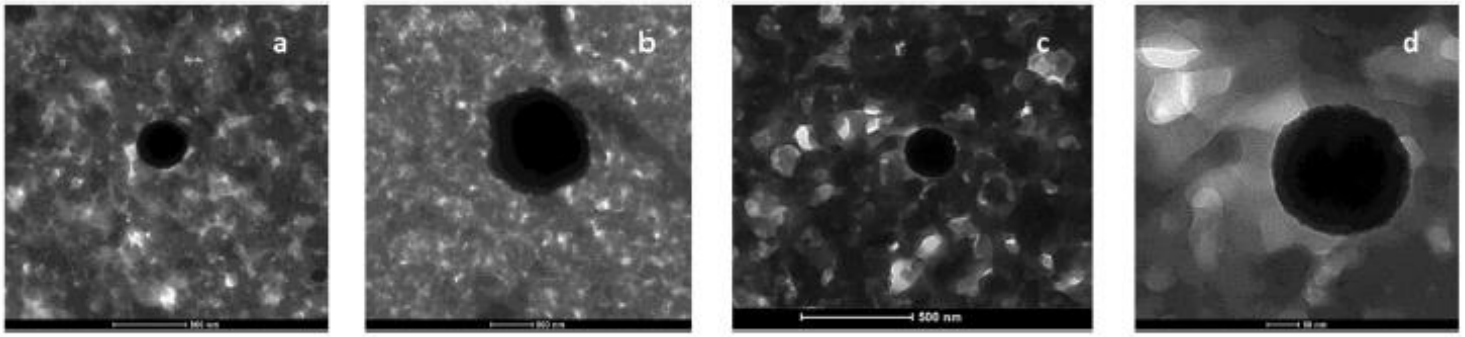

Fig. 2. Different TEM images on microtomized sections of Purolite® A520E saturated with PFOA. a), b) and c) show three different aggregates at the same magnification (the bar corresponds to 500 nm); d) shows aggregate c) at an additional magnification of 10× (bar corresponds to 50 nm).

PFAAs

↓

AMPHIPHILIC MOLECULES

↓

AGGREGATES - ADSORPTION

↕

TURBIDITY – SOLIDS IN SUSPENSION
ADHESION

REPRESENTATIVE SAMPLE VOLUME

(ng/L!!)

PFAAs

SAMPLING?

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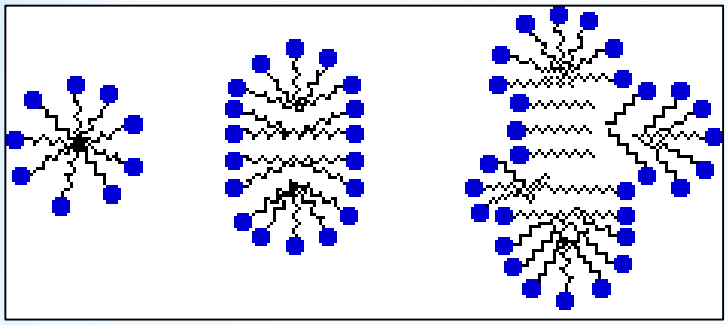
CONSERVATION – PFAAs SOLUTION STABILITY



PFOS
PFNA
PFDA
PFUnA
PFDoA

con Met-OH ≥ 30%
conservazione T ≤ 6°C

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PFAAs

SPE

RECOVERY

ANALYTICAL TIME

CLEAN-UP

QL

DIRECT INJECTION

SOLUTION?

MATRIX EFFECT

SOLIDS

VISCOSITY
DENSITY

Environmental Science & Technology

Feature

pubs.acs.org/est

A Never-Ending Story of Per- and Polyfluoroalkyl Substances (PFASs)?

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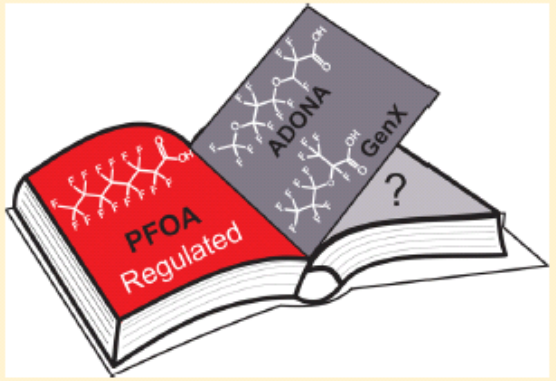
ABSTRACT: More than 3000 per- and polyfluoroalkyl substances (PFASs) are, or have been, on the global market, yet most research and regulation continues to focus on a limited selection of rather well-known long-chain PFASs, particularly perfluorooctanesulfonate (PFOS), perfluorooctanoic acid (PFOA) and their precursors. Continuing to overlook the vast majority of other PFASs is a major concern for society. We provide recommendations for how to proceed with research and cooperation to tackle the vast number of PFASs on the market and in the environment.



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