

### 5<sup>th</sup> International Congress

## Gestion des Risques Environnementaux & Sanitaires Management of Environmental & Health Risks

17, 18, 19 & 20/06/2025 - Paris

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Formation : Colloque reconnu par le SPW ARNE

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#### Mardi 17 juin 2025

08h45 Accueil des participants

#### **Présidents :**

• Laurence Lanoy, Avocate en Droit de l'Environnement - Laurence Lanoy Avocats, France

• Dr. Frank Karg, SFSE & ARET, Expert judiciaire, Directeur scientifique - HPC International, France & Allemagne

#### Contexte règlementaire international

#### 09h15

Discours de bienvenue & introduction au congrès Dr. Frank Karg, Organisation et Président du Congrès International PFAS

#### 09h30

Les PFAS et la politique de la santé publique Dr. Cyrille Isaac-Sibille, Secrétaire de la commission des Affaires Sociales, Député, Ouest et Sud de la Métropole de Lyon

09h45 Réglementation Internationale des PFAS : Enjeux et Contentieux Joanna Peltzman, Avocat - Osborne Clarke, France

#### 10h15

Les évolutions récentes du cadre juridique concernant les substances PFAS Laurence Lanoy, Avocate en Droit de l'Environnement - Laurence Lanoy Avocats, France

10h15 Pause-café & thé

#### 11h15 Focus : La crise des PFAS en Belgique : une leçon ?

#### Introduction :

Genèse, crise, réglementation : vers une saine gestion, dans un cadre réglementaire sécurisé ? Bernard Deltour, Avocat au Barreau de Bruxelles - Industrious Law, Belgique

#### 11h25

La crise. Impacts opérationnels : les défis pour l'économie circulaire Présentation/modération : *Aarnout Ecker, Directeur général - DENUO, Belgique* Témoignages :

- Andy Heurckmans, Directeur Grondbank, Belgique
- Isabelle Laurent, Directrice générale Walterre, Belgique
- Wouter Vermin, Directeur Traitement et Valorisation Bioterra, Belgique
- Andres Van Brecht, Responsable de la Législation sur les Déchets Groupe Indaver, Belgique
- Dieter Grimmelprez, COO & Chef de la Stratégie R&D Groupe Vanheede Environment, Belgique

#### 12h05

La réglementation : vers un cadre normatif clarifié Témoignages :

- La Région Flamande : Werner Annaert, Administrateur général OVAM, Belgique
- La Région Wallonne : Dr. Philippe Raucq, Coordinateur PFAS pour la Wallonie, Belgique

• La Région de Bruxelles-Capitale : *Saïd El Fadili, Directeur Département Sols - Bruxelles Environnement, Belgique* 

12h45 Déjeuner

#### Identification & Caractérisation des sources des PFAS

#### 14h00

Avancements dans l'identification et différentiation des sources de pollutions par les PFAS via la MVA sur base de l'IA : Intelligence Artificielle pour clarification des responsabilités Dr. Frank Karg, SFSE & ARET, Expert judiciaire, Directeur scientifique - HPC International, France & Allemagne

#### 14h30

Comment prolonger la durée de vie utile des CAG (Charbon Actif en Grains) afin de réduire les coûts d'exploitation pour l'élimination des PFAS et autres contaminants émergents : le projet LIFE Cascade *Cristian Carboni, Directeur du Développement Commercial - De Nora Water Technologies, Italie* 

#### 15h00

Apport de la chromatographie liquide couplée à la spectrométrie de masse à haute résolution pour l'identification de PFAS

Naïke Noyon, Responsable Laboratoire - Suez Groupe, France

15h30 Pause-café & thé

#### 16h00

Forensie Environnementale : Déploiement d'une approche interdisciplinaire pour l'identification et la répartition des sources de PFAS dans le cadre d'un litige environnemental

- Olivier Sibourg, Principal Ramboll, France
- Eric S. Wood, Principal Ramboll, USA

#### 16h30

Déploiement du système FRED-PFAS : Unité portable de mesure des PFAS sur le terrain Melanie McClare, Chef du service commercial - FREDsense Technologies Corp., Calgary, Canada

#### 17h00

Évaluation de la contamination par les PFAS dans les eaux résiduaires : approches innovantes via l'échantillonnage passif et oxydation TOP *Lucas Géraud, Doctorant - Ecométrique/Laboratoire E2LIM-Université de Limoges, France* 

#### 17h30

Gestion de la pollution des eaux souterraines par les PFAS en combinant l'hydrologie et le traitement de l'eau au charbon actif *Eric Chauveheid, Responsable Qualité de l'Eau - Vivaqua, Belgique* 

#### 18h00

Fin de la première journée



#### Tuesday, June 17, 2025

08:45 Welcoming participants

#### **Presidents:**

- Laurence Lanoy, Lawyer in Environmental Law Laurence Lanoy Avocats, France
- Dr. Frank Karg, SFSE & ARET, Court Expert, Scientific Director HPC International, France & Germany

#### **International Legal Context**

#### 09:15

Welcome speech & introduction to the congress Dr. Frank Karg, Organization & President of International PFAS Congress

#### 09:30

PFAS and public health politics Dr. Cyrille Isaac-Sibille, Secretary of the Social Affairs Committee, Member of Parliament, West and South of Lyon Métropole

#### 09:45 International regulation of PFAS: Issues and Litigation Joanna Peltzman, Lawyer - Osborne Clarke, France

#### 10:15

Recent developments in the legal framework for PFAS substances Laurence Lanoy, Attorney in Environmental Law - Laurence Lanoy Avocats, France

10:15 Coffee & Tea break

#### 11:15 Focus: The PFAS crisis in Belgium: a lesson?

#### Introduction:

Genesis, crisis, regulation: towards sound management within a secure regulatory framework? *Bernard Deltour, Lawyer at the Brussels Bar - Industrious Law, Belgium* 

#### 11:25

The crisis. Operational impacts - Challenges for the circular economy Presentation/moderation: *Aarnout Ecker, General Manager - DENUO, Belgium* Testimonies:

- Andy Heurckmans, Director Grondbank, Belgium
- Isabelle Laurent, General Manager Walterre, Belgium
- Wouter Vermin, Director Treatment and Valorisation Bioterra, Belgium
- Andres Van Brecht, Head of Waste Legislation Indaver Group, Belgium
- Dieter Grimmelprez, COO & Head of R&D Strategy Vanheede Environment Group, Belgium

#### 12:05

Regulations. Towards a clearer regulatory framework Testimonies:

- Flemish Region: Werner Annaert, General Administrator OVAM, Belgium
- Walloon Region: Dr. Philippe Raucq, PFAS coordinator for Wallonia, Belgium

• Brussels-Capital Region: Saïd El Fadili, Director of the Soils Department - Brussels Environment, Belgium

12:45

Lunch

#### **Identification & Characterization of PFAS Sources**

#### 14:00

Advancements of PFAS Source Identification & Differentiation based on MVA and AI : Artificial Intelligence Clustering for Responsibility Clearing Dr. Frank Karg, SFSE & ARET, Court Expert, Scientific Director - HPC International, France & Germany

#### 14:30

How to extend the useful life of GACs (Granular Activated Carbon) to reduce opex for the removal of PFAS and other emerging contaminants: the LIFE Cascade project *Cristian Carboni, Business Development Manager - De Nora Water Technologies, Italy* 

#### 15:00

Contribution of liquid chromatography coupled to high-resolution mass spectrometry for the identification of PFAS *Naïke Noyon, Laboratory Manager - Suez Groupe, France* 

15:30 Coffee & Tea break

#### 16:00

Environmental Forensics: Deploying an interdisciplinary approach to identify and apportion PFAS sources in the context of environmental litigation
Olivier Sibourg, Principal - Ramboll, France

• Eric S. Wood, Principal - Ramboll, USA

#### 16:30

Field Deployment of FRED-PFAS: a portable unit for on-site measuring total PFAS Melanie McClare, Chief Commercial Officer - FREDsense Technologies Corp., Calgary, Canada

#### 17:00

Assessment of PFAS contamination in wastewater: innovative approaches using passive sampling and TOP oxidation Lucas Géraud, PhD Student - Ecométrique / E2LIM Laboratory-University of Limoges, France

#### 17:30

Managing groundwater PFAS pollution by combining hydrology and activated carbon water treatment Eric Chauveheid, Water Quality Manager - Vivaqua, Belgium

18:00 End of the first day

#### International Regulation of PFAS: Issues and Litigation

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#### Introduction

Per- and polyfluoroalkyl substances (PFAS), known as "forever chemicals," are synthetic compounds designed to resist degradation. This durability has made them useful across a wide range of industries—from non-stick cookware and water-repellent textiles to fire-fighting foams and food packaging. However, these same properties have led to unprecedented global contamination. PFAS are persistent in the environment, bioaccumulate in the human body, and are linked to numerous adverse health outcomes including cancers, immune dysfunction, and developmental toxicity.

Despite growing scientific consensus and alarming exposure data, the international legal response remains heterogeneous and often insufficient. Regulatory systems struggle to keep pace with the diversity of PFAS molecules (more than 10,000 identified), and litigation is often the only means of obtaining redress. This note provides an overview of the current regulatory landscape and emerging legal battles surrounding PFAS, arguing that only a coherent and precautionary approach—both legislative and judicial—can provide adequate protection.

#### 1. A Growing Patchwork of Regulations

At the international level, the 2001 Stockholm Convention on Persistent Organic Pollutants (POPs) laid the groundwork by restricting the use and production of certain PFAS (notably PFOS and PFOA). The European Union has gone further, integrating PFAS into key regulations such as REACH and the Drinking Water Directive. In 2024, the EU adopted the Packaging and Packaging Waste Regulation (PPWR), introducing specific PFAS thresholds for food-contact packaging materials. A broader ban on over 10,000 PFAS substances is now being considered under REACH, following a joint proposal by five Member States.

On the national level, countries have adopted various measures:

- Denmark banned PFAS in food packaging as early as 2020 and prohibited PFAS in firefighting foams from 2024. A public database allows consumers to identify PFAS-containing products.
- The Netherlands introduced a zero-transition ban on PFAS in food packaging in 2022.
- The United States has adopted a sectoral approach, with the EPA requiring industry reporting through the Toxic Release Inventory and several states (e.g., California, Maine, Washington) imposing PFAS bans in specific products.
- New Zealand and Sweden are phasing out PFAS in cosmetics and firefighting foams, while encouraging the development of alternatives through tools like Sweden's PRIO database.

#### 2. PFAS Litigation: From the U.S. to Europe

Litigation has emerged as a critical lever for enforcing accountability and exposing regulatory gaps. As regulatory action lags, affected communities and local governments have increasingly turned to the courts.

- In the U.S., numerous lawsuits have been filed by municipalities and individuals against major chemical companies like 3M and DuPont. These cases have resulted in billion-dollar settlements and a landmark decision by 3M to phase out PFAS production by 2025. Bipartisan support in Congress has led to the creation of a PFAS Task Force to develop a national legislative response.
- In France, local authorities issued a prefectural order against chemical giant Arkema for PFAS pollution in the Rhône Valley the country's most contaminated region.
- A 2023 Swedish court ruling recognized the right of citizens exposed to PFAS to be compensated for physical harm, setting a legal precedent that could influence future EU litigation.

At the same time, policy discussions around the creation of compensation funds and the application of the polluter-pays principle signal a growing legal consciousness of the societal costs linked to PFAS contamination.

#### 3. Regulatory Challenges

The complexity of PFAS regulation stems not only from scientific uncertainties but also from structural legal and economic barriers:

- Chemical diversity: With over 10,000 known PFAS compounds, regulating them individually is both scientifically and administratively unfeasible.
- Regrettable substitution: Efforts to phase out substances like PFOA have often led to replacement with similarly harmful alternatives like GenX.
- Trade and regulatory asymmetry: Products containing PFAS can be imported from countries with weaker regulations, undermining local bans.
- Slow institutional response: Even within the EU, regulatory reform can take years leaving populations exposed in the interim.

These challenges demand a shift toward class-based regulation, guided by the precautionary and prevention principles foundational to environmental law.

When comparing the regulatory frameworks for asbestos and PFAS, a difference in maturity is striking. Asbestos regulation, now largely harmonised at international level, is the result of decades of scientific, trade union and legal action that led to its ban in many countries, based on the precautionary principle and recognition of its definite carcinogenic effect. In contrast, PFAS regulation is still in its infancy. Despite comparable health effects in terms of severity – cancers, immune disorders, developmental effects – and widespread exposure of the population, PFAS are not yet subject to a global ban. Their regulation remains fragmented, depending on their use (food, cosmetics, industry).

#### Conclusion

PFAS regulation and litigation are two sides of the same coin: both aim to control the uncontrolled and bring justice to the exposed. While some jurisdictions have pioneered meaningful restrictions, others lag dangerously behind. Litigation is filling this void by forcing transparency, securing compensation, and catalyzing regulatory reform.

What is urgently needed is a coordinated, global exit strategy from non-essential PFAS use. This means stronger regulation, better enforcement, and legal mechanisms that ensure polluters – not the public – bear the cost of contamination.





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#### Les évolutions récentes du cadre juridique concernant les substances PFAS

Les substances per- et polyfluoroalkylées (PFAS) appartiennent à une grande famille de perturbateurs endocriniens utilisés dans de nombreux processus industriels et la fabrication d'objets de la vie quotidienne. En effet, par leurs propriétés antiadhésives, résistantes aux fortes chaleurs et imperméabilisantes, ces substances uniques constituent des polluants persistants et nocifs pour l'environnement et la santé humaine puisqu'elles se retrouvent dans les eaux potables, souterraines ou de surface, les sols, l'air ou encore l'alimentation.

La prise de conscience relative à ces substances et leurs effets est à la fois récente et majeure : des mesures de surveillance et de limitation des PFAS dans l'environnement commencent à s'imposer afin de restreindre l'usage de ces composés et de réduire au maximum l'exposition et les risques pour la santé humaine.

La règlementation des PFAS évolue plus ou moins rapidement selon les secteurs.

Elle se démarque en matière d'eau, puisque dans le contexte de la transposition en droit français de la directive européenne du 16 décembre 2020 dite « eau potable », qui renforce leur suivi et fixe des valeurs limites dans l'eau destinées à la consommation humaine pour une vingtaine de ces substances, le Gouvernement a publié un plan d'action interministériel contre les PFAS. À travers les différents axes d'action qu'il déploie, ce document à un impact sur les obligations des exploitants des ICPE qui sont concernés par ces problématiques avec le récent arrêté du 31 octobre 2024 qui met en place l'analyse des PFAS dans les émissions atmosphériques des installations d'incinération et co-incinération pour les ICPE déchets sous les rubriques 2770, 2771, 2971 et 3520, selon un calendrier précis entre 2025 à 2028.

Enfin la proposition de loi française sur les PFAS a été adoptée en seconde lecture par la Commission du développement durable le 12 février 2025. Elle prévoit l'interdiction, dès janvier 2026, de certains produits contenant des PFAS, comme les cosmétiques et vêtements, avec quelques exemptions.

Enfin l'ECHA et cinq États membres ont publié un état d'avancement sur la restriction des PFAS le 20 novembre 2024, après la demande d'interdiction de janvier 2023. Des discussions sont en cours, avec des options de restriction, notamment pour les batteries, afin de limiter les impacts socio-économiques.

Laurence Lanoy, avocat spécialiste en droit de l'environnement, effectuera un tour d'horizon des évolutions du cadre réglementaire concernant ces substances préoccupantes, en présentant les enjeux relatifs à leur gestion pour le secteur industriel.

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Avocat depuis 1990 et Docteur en droit, Laurence Lanoy a développé une pratique approfondie en droit de l'environnement avant de fonder en 2005 le cabinet Laurence Lanoy Avocats. Elle conseille et assiste des entreprises nationales et internationales, des collectivités publiques et des cabinets d'avocats internationaux notamment en droit de l'environnement et du développement durable, en droit minier et en droit de l'énergie.





#### Avancements dans l'identification et différentiation des sources de pollutions par les PFAS via la MVA sur base de l'IA : Intelligence Artificielle pour clarification des responsabilités /

#### Advancements of PFAS Source Identification & Differentiation based on MVA and AI : Artificial Intelligence Clustering for Responsibility Clearing

#### Frank KARG

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#### 1. <u>Introduction</u>

Concerning PFAS contamination, mostly all Groundwater Contaminations Plumes are showing multi-contamination Sources. The principal question is which PFAS Contamination Source is responsible for which part of the complete PFAS contamination. The following Article is showing the approach of MVA-AI to solve this question for limiting responsibilities only on the real pollution part, for the Industry, different other polluters, the Insurance companies and in case of Court Disputes, to share correctly and in Transparency the financial burdens.

Since the 1960s, PFAS monomers (Per- & Polyfluoro-Alkyl Substances) have gradually become a major environmental and public health problem in industrialized countries, due to their multiple and wide-ranging applications (historical and still current). This threat to the environment and to public health has been gradually coming to the fore since the 2010s and will be considered to a greater extent in 2022 - 2024. As a result, PFAS are now found in the soil at polluted sites, in groundwater, in food, drinking water, in soil gas and in ambient air (volatile PFAS, e.g. FTOH: Fluorotelomer alcohols, etc.). Between 9,000 and 12,000 synthetic PFAS pollutants have been produced, with some publications indicating as many as 15,000 molecules.

Polymeric PFASs such as Teflon (or PFTE etc.) are not very bioavailable and are therefore much less toxic than monomeric PFASs. These monomeric PFAS are the subject of the work presented below. PFASs are known for their toxicological effects as endocrine disruptors, hepatotoxicity, immunotoxicity, effects on fetal development and, in some cases, carcinogenicity (e.g. PFOA).

An important characteristic of PFASs is their behavior in Environmental Chemistry, since only polyfluorinated PFASs are modified by microbiological bio-transformation into perfluorinated PFASs, which remain totally stable and non-degradable, and even bio-accumulable.





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Particularly in the case of groundwater pollution by PFAS, it is becoming increasingly important to identify and, above all, differentiate the contribution of each PFAS source to pollution plumes. This need for clarification regarding the contribution of each PFAS source to pollution, e.g. in the vicinity of drinking water wells, etc., is becoming crucial for the protection of water resources, (shared) responsibilities and the search for the (multiple) origins of pollution in the context of legal court expertise.

**Sources of pollution by PFAS** are varied and can be found on industrial sites that have used these products, sites that have suffered fires or fire training sites where fire-fighting foams have been used. (AFFF : Anti Fire Fighting Foams or Aqueous Film Forming Foam, e.g. at airports) have been used. Agricultural land is also a source of PFAS pollution, due to the input of sewage sludge, which contains accumulated PFAS.

The following (historical) activities may be the source of PFAS pollution:

- □ Fire-fighting training,
- Airport or air base on a military site,
- □ Fire site and use of AFFF,
- □ Electrochemical galvanisation,
- Production of waxed paper and cardboard,
- □ Manufacture of waterproof textiles,
- □ Sprays, paints, waterproofing lacquers,
- Production and application of Teflons (PTFE, etc.),
- Petroleum and chemical industry sites and/or production and application of paints, dyes, inks, pigments, chemical waxes and polishing products,
- Solvent applications (garages, dry cleaners, laundries, etc.),
- Landfill sites and former municipal landfill sites, etc. (ISDD, ISDND, ISDD, etc.),
- Dyeing & Tanning,
- Carpets, rugs, fabrics and plastics with flame retardants,
- Production of objects and furniture containing surfaces,
- □ Production of cleaning products,
- Dependence Photographic chemistry (laboratories, paper and film production, etc.),
- □ Production of electronic components,
- Production and application of pesticides and biocides,
- Production of cosmetics,
- □ Sites receiving WWTP sludge.

#### 2. <u>Environmental chemistry</u>

#### The environmental chemistry of PFAS is particularly important and complicated.

There is no group of pollutants with a more complex environmental chemistry than PFAS. In particular, there are **more than 9,000 PFAS substances**, divided into **33 categories of substances**.



The best known are Perfluoroalkane sulphonic acids (PFASs), Perfluoroalkyl carboxylic acids (PFCAs), Perfluoroalkyl phosphates and their esters, Fluorotelomer alcohols (**FTOH**), etc. (including more than 29 other groups ...). Some of them, such as the **PFOA** : Perfluorooctanoic acid and **PFOS** : Perfluoro-octane-sulphonate are banned (and **prohibited in EC and USA & Canada**) by the **Stockholm Convention** in the category of **POPs** : Persistent Organic Pollutants. PFOA is carcinogenic. **Commercial products** mainly contain **mixes**.

The reason for the high solubility in water, associated with lipophilicity, is based on the fact that **PFAS** :

- > Anionics (e.g. sulphonates, sulphates, carboxylates and phosphates),
- > **Cationics** (e.g. quaternary ammonium),
- > Amphoteric (e.g. betaines and sulpho-betaines): base + acid and
- > Non ionic (e.g. polyethylene glycols, acrylamide oligomers).

It is very important to emphasize that poly-fluorinated PFAS that are not fully fluorinated ('precursors') can be converted by bio-transformation into persistent, fully fluorinated chemicals, per-fluorinated PFAS. The complete microbiological degradation of PFAS has not yet been demonstrated.

In recent AFFFs (since about 2006 – 2015) the part of 6,2-FTAB and 6,2-FTS is very dominant and especial that one of 6,2-FTAB (Capstone B).

Figs. 1a & 1b show an **example of the biotransformation of 6 :2-FTAB & 6 :2-FTS** in soil and water to volatile Fluorotelomer Alcohols (FTOHs) which then migrate into soil gas (vapor) and ambient air. FTOHs are then microbiologically transformed into stable per-fluorinated PFASs. For example, 6:2-FTOH is biotransformed into PFHxA, PFPeA and PFBA and 8:2-FTOH into PFOA, PFHpA, PFHxA, PFPeA and PFBA. Fig 1c shows the Photolysis pathways of 6:2-FTAB.

In top-soils and in surface waters an additional chemo-transformation happens by UVbased photolysis. In this case the final degradation products could be even ultrashort PFAS, like PFPrA (Perfluoro propionic Acid) and TFA (Trifluoro acetic acid), beside PFHxA, PFPeA and PFBA (Naveed, A. et al 2024, cf. Fig 1c).



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Fig. 1a : Biotransformation scheme of polyfluorinated PFAS (Precursors): Example: 6 :2 FTAB and its degradation via 6 :2 FTS and 6 :2 FTOH to per-fluorinated PFAS PFBA, PFPeA and PFHxA (LaFond et al. 2023, D.M.J. Shaw et al. 2019, Ying Shi, 2018 and V. Mendeza et. al. 2022)



Fig. 1b : Example of the biotransformation of 6 :2 FTAB, 6 :2 FTS, 8 :2 FTS and polyfluorinated alkyl phosphates (PAP) in soil and water to Fluorotelomer alcohols (FTOH) and per-fluorinated PFAS, e.g. to PFOA, PFHpA, PFHxA, PFPeA and PFBA (L. KOPF / HPC, 2017 and F. KARG, 2022 & 2024).





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Fig. 1c: Photolysis of 6:2-FTAB to ultrashort PFAS, like PFPrA (Perfluoro propionic Acid) and TFA (Trifluoro acetic acid), beside PFHxA, PFPeA and PFBA (Naveed, A. et al 2024)

In the event of a change in pH, some PFAS may become more or less soluble, which also has an impact on emissions of volatile fluorinated telomers such as FTOH, etc. in soil gas (vapor). Certain precursors could change their solubility (and their extractable properties during chemical analysis procedures). For example, the intrusion of seawater into the aquifer could result in an increase in the basic pH and therefore in the solubility of Capstone B. This was observed in 2022 in the port area of Hamburg/Germany following seawater flooding and groundwater intrusion (see Fig. 2). These effects could result in groundwater concentrations more than 10 times higher than before seawater intrusion into the soil and groundwater.



Fig. 2 : Solubility of 6:2-FTAB & 6:2-FTNO according to pH (example: before and after seawater intrusion into an aquifer and change to a slightly alkaline pH)

#### 3. Differentiation of sources of PFAS pollution

Pollution of soil, groundwater and surface water by PFAS is frequently a mixture of several sources of commercial products and sources of pollution. It is possible to carry out complete screening of individual PFAS substances to identify between 9,000 and 12,000 molecules, but in the day-to-day management of pollution in the environment this is not applicable due to the limits of technical and economic feasibility.

For this reason, it is necessary to reduce the number of PFAS compounds to be analysed during environmental assessments by means of the chemical Identification of commercial products suspected of being the source of contamination. As PFAS are a family of more than 9,000 compounds, it would be impossible to quantify them all.

Typical sources of pollution caused by PFAS include, for example, applications of fire-fighting foams (AFFF = Aqueous Film Forming Foam or Anti Fire Fighting Foams), for example during fire drills and fires at airports, refineries and oil depots, industries involved in the production and processing of PFAS polymers, galvanizing activities such as chromium plating, the agricultural application of sewage WWTP sludge or fertilizers containing PFAS, the application of water-repellent coatings to paper, cardboard and textiles, etc., but also when they are used as flame retardants in the electrical and





electronics industries... The list of sources of PFAS in the environment is long, and there are also volatile PFAS pollutants that can enter indoor air via soil gases, such as fluorotelomer alcohols (FTOH).

One of the current challenges in managing environmental problems linked to PFAS is to identify the various sources of pollution caused by PFAS or their commercial products, for example in the case of major contamination of groundwater by PFAS.

Experience and assessments of more than 800,000 PFAS analyses in soil, groundwater and surface water show the presence of various PFAS clusters and substances, such as perfluorinated carboxylic acids (PFCAs), perfluorinated sulphonic acids (PFSAs) and others, which are typical of certain industrial sectors. Other important PFAS groups are fluorotelomer sulphonic acids (FTS), fluorotelomer alcohols (FTOH) and fluorinated sulfo-betaines (FTAB), from a total of 33 different PFAS groups.

When identifying and differentiating PFAS sources, various relative concentration distributions, relationships with perfluorinated carboxylic acids (PFCAs) and with various poly-fluorinated PFASs as 'precursors', as well as various statistical distribution models, are taken into account.

Commercial PFAS products are examined using non-target analyses to identify as many individual PFAS substances as possible present in PFAS products. To this end, percolation tests to simulate the ageing of 'fresh' commercial PFAS products are being carried out on lysimeters, with bacteriological degradation allowing the biotransformation of poly-fluorinated PFAS (precursors) to per-fluorinated PFAS, and to identify the chromatographic effects of soils (F. Karg et al.: 2023 and 2024).

The purpose is to record the resulting mixtures of individual PFAS molecules by HR-MS (high-resolution mass spectrometry). The results obtained are typical fingerprints of fresh and degraded commercial PFAS products for the complete database of parent mixtures of PFAS products.

PFAS sources can be identified from this database using standard analyses and a multivariate statistical identification program. Multi-Vector-Analysis (MVA) is carried out in several dimensions using artificial intelligence (machine learning), so that PFAS sources can also be determined in the event of groundwater pollution containing a mixture of several different PFAS sources.

In addition to the use of standard environmental analyses of polluted sites for MVA identification of the different sources of PFAS pollution, other analytical tools may also be applicable, such as the 'Top Assay' (Houtz & Sedlak: 2012, Glöckner et al.: 2021) to best determine the mass of poly-fluorinated precursors present, which can be oxidized to perfluorinated carboxylic acids (PFCAs)





(see Fig. 3). In addition, measurements of isotope ratios between PFAS source areas and plume areas can be carried out to determine PFAS sources (Quian et al. 2023), as well as the use of comprehensive 'untargeted analyses', in which hundreds of individual PFAS molecules can be identified and at least semi-quantified.

Such applications are more time-consuming and expensive than the normal standard PFAS analysis for polluted sites, which is mainly used to identify PFAS sources. The MVA application can avoid this and be more cost-effective.

More than 9 000 – 12 000 PFAS have been polluting the environment and our health for decades. This also includes volatile PFAS, such as fluorotelomer alcohols (6:2 or 8:2 FTOH, etc.). The problem in investigating PFAS-contaminated sites lies mainly in the huge quantity of poly-fluorinated PFASs, which are biotransformed into per-fluorinated PFASs over time. Individual poly-fluorinated PFASs may also be relatively resistant in the environment and transform only very slowly into perfluorinated PFASs (such as Capstone B - FTAB, FTS or FTOH). In order to improve transparency and knowledge of this large number of PFAS pollutants, there are several approaches to the investigation, risk assessment and remediation of PFAS.

An important characteristic of PFASs is their chemical behavior in the environment, as polyfluorinated PFASs (precursors) are converted to stable perfluorinated PFASs by biotransformation. The risk assessment of individual cases via a QSRA\* (or RRA\*) can estimate the future risks of most of the "precursors", since an analysis based on 28 to 70 individual PFASs is carried out before and after examination by the 'Top Assay' test (see Fig. 3). In the Top Assay, poly-fluorinated PFAS are converted into stable per-fluorinated PFAS by oxidation via persulphate, into which they are generally biotransformed after a certain residence time in the environment (see also Fig. 2a & b). Thanks to the 'Top Assay' test, investigations and risk assessments include almost all transformable 'precursors' as well as the remaining per-fluorinated PFAS end products.

(\*EQRS & \*ARR: Quantitative Health Risk Assessment and Residual Risk Analysis).



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Fig. 3 : Top Assay (Total Oxidizable Precursor method) to quantify oxidizable poly-fluorinated PFAS, transformable into per-fluorinated carboxylic acids (F. Karg 2015, Houtz & Sedlac 2012, Glöckner et al.: 2021).

The following examples illustrate some of the ways in which sources of PFAS can be identified and differentiated using certain standard analyses, taking into account experience of MVA (Multi-Vector-Analysis) applications based on artificial intelligence in Europe (EU) and the United States.

**Typical sources of PFAS** include areas that have experienced fire and fire training events, civil and military airports (anti fire-fighting foams / AFFF), but other activities can also cause PFAS contamination in soils and groundwater.

These activities include, for example, the spreading of sewage sludge from wastewater treatment plants, galvanic chromium plating, landfill leachates, paper production, textile impregnation, the electrical and electronics industries, paint companies, cleaning products and fluor-polymer production, etc.

Various statistical analyses and visualizations, based on experiments and PFAS clusters from over 800,000 soil and groundwater analyses (NAS: 2023, F. Karg: 2024), help to identify PFAS sources.



# The methodology for differentiation of PFAS sources can be divided into three main categories:

I. Analysis of data and (historical) information available that can be used to identify potential sources of PFAS at different sites (specific industrial sites airports, landfills, etc.).

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- II. Laboratory methods for analyzing PFAS in environmental samples.
- III. Advanced forensic investigations to identify potential sources of PFAS more precisely.

#### Category I. includes the following phases :

- Historical investigation of potential sources of PFAS from suspect areas. The objective is to list and map the areas of potential sources of PFAS pollution.
- Study the hydrogeological zones above the suspected areas to determine whether there are (or have been) activities that use (or have used) PFAS.
- To obtain a detailed understanding of the geology, hydrogeology, hydrology, stormwater infrastructure and surface water run-off of the sites. This also includes the nature of the soil, the location and depth of nearby wells, and the depth and direction of surface and groundwater flow.
- Prepare a conceptual plan of the site concerning PFAS sources, transfer paths and exposure paths to targets.

#### Categories II and III focus on :

- Evaluation of PFAS chemical analysis data as a screening step to determine whether indications or evidence of source differentiation can be identified.
- Analysis of advanced PFAS forensics on selected samples to differentiate potential sources of PFAS (e.g. by structural isomerism, isotopic studies, Top Assay, MVA applications, etc.).

# The general differences between PFAS's commercial products can be identified, for example, through the following distinctions:

Modern anti fire-fighting foams (AFFF) are based on fluoro-telomers, mainly with C6-PFAS molecules. Although these firefighting foams do not contain PFOA or PFOS, when emitted into the environment they can degrade to other shorter-chain PFAS, such as perfluoro-hexanoic acid (PFHxA), perfluoro-pentanoic acid (PFPeA), buntanonic acid and 5:3 fluorotelomer carboxylic acid (FTCA).





- In the early 2000s (following general requests from the US and European authorities), the chemical industry began to phase out anti fire-fighting foams based on C8-PFAS, as PFOS and PFOA were classified as being "too toxic". Producers of fluorotelomer-based firefighting foams then turned to the use of short-chain forms of PFAS with six fluorinated carbons (called C6-PFAS), which did not contain PFOS or PFOA (and could not be broken down into these products either). These C6-PFAS firefighting foams can also contain PFHxA, PFPeA and 6:2-fluorotelomersulphonate (6:2-FTS) as well as fluorotelomers, which can be transformed over time into their end groups, perfluoro carboxylic acids with less than six carbon atoms. The exact period of transition from C8-PFAS fire-fighting foams to C6-PFAS fire-fighting foams varies according to the application site (civil or military airports, fire training areas, oil sites, etc.).
- Before 2016, many firefighting foams contained PFAS with eight fluorinated carbon chains. Some of these long-chain firefighting foams, or C8 PFASs, contained PFOS until the early 2000s, and PFOA and other long-chain PFCAs until around 2015, when these products were withdrawn from the market.
- Analyses of the molecular chemical structures of PFASs can be useful in relation to existing information such as origin, period and likely use. Structural differences include, for example, structural isomerism (linear and branched isomers, etc.). For example, if a particular cyclic PFAS with an eight-carbon chain, such as perfluoroethyl-cyclohexane sulphonate (PFECHS: CAS 335-24-0) is observed, it could originate from a corrosion inhibitor used in aircraft hydraulic fluids (MPART: 2020). PFECHS is not known to be a component of fire-fighting foams.
- Specific PFASs can be used as markers in the production of fluoropolymers, food packaging and paper coatings, as well as cosmetics. They are not currently thought to be related to firefighting foam products.
- Some PFASs can be chemically biotransformed (i.e. broken down into smaller, stable chemicals), but these poly-fluorinated PFASs (precursors) often transform into other PFASs, particularly per-fluorinated PFAAs.

#### Identifying and differentiation of PFAS Contamination Sources:

The standard environmental analyses used during investigations of sites polluted by PFAS can, in principle, be used to identify and differentiate sources of PFAS. Between 30 and 70 individual PFAS substances are analyzed. The analytical data obtained can be very complex due to the





presence of many different PFAS in an environmental sample and pose a challenge in interpreting the data. For this reason, a computerized MVA application with artificial intelligence is required.

Because of this complexity, large amounts of data need to be interpreted on the basis of statistical experience gained from large amounts of analysis in multi-vector analysis (MVA or polytopic analysis) with artificial intelligence, so that the sources of PFAS can be identified and differentiated with a very high probability. This can only be done as part of a parallel analysis of different pieces of evidence, as each analysis on its own is generally not sufficiently significant. The basis for this are 'target' (and 'non-target') analyses as well as the 'Top Assay', total organic fluorine (TOF) or **AOF (adsorbable total organic fluorine) and isotopic analyses**.

The following Experiences can be applied for Identifying and differentiation of PFAS Contamination Sources:

- TOF or Total Organo Fluorine is measuring PFAS Monomers & Polymers and other organo-fluorine Compounds, like Pesticides, Pharmaceuticals, etc. No Compound Identification is realized. The use of TOF can't be really recommended for PFAS analysis appreciation.
- AOF: Adsorbable Organic Fluorine is only measuring compounds which were priorly adsorbed in Water samples. AOF is measuring PFAS Monomers, small PFAS Polymer particles and other organo-fluorine Compounds, like Pesticides, Pharmaceuticals, etc. No Compound Identification is realized. The use of AOF can't be really recommended for PFAS analysis appreciation.
- NTA: Non-Target Analysis is the semi-quantitative Identification of up to 12 000 Compounds of PFAS Monomers. The costs and time needs are very important.
- QTA: Quantitative Target Analysis of up to 500 700 Compounds of PFAS Monomers (in general 20 – 70 compounds).
- QTA + TA: Quantitative Target Analysis after Top Assay of 20 200 Compounds PFCA Monomers (in general 20 – 70 compounds). Only per-fluorinated PFAS are analyzed, including poly-fluorinated PFAS after transformation to PFCA by persulfate.

Recommended are QTA: Quantitative Target Analysis of about 70 choosen compounds before and after Top Assay. Its important to analyse via the QTA also the ultrashort PFCA, like TFA (trifluoro-acetic acid), PFPrA (perfluoro-propanoic acid), TFMS (trifluoromethane-sulfonic acid), PFES (perfluoro-ethane-sulfonic acid) and PFPrS (perfluoropropane-sulfonic acid).







Fig. 4 : Analytical procedures for the identification and differentiation of PFAS sources using multivector analysis (MVA or poly-topic analysis) with artificial intelligence.

Figure 5 shows the procedure for identifying and differentiating potential sources of PFAS using MVA based on artificial intelligence (e.g. AFFF fire-fighting foams, sewage sludge, electroplating activities, textile and paper industries, waste dumps, etc.).

The procedure principles for identifying and differentiating potential PFAS Sources are the following:

- A. Lots of different Statistical Comparisons of Sample analyses with registered Standards of chemical PFAS Spectrums on about 70 analyzed compounds from commercial PFAS Products (AFFF, Galvanic products, Paper related production Products, Surfactants mixtures, etc.).
- B. Identification and Differentiation of PFAS Clusters by MVA on Artificial Intelligence via Clustering.









Fig. 5 : MVA analysis approach based on artificial intelligence to identify and differentiate sources of PFAS (e.g. AFFF fire-fighting foams, sewage sludge, galvanic activities, textile and paper industries, waste dumps, petroleum sites, etc.) (F. Karg et al.: 2023 & 2024).

Because of the many individual PFASs within this chemical family, 'non-target analysis' also offers the possibility of identifying unknown molecules, as the result is open-ended without any limitation on a standard list of pre-calibrated pollutants. Hundreds of individual compounds can be identified. The disadvantage is that non-targeted analysis is relatively expensive and time-consuming. Figure 6 shows the differences compared with standard analysis of selected PFAS parameters.

Figure 6 shows different AFFFs in Soil samples and the 6:2-FTAB & 6:2-FTS domination. The similarity of these analytical results needs the MVA-Application for product identification.

Figures 7 and 8 show PFAS cluster analyses to differentiate between several PFAS sources and Figure 9 shows PFAS Cluster Sources Overlapping and percentage parts of Groundwater pollution plume participation.



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Fig. 6: Different AFFFs in Soil samples and the 6:2-FTAB & 6:2-FTS domination.



Fig. 7: Various PFAS Clusters and Cluster Mix, identified to determine the sources of PFAS







Fig. 8 : Various PFAS Clusters:Commercial PFAS Products and their chemical signature in Radargrams



Fig. 9 : Identification and differentiation of 4 different Sources of 3 different PFAS Clusters and commercial PFAS products on the basis of groundwater analysis and MVA-AI.



The multi-vector analysis (MVA) computer tool with artificial intelligence to identify and differentiate the sources of PFAS clusters was created on real commercial PFAS Product and environmental analyses.

The basis is, among others, the experiences of the US Airport Cooperative Research Program (ACRP), the Airports Council International - North America (ACI-NA), the American Association of Airport Executives (AAAE), the USA National Academy of Sciences Guide (NAS: 2023), as well as chemical cluster determinations of fresh commercial PFAS products and aged commercial PFAS products (according to degradation procedures applied by HPC International; F. Karg et al. 2023 - 2024) as standards for the MVA-PFAS database. The ageing of commercial PFAS products was carried out using bio-lysimetric tests in 2024 & 2025 with exposure to degrading bacteria for biotransformation of poly-fluorinated PFAS into per-fluorinated PFAS (F. Karg et al. 2023 – 2024, NAS, 2023).

The analysis of PFAS clusters takes into account the environmental chemistry of PFAS and in particular the metabolism or biotransformation of poly-fluorinated PFAS into stable per-fluorinated PFAS, e.g. the metabolism of poly-fluorinated PFAS into stable per-fluorinated PFAS, for example  $6:2 \text{ FTAB} \rightarrow 6:2 \text{-FTS} \rightarrow 6:2 \text{-FTOH} \rightarrow \text{PFHxA} + \text{PFPeA} + \text{PFBA}$  etc. to determine the 'precursors' and final products in the event of environmental contamination by mixtures of PFAS through the biotransformation chain.

Site-specific investigations and the identification and differentiation of PFAS sources include the following multi-criteria applied in the MVA tool. All the criteria are always applied at the same time:

- A. Concentration ratios between PFAS: this is a simple first screening technique, where one or more concentration ratios between two or more different PFAS compounds can be applied. For example, PFHxS/PFOS ratios within a certain range have been used as an indicator of a source of fire-fighting foam (McGuire et al. 2014), and PFHxS/PFOA ratios have been identified as markers of other sources of industrial PFAS production (Guelfo and Adamson 2018).
- B. Circular diagrams and other data visualizations: These are statistical graphs showing the relative contribution of several molecules to the total PFAS concentration in the same study area.



They can help identify compositional differences between PFAS mixtures in different samples to determine whether there are chemical clues or evidence from different PFAS sources. It can also identify the mixing of different PFAS sources or changes in source signatures along a transport pathway (e.g. migration in groundwater) and also reveal biotransformations and soil chromatography effects.

C. Isomer ratios: Individual PFAS can exist in different isomeric forms and the relative abundance of different isomers in a sample can be used to infer the source of PFAS (Charbonnet et al. 2021). The presence of branched isomers (structural isomers) in a sample can be measured using standard methods (which analyse around 60 to 70 individual substances). The two different PFAS production processes, ECF (electrochemical fluorination) and telomerisation, result either in a mixture of branched and linear PFAS (ECF) or in isomers of purely linear PFAS.

This information could be useful as evidence to identify sources of PFAS. **Branched isomers indicate that PFAS come from the specific electrochemical fluorination (ECF)** manufacturing process. The proportion of branched isomers in ECF products falls within a relatively narrow range (ITRC 2022 a&b). If the proportion of branched isomers in a sample is below this range, this may indicate that products containing PFAS generated by telomerisation-based manufacturing processes are also present.

Modern firefighting foams (AFFFs) contain PFASs that are produced solely by telomerization processes. Older AFFF blends may contain PFAS produced by ECF manufacture or telomerization. In the USA and somewhat later in Western Europe, the production of AFFF mixes using ECF production was discontinued in the early 2000s.

Another important fact is that branched isomers of some PFAS (e.g. PFOS) propagate more rapidly in groundwater than linear isomers of the same compound (different chromatographic effects in soil) due to the different interactions of the isomers with the soil. This can lead to enrichment of **ramified isomers** in groundwater hydro-geologically downstream of a PFAS source (Nickerson et al. 2020).

D. Analysis of the main components of PFAS and hierarchical classification study of PFAS clusters: These statistical methods are used to analyze data associated with many variables (e.g. measured PFAS) in order to identify differences between clusters of PFAS that can be visualized graphically. This data analysis facilitates the identification of





different groups (or clusters) of PFAS mixtures (each of which may come from different sources or commercial PFAS products (see Figs. 7 & 8), which clusters may overlap).

- E. Research methods (screening): identification and characterization of sources of PFAS :
  - Frequency of detection: Experimental statistics (from over 800,000 individual analyses from different PFAS source locations) on the relative detection of individual PFAS (in each analysis data set) help to distinguish between different PFAS sources. The absence (or low frequency of detection) of specific PFAS in samples from one type of site can also be used in the same way. Experiments with over 800,000 individual analyses of samples from various PFAS source locations show very clearly these differences depending on the PFAS source (NAS: 2023).
  - Concentration distributions: These data help to determine the relative distribution of individual PFAS found in environmental samples from different locations. The average concentration of individual PFAS from the samples examined is used in the MVA assessment (see Fig. 7a - d). These data provide information on the relative proportion of different PFAS sources in a given site type, as well as the relative presence of PFAS associated with different sources.
  - PFAS composition ratios: These ratios show the general distribution of PFAS within the sample groups. This includes the percentage of perfluoroalkyl carboxylic acids (PFCAs), perfluoroalkyl sulphonic acids (PFSAs) and non-perfluoroalkyl acids (non-PFAAs) relative to the total of overlapping PFAS mixtures (e.g. present in groundwater). The application of this methodology also includes the quantification of concentration ratios between frequently detected individual PFASs.
  - Statistics for identifying and characterizing sources of PFAS using multi-vector analysis (MVA):

The results of the PFAS source determination are presented in five different types of statistical visualizations of PFAS source categories. These five types of MVA are:

- □ MVA1 : PFAS composition circular diagrams,
- □ MVA2 : Boxplot distribution of concentrations and PFAS histograms,
- □ MVA3 : Average PFAS concentrations and detection frequency: Heat-Maps,
- □ MVA4 : Average PFAS concentration and detection frequency: 'Cross-plots',





# International PFAS Congress – Paris 17 - 20 June 2025 HPC INTERNATIONAL SAS MVA5 : Report of PFAS logarithmic average concentrations: Relative and radial point diagrams.

Figure 10 shows a summary of the parameters evaluated for the identification and differentiation of AI-MVA (artificial intelligence-based multi-vector analysis) PFAS sources.



Fig. 10 : Parameters evaluated for the identification and differentiation of PFAS sources using AI-MVA (artificial intelligence-based multi-vector analysis).

Table 1a & b shows the recommended standard analysis parameters for using the multivector analysis (MVA) computer tool with artificial intelligence to identify and differentiate sources of PFAS.





| PFAS  |        | Q           | CAS         | VTR | Dir. CE | AM       |
|---|--------|-------------|-------------|-----|---------|----------|
|   |        | nv          |             |     | EP2020/ | 20/06/23 |
| DEP A (acida parfluorabutanaiqua)                           |        |             | 275 22 4    |     | 2184    | гапсе    |
| DEDa ( (acida parfluoropartenaigua)                         | 11g/1  | 5           | 2706.00.2   |     |         |          |
| DELLyA (acide perfluorobevencique)                          | 11g/1  | 1           | 2700-90-3   |     |         |          |
| DELla A (acide perfluorohexanoique)                         | 11g/1  | 1           | 275.85.0    |     |         |          |
| PFHpA (acide perintoroneptanoique)                          | 11g/1  | 1           | 375-85-9    |     |         |          |
| PFOA ineaire (acide perituorooctanoique)                    | ng/l   | 1           | 335-07-1    |     |         |          |
| PFOA ramifie (acide perfluorooctanoique)                    | ng/l   | 1           | 335-67-1    |     |         |          |
| PFOA totale (acide perfluorooctanoique)                     | ng/l   | 1           | 335-67-1    |     |         |          |
| PFNA (acide perfluorononanoique)                            | ng/l   | 1           | 375-95-1    |     |         |          |
| PFDA (acide perfluorodecanoique)                            | ng/l   | 1           | 335-76-2    |     |         |          |
| PFUnDA (acide perfluoroundecanoique)                        | ng/l   | 1           | 2058-94-8   |     |         |          |
| PFDoDA (acide perfluorododecanoique)                        | ng/l   | 2           | 307-55-1    |     |         |          |
| PFTrDA (acide perfluorotridecanoique)                       | ng/l   | 1           | 72629-94-8  |     |         |          |
| PFTeDA (acide perfluorotetradecanoique)                     | ng/l   | 1           | 376-06-7    |     |         |          |
| PFHxDA (acide perfluorohexadecanoique)                      | ng/l   | 2           | 67905-19-5  |     |         |          |
| PFODA (acide perfluorooctadecanoique)                       | ng/l   | 1           | 16517-11-6  |     |         |          |
| PFBS (acide perfluorobutane sulfonique)                     | ng/l   | 1           | 375-73-5    |     |         |          |
| PFPeS (acide perfluoropentane sulfonique)                   | ng/l   | 1           | 2706-91-4   |     |         |          |
| PFHxS linéaire (acide perfluorohexane sulfonique)           | ng/l   | 1           | 355-46-4    |     |         |          |
| PFHxS ramifié (acide perfluorohexane sulfonique)            | ng/l   | 1           | 355-46-4    |     |         |          |
| PFHxS totale  | ng/l   | 1           | 355-46-4    |     |         |          |
| PFHpS (acide perfluoroheptane sulfonique)                   | ng/l   | 1           | 375-92-8    |     |         |          |
| PFOS linéaire (acide perfluorooctane sulfonique)            | ng/l   | 1           | 1763-23-1   |     |         |          |
| PFOS ramifié (acide perfluorooctane sulfonique)             | ng/l   | 1           | 1763-23-1   |     |         |          |
| PFOS totale (acide perfluorooctane sulfonique)              | ng/l   | 1           | 1763-23-1   |     |         |          |
| PFDS (acide perfluorodecane sulfonique)                     | ng/l   | 1           | 335-77-3    |     |         |          |
| 4:2 FTS (acide 4:2 fluorotelomer sulfonique) H4-PFOS        | ng/l   | 1           | 757124-72-4 |     |         |          |
| 6:2 FTS (acide 6:2 fluorotelomer sulfonique)                | ng/l   | 1           | 27619-97-2  |     |         |          |
| 8:2 FTS (acide 8:2 fluorotelomer sulfonique)                | ng/l   | 1           | 39108-34-4  |     |         |          |
| 10:2 FTS (acide 10:2 fluorotelomer sulfonique)              | ng/l   | 1           | 120226-60-0 |     |         |          |
| MePFOSAA (acide N-méthylperfluorooctane sulfonamide         |        | -           |             |     |         |          |
| acétique)   | ng/l   | 1           | 2355-31-9   |     |         |          |
| EtFOSAA (acide N-éthylperfluorooctane sulfonamide acétique) | ng/l   | 1           | 2991-50-6   |     |         |          |
| PFOSA linéaire (perfluoro-n-octanesulfonamide)              | ng/l   | 2           | 754-91-6    |     |         |          |
| PFOSA ramifié (perfluoro-n-octanesulfonamide)               | ng/l   | 2           | 754-91-6    |     |         |          |
| PFOSA totale (perfluoro-n-octanesulfonamide)                | ng/l   | 2           | 754-91-6    |     |         |          |
| MeFOSA linéaire (N-méthylperfluorooctanesulfonamide)        | ne/1 1 | 21.506.22.2 |             |     |         |          |
| (MePFOSA)   | ng/I   | 1           | 31506-32-8  |     |         |          |
| 6:2-FTAB (6:2 fluorotelomer sulfonamido propyl betaine)     |        |             | 34455-20-3  |     |         |          |
| Capstone B  | ng/l   | 10          | 54455-29-5  |     |         |          |

## Table 1a: Recommended standard analysis parameters for using the multi-vector analysis (MVA) computer tool with artificial intelligence to identify and differentiate sources of PFAS.





| PFAS  | LQ<br>Eaux |    | CAS          | VTR | Dir. CE<br>EP2020/<br>2184 | AM<br>20/06/23<br>France |  |  |  |  |
|---|------------|----|--------------|-----|----------------------------|--------------------------|--|--|--|--|
| MeFOSA ramifié (N-méthylperfluoro-n-octanesulfonamide)        | no/1       | 1  | 31506-32-8   |     |                            |                          |  |  |  |  |
| MeFOSA totale (N-méthylperfluoro-n-octanesulfonamide)         | 11g/1      | 1  |              |     |                            |                          |  |  |  |  |
| (MePFOSA)   | ng/l       | 1  | 31506-32-8   |     |                            |                          |  |  |  |  |
| 8:2 DiPAP (8:2 polyfluoroalkyl phosphate diester)             | ng/l       | 1  | 678-41-1     |     |                            |                          |  |  |  |  |
| HFPO-DA (acide hexafluoropropyleneoxide dimer) Gen X          | ng/l       | 1  | 13252-13-6   |     |                            |                          |  |  |  |  |
| EtFOSA linéaire (N-éthylperfluorooctanesulfonamide) (EtPFOSA) | ng/l       | 1  | 4151-50-2    |     |                            |                          |  |  |  |  |
| EtFOSA ramifié (N-éthylperfluorooctanesulfonamide) (EtPFOSA)  | ng/l       | 1  | 4151-50-2    |     |                            |                          |  |  |  |  |
| EtFOSA totale (N-éthylperfluorooctanesulfonamide) (EtPFOSA)   | ng/l       | 1  | 4151-50-2    |     |                            |                          |  |  |  |  |
| MeFBSAA (perfluorobutanesulfonamide(N-méthyl)acetate)         | ng/l       | 5  | 159381-10-9  |     |                            |                          |  |  |  |  |
| 5:3-FTCA: 5:3 acide carboxylique fluorotélomère)              | ng/l       | 1  | 914637-49-3  |     |                            |                          |  |  |  |  |
| 6-2-FTCA: 6:2 acide carboxylique fluorotélomère               | ng/l       | 5  | 53826-12-3   |     |                            |                          |  |  |  |  |
| 8:2 FTUCA (acide 2H-perfluoro-2-décenoique)                   | ng/l       | 1  | 70887-84-2   |     |                            |                          |  |  |  |  |
| DONA (acide 4,8-dioxa-3H-perfluorononanoique)ADONA            | ng/l       | 1  | 919005-14-4  |     |                            |                          |  |  |  |  |
| MeFBSA (n-méthylperfluorobutanesulfonamide)                   | ng/l       | 1  | 68298-12-4   |     |                            |                          |  |  |  |  |
| PFBSA (perfluorobutanesulfonamide)                            | ng/l       | 1  | 30334-69-1   |     |                            |                          |  |  |  |  |
| PFECHS (acide perfluoro-4-éthylcyclohexanesulfonique)         | ng/l       | 1  | 646-83-3     |     |                            |                          |  |  |  |  |
| PFNS (acide perfluorononane sulfonique)                       | ng/l       | 1  | 68259-12-1   |     |                            |                          |  |  |  |  |
| PFDoDS (acide perfluorododecane sulfonique)                   | ng/l       | 1  | 79780-39-5   |     |                            |                          |  |  |  |  |
| 6:2 diester de phosphate fluorotélomérique. 6:2 diPAP         | ng/l       | 10 | 57677-95-9   |     |                            |                          |  |  |  |  |
| 6:2 8:2 diester de phosphate fluorotélomérique. 6:2 8:2 diPAP | ng/l       | 10 | 943913-15-3  |     |                            |                          |  |  |  |  |
| PFHxSA (perfluorohexanesulfonamide)                           | ng/l       | 1  | 41997-13-1   |     |                            |                          |  |  |  |  |
| PFUnDS (acide perfluoroundecane sulfonique)                   | ng/l       | 2  | 749786-16-1  |     |                            |                          |  |  |  |  |
| PFTrDS (acide perfluorotridecane sulfonique)                  | ng/l       | 2  | 791563-89-8  |     |                            |                          |  |  |  |  |
| EtFOSE (2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol)     | ng/l       | 5  | 1691-99-2    |     |                            |                          |  |  |  |  |
| MeFOSE (2-(N-methylperfluoro-1-octanesulfonamido)-ethanol)    | ng/l       | 5  | 24448-09-7   |     |                            |                          |  |  |  |  |
| NFDHpA (Nonafluoro-3,6-dioxaheptanoic acid)                   | ng/l       | 1  | 151772-58-6  |     |                            |                          |  |  |  |  |
| PFMPA (Perfluoro-3-methoxypropanoic acid)                     | ng/l       | 1  | 377-73-1     |     |                            |                          |  |  |  |  |
| PFMBA (perfluoro-4-methoxybutanoic acid)                      | ng/l       | 1  | 863090-89-5  |     |                            |                          |  |  |  |  |
| C6O4 (Perfluoro([5-methoxy-1,3-dioxolan-4-yl]oxy)acetic acid) | ng/l       | 10 | 1190931-41-9 |     |                            |                          |  |  |  |  |
| 6:2-FTOH (6:2 fluorotelemer alcohol) FHET                     | ng/l       | 20 | 647-42-7     |     |                            |                          |  |  |  |  |
| 8:2-FTOH (8:2 fluorotelemer alcohol) FOET                     | ng/l       | 10 | 678-39-7     |     |                            |                          |  |  |  |  |
| PFAS Ultrashorts  |            |    |              |     |                            |                          |  |  |  |  |
| TFA (trifluoroacetic acid)                                    | ng/l       | 10 |              |     |                            |                          |  |  |  |  |
| PFPrA (perfluoropropanoic acid)                               | ng/l       | 10 |              |     |                            |                          |  |  |  |  |
| TFMS (trifluoromethanesulfonic acid)                          | ng/l       | 10 |              |     |                            |                          |  |  |  |  |
| PFES (perfluoroethanesulfonic acid)                           | ng/l       | 10 |              |     |                            |                          |  |  |  |  |
| PFPrS (perfluoropropanesulfonic acid)                         | ng/l       | 10 |              |     |                            |                          |  |  |  |  |

Table 1b: Recommended standard analysis parameters for using the multi-vector analysis (MVA) computer tool with artificial intelligence to identify and differentiate sources of PFAS.

#### Summary

In the context of groundwater pollution by PFAS, it is becoming increasingly important to identify and, above all, to differentiate the contribution of each PFAS source to pollution plumes. This need for clarification of the contribution of each PFAS source to pollution, e.g. in the vicinity of catchments (for drinking water, etc.), is becoming increasingly crucial for the protection of water resources, (shared) responsibilities and the search for the (multiple) origins of pollution in the context of legal expertise.



To conclude, it is now possible (using comprehensive statistical, graphical & chemicalmathematical studies, including Artificial Intelligence based Clustering with Machine Learning) to use the Multi-Vector-Analysis (MVA) in several dimensions to identify and differentiate the sources of PFAS pollution in soils, groundwater and surface water, and in some cases even to identify the commercial PFAS products causing the pollution.

The procedure principles for identifying and differentiating potential PFAS Sources are the following:

- A. Lots of different Statistical Comparisons of Sample analyses with registered Standards of chemical PFAS Spectrums on about 70 analyzed compounds from commercial PFAS Products (AFFF, Galvanic products, Paper related production Products, Surfactants mixtures, etc.).
- B. Identification and Differentiation of PFAS Clusters by MVA on Artificial Intelligence via Clustering.

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### How to extend the useful life of GACs to reduce opex cost for removing PFAS and other emerging contaminants: the LIFE Cascade project

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The EU LIFE CASCADE project (Closed-loop wAter Systems in textile industrial distriCts: orchestrAteD rEmoval of emerging pollutants from textile wastewater) (EU Call LIFE-2022-SAP-ENV) aims to develop analytical procedures and wastewater treatment technologies to detect and remove poly- and per-fluorinated substances (PFAS).

The project, coordinated by Centro Tessile Serico Sostenibile (CTSS), involves research laboratories of different universities and organizations (Politecnico di Milano - Italy, Università degli Studi dell'Insubria - Italy, Università degli Studi di Brescia - Italy, AquaSoil - Italy, Acquedotto Industriale - Italy, Biochemie - Italy, CITEVE - Italy, COMO ACQUA - Italy, De Nora- Italy, Lariana Depur - Italy, ZDHC - the Netherlands) with world-class expertise in the management of pollutants in wastewater (WW).

The implementation of the treatments will reduce the discharge into the environment of these micropollutants both from the treated wastewater and from the sewage sludge and will allow the reuse of the treated wastewater.

The output of the project includes three layers: standardized and multi-lab validated analytical protocols to detect and quantify PFAS in heterogeneous textile wastewaters (WW); a modular set of WW treatment units to be installed at factory- and central treatment plant levels; a risk-based methodology to support the design and implementation of the best combination of treatment modules according to the textile district configuration and WW characteristics.

The project also foresees deepening an evaluation of the ecotoxicity of treated WWTP effluents and the response of the macrobenthos community to PFAS presence.

During the project, combinations of different technologies are tested to identify the best combinations to extend the useful life of GACs and reduce the opex cost for removing PFAS and other emerging contaminants. The use of ozone or other technologies to remove organics or other PFAS competitors has proven to be very useful in significantly extending the useful life of GACs while reducing the operating costs of media replacement.

## Contribution of Liquid Chromatography Coupled with High-Resolution Mass Spectrometry for the Identification of PFAS

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"Theme: Identification & Characterization of PFAS Sources"

Keywords: PFAS, Non-targeted Analysis, Liquid Chromatography, High-Resolution Mass Spectrometry.

#### Abstract:

The presented study focuses on the application of non-targeted analysis methodology for the specific search of "unknown" PFAS or a wide range of PFAS.

#### Context:

Per- and polyfluorinated substances (PFAS) constitute a large family of fluorinated chemicals comprising several thousand substances. More than 14,000 PFAS molecules are listed, including 4,700 identified by a CAS number (OECD, 2021). The widespread use of these PFAS substances, combined with their physicochemical properties, result in the presence of these molecules in all environmental compartments. Toxic effects on human health and the environment have been observed for several molecules in this large family of industrial chemicals. Regulations have been established in several countries to limit human and environmental exposures.

Standardized methods (ISO or US EPA) commonly used for the analysis of these molecules are based on gas chromatography or liquid chromatography coupled with mass spectrometry. These analytical methods attain detection limit in accordance o meet various regulations. However, they only allow the analysis of a defined and limited list of PFAS. The use of liquid chromatography coupled with highresolution mass spectrometry (LC-HRMS) will allow to attempt the identification of unknown PFAS that may be present in a sample.

#### Methodology and Results:

A non-targeted analysis by LC-HR-MS generates the exact mass, retention time, and fragmentation spectrum of species or entities present in a sample. After a series of validation steps including comparison with internal and external data bases and even acquisition of new experimental data, this analytical approach would enable the identification of initially unknown molecules (C.Soulier, 2021). A non-targeted analysis methodology specifically focused on PFAS has been implemented at CIRSEE. It combines the use of databases and tools specific to the chemical structures of PFAS, such as the search for homologs using Kendrick Mass Defect calculations (S.Merel, 2023) and characteristic fragments (Hu, 2023), for the identification of molecules belonging to this family.

This methodology was applied to the analysis of resources used for drinking water production influenced by activities likely to release PFAS and the corresponding treated waters. Thus, a total of 14 samples were processed with this non-targeted method as well as with a targeted analysis method. Among the 61 molecules measured by targeted analysis, only 13 different PFAS were quantified in all samples. The non-targeted analysis identified 136 entities that could belong to the PFAS family. Further research was conducted on ten of the most relevant entities to propose structures by cross-referencing the acquired data.

The identify of a suspected molecule car only be confirmed by the purchase and analysis of the standard, when available. Two suspected PFAS were confirmed following the acquisition of the

corresponding standard. The comparison of data acquired during the sample analysis and the standard is illustrated in **Figure 1**. Molecules identified by this non-targeted method could then be added to the lists of targeted molecules for monitoring.

This technology is expensive, requires a high level of expertise, and processing of acquired data is timeconsuming. It is therefore unlikely to be used for routine analysis. However, it is extremely useful to characterize PFAS pollution of a resource, to evaluate the PFAS transport to the environment of wastewater or industrial discharges. It also has the advantage of allowing retrospective analyses by searching for the presence of a molecule in samples without having to reanalyze them.



## Figure 1: Comparison of retention time (9.9 min), exact mass (569.0789), and fragmentation spectrum of Capstone product B in the real sample (a) and the analysis standard (b).

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#### Environmental Forensics: Implementation of an interdisciplinary approach for the identification and allocation of PFAS sources in the context of environmental litigation

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#### 1. Introduction:

The presence of PFAS in our environment is now considered ubiquitous. In investigating the origin of contamination, identifying PFAS sources is complex due to the many primary, secondary, and tertiary sources of PFAS, migration pathways, and complex cycling behaviors.

- Example of a primary source: A production plant.
- Example of a secondary source: A wastewater treatment plant (WWTP) or a landfill.
- Example of a tertiary source: Sludge from a treatment plant spread on agricultural land.

These complexities make it challenging to identify the source of PFAS when they are found in the environment.

The proposed presentation focuses on the application of a robust and reliable approach in the context of environmental litigation at a site contaminated with PFAS in the United States. High concentrations of contaminants were detected in groundwater and surface water, affecting several drinking water wells.

#### 2. Methodology:

The approach deployed by Ramboll relied on the collection and analysis of multiple data sources:

a. Historical Data Analysis:

Conducting a detailed historical inventory to trace the evolution of practices and the use of products containing PFAS by the site.

b. Sampling and Testing:

Defining an investigation plan and conducting sampling of chimney residues, filtering materials, and dust to identify potential sources.

Discussions with laboratories to implement procedures adapted to the different matrices sampled.

c. Data Analysis:

Performing mass balance calculations, mapping the measured concentrations in different media (soil, sediments, groundwater), and modeling the transfer in groundwater.

d. Statistical Analysis:

Using statistical methods such as simple ratios, double ratios, and multivariate analyses (PCA, CHA) to establish PFAS profiles specific to sources from the acquired data.



#### 3. Results:

In this study, three major sources of PFAS were identified on the studied site, including off-site sources contributing to the contamination of drinking water wells. It was demonstrated that current site atmospheric emissions are not the main sources, with residues from previous operations being primarily responsible. The study enabled successful negotiations for the resolution of the dispute, reducing costs for our client through solid scientific evidence.

#### 4. Conclusion:

Ramboll's approach, supported by its technical expertise and innovation, proved effective in identifying and attributing PFAS contamination sources. This facilitated favorable negotiations for dispute resolution, dramatically reduced costs for our client, and implemented solutions based on rigorous scientific data.

#### In-field demonstration of FRED-PFAS: a novel, portable field sensor for rapid PFAS measurement onsite

#### Abstract

With over 50,000 potentially contaminated PFAS sites identified so far in North America (EWG, 2024), there is a strong need for advances in PFAS monitoring technologies to enable rapid, accurate and broadly accessible surveillance for PFAS identification, destruction, and remediation efforts. Traditional PFAS monitoring technologies utilize combustion ion chromatography (e.g., TOF) or liquid-chromatography mass-spectrometry (e.g., EPA 537.1 and 1633) techniques, which are expensive, require highly trained personnel, and are confined to analytical laboratory settings. This makes PFAS data difficult, slow, cumbersome, and expensive to acquire, with turnaround times ranging from weeks to months. With ever increasing site numbers and backlogs at analytical laboratory facilities, it is critical that industry has new tools to assess PFAS contamination, with on-site screening tools being particularly crucial.

FREDsense Technologies has developed such a screening tool: FRED-PFAS. The system utilizes a custom polymeric binding system capable of detecting the fluorocarbon chains that make PFAS so unique. This polymer produces a high level of fluorescence that decreases in a dose-dependent manner when exposed to PFAS compounds. When combined with a modified solid phase extraction (SPE) process, the system can reach ppt-level detection limits and has been demonstrated on multiple sample matrices, including AFFF rinsates, industrial wastewater, groundwater, and more. Significant comparisons against gold standard PFAS detection technologies, including TOF and LC-MS/MS (EPA 1633) were conducted at the laboratory and the field demonstration stages to verify the efficacy of this new PFAS screening technology.

This talk will highlight the technical validation of the FRED-PFAS system on a variety of samples. Additionally we will cover different use cases for rapid PFAS data generation and discuss the significant benefits that in-field analysis can provide. Finally, we will share data from a recent field pilot of the system completed in Q4 of 2024. During this pilot, FRED-PFAS was tested alongside samples analyzed by third-party laboratories using EPA Method 1633 and Total Organic Fluorine (TOF). The FRED-PFAS results correlated well with third-party analyses, even in complicated matrices, and produced same-day results as opposed to standard multi-week turnaround times.

Presenter:

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#### Assessment of PFAS Contamination in Wastewater: Innovative Approaches Using Passive Sampling and TOP Assay

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Per- and polyfluoroalkyl substances (PFAS) represent a broad class of organofluorine compounds widely used for their hydrophobic and oleophobic properties. Despite their typically low concentrations in aquatic environments, concerns are growing due to their persistence, toxicity potential, and ability to bioaccumulate. Although regulatory monitoring currently focuses on a defined list of 20 PFAS, thousands of other PFAS are potentially present, complicating the comprehensive characterization of contaminated environments. Moreover, in complex matrices such as urban or industrial wastewater, significant matrix effects can impair the effectiveness of conventional analytical methods.

In this context, passive sampling appears as a promising alternative. Deploying passive sampling devices in the water body for periods ranging from 3 to 15 days allows for in situ preconcentration of PFAS, resulting in lower limits of quantification. Thanks to this improvement, passive sampling becomes particularly relevant for mass flux evaluation: a station previously considered as a low or non-contributor based on grab sampling can reveal significant PFAS fluxes when assessed via passive sampling. This technique also offers enhanced temporal representativeness compared to traditional grab samples, which are limited by their instantaneous nature, and it provides in situ purification, thus reducing matrix effects during analysis.

Furthermore, for a more comprehensive characterization of environmental PFAS contamination, combining passive sampling with the TOP (Total Oxidizable Precursor) assay is a promising strategy. While the TOP assay is primarily applied to grab water samples, it enables the transformation of unknown PFAS precursors into detectable PFAS belonging to the regulatory list of 20 compounds. This provides an estimate of total PFAS content while benefiting from the advantages of passive sampling. Such a coupled approach—integrating both targeted analysis and precursor oxidation—offers a more complete understanding of contamination and supports improved assessment of environmental and health risks associated with PFAS.

To validate this combined approach, 11 urban wastewater monitoring stations were equipped with six DGT (Diffusive Gradient in Thin Films) passive samplers, in February 2025. Parallel grab samples were also collected to compare both sampling strategies. Each sample will be analyzed both before and after applying the TOP assay. This setup will allow for a comparative evaluation of the benefits and limitations of passive sampling versus conventional methods also when combined with the TOP assay. Additionally, the data will support the diagnostic assessment of PFAS contamination across the urban wastewater network.

Keywords: Passive sampling, TOP assay, Total PFAS, Metrology, Wastewater

## Managing groundwater PFAS pollution by combining hydrology and activated carbon water treatment

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VIVAQUA is a large drinking water operator in Belgium delivering more than 2 million people. The drinking water is produced from 70% groundwater and 30% surface water. Around 25 groundwater catchment sites are operated daily to deliver drinking water. A few years ago, we discovered a large PFAS groundwater pollution at two large catchment fields in the same area. Through intensive chemical monitoring, we studied and modelled the hydrogeological behavior of the PFAS in these groundwater wells and their activated carbon filtration at a large treatment plant. We also monitored many others chemical micropollutants to gain more insight into the origin of these pollutions. It appears that identifying the origin of PFAS groundwater pollution is not always straightforward and multiple PFAS origin can be considered. The objectives are to track the origin of the pollutions and to optimize the regeneration of activated carbon, in order to control the cost for removing these PFAS pollutions to produce drinking water complying with the European drinking water directive 2020/2184. Based on our study, some scenarios and their associated costs for other drinking water quality targets can be drawn up, as for example complying with for the sum of the four EFSA PFAS at 4 ng/L.

Keywords: Groundwater, Water treatment, Activated carbon, Hydrogeology, Cost

## L'ÉTERNITÉ S'ARRÊTE ICI Technologie exclusive de destruction totale des PFAS jusqu'aux fluorures

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#### Mercredi 18 juin 2025

08h30 Accueil des participants

#### **Présidents :**

• Dr. Christophe Rousselle, Agence Nationale de Sécurité Santé (ANSES) - Société Francophone de la Santé et de la Santé et de l'Environnement (SFSE) & Association pour la Recherche en Environnement (ARET), France

• Philippe Bodenez, Chef de service Santé, Environnement et Économie Circulaire - Direction Générale de la Prévention des Risques - Ministère de la Transition écologique, de l'énergie, du climat et de la prévention des risques, France

• Dr. Frank Karg, SFSE & ARET, Expert judiciaire, Directeur scientifique - HPC International, France & Allemagne

#### Évaluation des risques environnementaux et sanitaires, Toxicologie & Épidémiologie

#### 09h00

La présence des PFAS dans les ressources en eau de la ville de Lausanne : méthode d'analyse et résultats récoltés

Fereidoun Khajehnouri, Responsable du Laboratoire - Service de l'eau de Lausanne, Suisse

#### 09h30

Contamination de la biodiversité par les PFAS : Perspectives et outils émergents pour l'évaluation de l'exposition Maxime Louzon, Responsable du Pôle Écosystèmes - Envisol, France

#### 10h00

Evaluation des risques sanitaires et toxicologiques (EQRS & ARR) des PFAS concernant les chaines de bio-transformation des PFAS poly-fluorés vers les PFAS per-fluorés et application du Top Assay *Dr. Frank Karg, SFSE & ARET, Expert judiciaire, Directeur scientifique - HPC International, France & Allemagne* 

10h30 Pause-café & thé

#### 11h00

Émissions dans l'air, transport et intrusion de vapeur dans les bâtiments. Du schéma conceptuel au retour d'expérience sur les mesures de concentration de PFAS dans l'air intérieur/extérieur *Sylvie Traverse, Directrice de projet - Ginger Burgeap, France* 

#### 11h30

De l'AOF à l'identification des PFAS : Une approche intégrée Emiliano De Dominicis, Directeur de la R&D - Mérieux NutriSciences, Italie

#### 12h00

Mesure de 7 composés PFAS critiques dans le sérum humain en utilisant une préparation d'échantillon par SPE dispersive en pipette avant une analyse par LC-MS/MS *Kristen Schultz, Préparation des échantillons & surveillance de l'air, Chef de produit mondial - MilliporeSigma, Etats-Unis* 

#### 12h30

Déjeuner

#### 13h45

Évaluation quantitative des risques sanitaires liés aux PFAS sur le site d'une ancienne fabrique de papier : Enjeux et Défis Lucie Robin Vigneron, Directrice générale - HPC International, France

#### 14h15

L'eau douce est une ressource qui ne peut plus être considérée comme acquise - nous avons besoin d'une transformation circulaire, mais elle est compromise/mise en danger par les PFAS *Tobias Eriksson, Responsable du bureau européen - Ragn-Sells Group, Suède* 

#### 14h45

Niveaux sériques de substances perfluoroalkylées et polyfluoroalkylées en fonction de la durée de résidence aux États-Unis

*Eloïse Brosset, Doctorante - Centre de Recherche du Centre Hospitalier Universitaire de Sherbrooke (CRCHUS), Québec, Canada* 

15h15 Pause-café & thé

#### 15h45

Développement d'un modèle PBPK pour la souris femelle de sa naissance à la gestation et allaitement du souriceau : application à une exposition orale au PFOA *Albert Paré, Doctorant - Institut National de l'Environnement Industriel et des Risques (INERIS), France* 

#### Plans de gestion & objectifs de réhabilitation et traitements industriels

#### 16h15

Proposition de valeurs limites à l'émission pour les substances per- et polyfluoroalkylées (PFAS) à partir de l'étude des valeurs toxicologiques de référence (VTR) Azevedo Goncalves, Chargé du Suivi Environnemental - Groupe Séché, France

#### 16h45

Immobilisation des PFAS dans le sol : Quel est le temps nécessaire ? *Richard Stewart, Président - Rembind, Australie* 

#### 17h15

Caractérisation des PFAS dans les émissions atmosphériques : méthodes de surveillance et défis en matière de remédiation Jelle Hofman, Chercheur en R&D - Institut Flamand de Recherche Technologique (VITO), Belgique

#### 17h45

Rompre le cycle : Le pouvoir du charbon actif dans l'élimination et la destruction des PFAS *Johan Craeye, Directeur des Nouvelles Technologies - Desotec, Belgique* 

#### 18h15

Fin de la seconde journée



#### Wednesday, June 18, 2025

08:30 Welcoming participants

#### **Presidents:**

• Dr. Christophe Rousselle, National Agency for Health Safety (ANSES) - French-speaking Society for Health and Health and the Environment (SFSE) & Association for Environmental Research (ARET), France

 Philippe Bodenez, Head of Department, Health, Environment and Circular Economy - Risk Prevention Department - Ministry for Ecological Transition, Energy, Climate and Risk Prevention, France
 Dr. Frank Karg, SFSE & ARET, Court Expert, Scientific Director - HPC International, France & Germany

#### Environmental and Health Risk Assessments, Toxicology & Epidemiology

#### 09:00

The presence of PFAS in the water resources of the city of Lausanne: analysis method and results gathered

Fereidoun Khajehnouri, Laboratory Manager - Lausanne Water Department, Switzerland

#### 09:30

PFAS Contamination of Biodiversity: Insights and Emerging Tools for Exposure Assessment *Maxime Louzon, Ecosystems Division Manager - Envisol, France* 

#### 10:00

Human Health Risk Assessment via Toxicological Exposure Risk Quantification (HHRA & TERQ) concerning bio-transformation chains of poly-fluorinated PFAS to per-fluorinated PFAS and top assay application

Dr. Frank Karg, SFSE & ARET, Court Expert, Scientific Director - HPC International, France & Germany

10:30 Coffee & Tea break

#### 11:00

Air Emissions, transport and vapor intrusion in buildings. From conceptual diagram to feedback on indoor/outdoor air concentration measurements of PFAS *Sylvie Traverse, Project Manager - Ginger Burgeap, France* 

#### 11:30

From AOF to PFAS identification: An integrated approach Emiliano De Dominicis, R&D Director - Mérieux NutriSciences, Italy

#### 12:00

Measurement of 7 critical PFAS compounds from human serum utilizing dispersive in-pipette SPE sample prep prior to LC-MS/MS Analysis

*Kristen Schultz, Sample Preparation & Air Monitoring, Global Product Manager - MilliporeSigma, United Sates (USA)* 

#### 12:30

Lunch

#### 13:45

Human health risk assessment of PFAS on the site of an old paper manufacture: Stakes and challenges

Lucie Robin Vigneron, General Manager - HPC International, France

#### 14:15

Freshwater is a resource that can no longer be taken for granted – we need a circular transformation to take place, but it is compromised/endangered by PFAS *Tobias Eriksson, Head of EU office - Ragn-Sells Group, Sweden* 

#### 14:45

Serum levels of Per- and Polyfluoroalkyl Substances in relation to the length of residency in United States

Eloïse Brosset, PhD Student - Centre de Recherche du Centre Hospitalier Universitaire de Sherbrooke (CRCHUS), Québec, Canada

#### 15:15

Coffee & Tea break

#### 15:45

Development of a PBPK model for the female mouse from birth to gestation, lactation to offspring: application to a PFOA oral exposure

Albert Paré, PhD Student - French National Institute for Industrial Environment and Risks (INERIS), France

#### **Remediation Plans & Remediation Goals and Industrial Treatments**

#### 16:15

Proposal of emission limit values for per- and polyfluoroalkylated substances (PFAS) based on the study of toxicological reference values (TRVs) *Azevedo Goncalves, Head Mission for Environmental Monitoring - Séché Group, France* 

#### 16:45 PFAS Immobilisation in Soil: How long is long enough? *Richard Stewart, CEO - Rembind, Australia*

#### 17:15

Characterizing PFAS in air emissions: monitoring methods and remediation challenges Jelle Hofman, R&D Researcher - Flemish Institute for Technological Research (VITO), Belgium

#### 17:45

Breaking the Cycle: The Power of Activated Carbon in PFAS Removal and Destruction Johan Craeye, CTO - Desotec, Belgium

#### 18:15

End of second day

#### The presence of PFAS (ans TFA) in the water resources of the city of Lausanne : Analysis method and results collected

Dr. Fereidoun Khajehnouri, Chef de division « Contrôle de l'eau », Service de l'eau, Ville de Lausanne

Per- and polyfluoroalkyl substances (PFAS) are synthetic substances found in everyday objects. Due to their toxicological properties, regulations concerning these substances have become stricter in recent years. Regarding drinking water, the maximum allowable concentrations of PFAS set by various national and international organizations are progressively lower. The tightening of standards poses an analytical challenge. Indeed, these substances carry a significant risk of contamination, making their analysis at very low concentrations difficult. A large amount of data has been accumulated over the years. The analyses conducted focus on surface water, groundwater, water intended for consumption, and the water entering and leaving the Vidy wastewater treatment plant (WWTP). In water resources, PFAS concentrations are much lower than the maximum permissible levels in Switzerland and the European Union. As for WWTP samples, concentrations are much higher, but they are not regulated. The Vidy WWTP does not have a treatment stage specific to micropollutants, so no reduction is observed. In fact, PFAS concentrations are even significantly higher in the effluent, possibly due to the formation/transformation of PFAS during the treatment stages. Among all these samples from different sources, the PFAS with the highest concentrations are : PFHxA, PFOA, PFBA, PFOS, and PFPeA.

#### PFAS Contamination of Biodiversity: Insights and Emerging Tools for Exposure Assessment

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Keywords: biodiversity, aptameres, bioavailability, risks

Per- and polyfluoroalkyl substances (PFAS) are persistent organic pollutants with wide-ranging implications for biodiversity and ecosystem health. Their ubiquity in the environment, bioaccumulative properties, and resistance to degradation have led to significant concerns regarding their role in biodiversity loss and their intersection with the One Health concept, which emphasizes the interconnected health of humans, animals, and ecosystems. This presentation explores the impregnation of biodiversity by PFAS around the world, highlighting their contribution to the erosion of biodiversity and the cascading impacts on ecosystem health and services.

We discuss emerging perspectives in PFAS diagnostics and risk assessment, stemming from our ongoing research projects. The ADEME-funded ATOLIX project focuses on biosensors capable of identifying residual risks for freshwater aquatic organisms following process of degradation and elimination of PFAS cocktails. The MITI CNRS LUMIN-KISS project leverages aptamer-based biosensors for rapid detection and diagnosis of PFAS families in various environmental matrices and organisms. Additionally, the ADEME-funded BORG project develops innovative tools to evaluate the environmental bioavailability and oral bioaccessibility of PFAS in soils, targeting exposure pathways for humans, wildlife, and livestock.

Together, these projects aim to bridge critical knowledge gaps, enabling a more accurate evaluation of PFAS risks and their ecological and health implications. By integrating advanced diagnostic tools with the One Health framework, our work paves the way for more effective strategies to mitigate PFAS contamination and preserve biodiversity.



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#### Evaluation des risques sanitaires et toxicologiques (EQRS & ARR) des PFAS concernant les chaines de bio-transformation des PFAS poly-fluorés vers les PFAS per-fluorés et application du Top Assay /

#### Human Health Risk Assessment via Toxicological Exposure Risk Quantification (HHRA & TERQ) concerning Bio-transformation Chains of poly-fluorinated PFAS to per-fluorinated PFAS and Top Assay Application.

Frank KARG<sup>1</sup> (Géochimiste en Environnement, Evaluateur des Risques et Expert Judiciaire),

Jean-François HEILIER<sup>2</sup> (Toxicologue) / Sylvaine RONGA-PEZERET<sup>3</sup> (Toxicologue) /

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#### 1. Introduction

Les PFAS : per- & poly-fluoro-alkyl substances sont devenus progressivement depuis des années 1960 un problème environnemental majeur, également pour la Santé Publique du fait de leurs effets sur la santé humaine et leur application multiple et vaste (historique et actuelle) par exemple dans les détergents, des mousses anti-incendies (AFFF), le traitement et l'imperméabilisation des textiles, papiers, cartons et bois, pour la galvanisation, dans les peintures et laques, dans les éléments électroniques, certaines huiles hydrauliques et pour la production des fluoro-polymères.

D'autres sources de pollution de l'environnement (air, eaux, sols et aliments) sont les lixiviats des anciennes décharges et les épandages des certaines boues de stations d'épurations sur des terrains agricoles voire les émissions des incinérateurs encore mal caractérisées. Cette menace environnementale et pour la santé publique des PFAS est peu à peu prise en compte en Europe depuis les années 2010 et la problématique médiatisée et de plus en plus gérée et réglementée depuis 2022. Les PFAS se retrouvent aujourd'hui dans les sols, les eaux souterraines, les aliments et l'eau potable ainsi que dans les gaz du sol et l'air ambiant Entre 9 000 à 12 000 polluants synthétiques de PFAS ont été produits.

Les PFAS polymères du type « Teflon » (ou PFTE) ne disposent pas une bonne biodisponibilité et sont considérés comme beaucoup moins toxiques (voir non-toxiques) que les PFAS monomères, cependant leur fabrication et élimination peut engendrer des rejets de PFAS toxiques. La toxicité des es PFAS monomères est la plus étudiée, du moins pour quelques-uns d'entre eux et fait l'objet du chapitre présenté ci-après. Au total, trente-trois familles de PFAS monomères existent, comme par ex. les acides carboxyliques ou sulfoniques per-fluorés ou divers PFAS poly-fluorés (ou « Precurseurs ») qui peuvent être (bio-)transformés en PFAS perfluorés stables.

Les PFAS sont connus notamment pour leurs effets toxicologiques de perturbateurs endocriniens, d'hépatotoxicité, d'immunotoxicité, sur le développement des fœtus et pour certains, la cancérogénicité (PFOA...). Dans le cas des expositions aux PFAS, les évaluations des risques toxicologiques et sanitaires (EQRS) sont nécessaires afin de juger des besoins en mesures de gestion de réduction des



expositions.

#### 2. Toxicité des PFAS :

L'OCDE (OCDE, 2021 [1]) définit les PFAS comme des substances contenant au moins un méthylène totalement fluoré sans être lié à un atome d'hydrogène ou à un autre halogéné. Les PFAS constituent donc un large groupe de congénères qui diffèrent par la longueur de leur chaîne fluor-carbonée, la présence d'une chaine alkylénique (poly-fluorée) et la fonctionnalisation de leur terminaison hydrophile (carboxylique ou sulfonique par exemple). Par contre, la simple définition des PFAS par « *un méthylène totalement fluoré sans être lié à un atome d'hydrogène ou à un autre halogéné* » est couramment critiquée, car elle concerne également certains médicaments (monomères), fluor-polymères inertes (comme des pièces médicales d'implantation, etc.) et d'autres produits plutôt bénéfiques en industrie aérospatiale, automobile etc. Uniquement les PFAS monomères toxiques sont visées par les évaluations quantitatives des risques sanitaires et toxicologiques (EQRS).

Parmi les congénères perfluorés, possédant une fonctionnalisation carboxylique (PFCA) ou sulfonique (PFSA), on distingue les composés à longue chaine (PFCA avec plus de 7 atomes de carbone (p.ex. PFOA et PFNA) et PFSA avec plus de 5 atomes de carbone (p.ex. PFOS et PFHxS). Les composés à chaines courtes (p.ex. PFBA) et à chaines très courtes (p.ex. TFA, acide trifluoro-acétique) complètent le tableau.

Les caractéristiques des congénères influencent leurs paramètres toxicocinétiques mais également leurs propriétés toxico-dynamiques, à savoir notamment l'interaction avec des récepteurs nucléaires.

Les effets délétères associés aux PFAS sont nombreux et touchent des cibles biologiques diverses . Les données toxicologiques sont historiquement obtenues avec des composés à chaines longues. Les récentes évaluations réalisées par des organismes nationaux ou supranationaux (ATSDR, 2021 [2] ; IARC, 2016 [3]; US-EPA, 2016a,b [4, 5] ; Zahm et al., 2023 [6], US-EPA 2024a [7], 2024b [8]) indiquent que certains PFAS à longue chaine sont (sur base de données souvent issues de l'étude du PFOA et du PFOS) cancérigènes, reprotoxiques, immuno-toxiques et perturbateurs endocriniens. En outre, ils exercent une toxicité sur le métabolisme des lipides.

#### a. Effets non-cancérigènes (effets systémiques):

Les PFAS sont hépatotoxiques chez le rongeur. La toxicité hépatique, mise en évidence essentiellement pour des composés à longues chaines, se caractérise par une hypertrophie hépatocellulaire due à une augmentation du nombre et de la taille des peroxysomes. Les PFAS sont susceptibles d'augmenter la α-oxydation des acides gras. D'une manière générale les PFAS perturbent le métabolisme lipidique. De récentes publications montrent une association positive entre les taux circulant de PFOS et les taux de cholestérol total et de cholestérol non HDL (« mauvais cholestérol ») (Ho, 2022 [9]; Rosen, 2022 [10]).

Dans l'ensemble, les effets métaboliques rapportés dans les études disponibles sont des indicateurs potentiels d'altérations induites par le PFHxS dans la fonction hépatocytaire, qui pourraient éventuellement conduire à un métabolisme hépatique anormal et à une accumulation d'acides gras entraînant une stéatose hépatique. L'accumulation hépatique excessive et prolongée d'espèces lipidiques lipotoxiques (par exemple, le cholestérol libre et les acides gras libres) (US-EPA-IRIS, 2025 [69], Younossi et Henry, 2024 [74] ; M et al., 2024 [75]) est associée à une stéatose hépatique, à la promotion de la lipotoxicité, aux réponses pro-inflammatoires, à la cytotoxicité et à la progression de la stéatose hépatique vers la stéatohépatite. L'exposition au PFHxS (0 et 60 à 110 mg/kg-jour) pendant





12 semaines a augmenté les taux d'ARNm hépatique de la cytokine pro-inflammatoire (IL-1β) et du facteur pro-fibrogène Col1α (He et al., 2022 [78]). L'IL-1 β et Col1 α jouent un rôle dans la perte des fonctions hépatiques et la progression de la stéatose hépatique vers la stéatohépatite et les lésions fibrotiques (US-EPA-IRIS, 2025 [69], Vesković et al., 2024 [76]; Sultan et al., 2017 [77]; He et al., 2022 [78]).

Le TFA présente également une toxicité hépatique mise en évidence dans plusieurs études par une perturbation des enzymes hépatiques (ASAT, ALAT, phosphatases alcalines). Dekant et Dekant (2023) [11] signent une revue récente de la littérature sur le sujet.

L'immunotoxicité des PFAS a été étudiée sous différents aspects, notamment l'hypersensibilité, la réponse auto-immune et la réduction de la production d'anticorps (niveaux d'anticorps) (Bil & Ehrlich et al., 2023 [12]). Dans ce contexte, l'EFSA (EFSA, 2020 [13]) a défini une dose hebdomadaire tolérable (TWI : total weekly intake) en se basant sur l'étude d'Abraham et al. (Abraham, 2020 [14]). Cette recherche a révélé des associations significatives entre les concentrations sériques de PFOA (mais non de PFOS) et les taux ajustés d'anticorps contre Haemophilus influenzae type b (Hib, r = 0.32), contre l'anatoxine tétanique (Clostridium tetani, r = 0.25) et l'anatoxine diphtérique (Corynebacterium diphtheriae, r = 0.23). Après avoir converti ces données à l'aide d'un modèle PBPK, l'EFSA [13] a fixé une TWI de 4,4 ng/kg de poids corporel par semaine pour la somme des PFOA, PFNA, PFHxS et PFOS.

L'ATSDR (ATSDR, 2021 [2]) a retenu des effets reprotoxiques, spécifiquement liés au développement, pour établir les niveaux minimaux de risque (MRL) de trois composés : le PFOA, le PFOS et le PFNA. Les effets critiques identifiés ont été observés chez la souris et incluent, pour le PFOA, une altération du squelette (Koskela, 2016 [15]), pour le PFOS, une réduction du poids des portées et un retard dans l'ouverture des yeux (Luebker, 2005 [16]), et pour le PFNA, une diminution du poids des portées ainsi qu'un retard dans le développement (Das, 2015 [17]). Pendant la grossesse et le développement précoce, les perturbations de la fonction thyroïdienne par les PFAS et particulièrement par le PFHxS peuvent avoir des répercussions sur la croissance normale et le développement neurologique de la progéniture (US-EPA-IRIS, 2025 [69], Zoeller et Rovet, 2004 [70]; Y et al., 2024 [71]; Street et al., 2024 [72]; Stagnaro-Green et Rovet, 2016 [73]).

Les MRL, calculés pour une durée d'exposition intermédiaire, sont respectivement fixés à 3, 2 et 3 ng/kg de poids corporel par jour pour le PFOA, le PFOS et le PFNA.

Concernant le PFHxS, l'ATSDR (ATSDR, 2021 [2]) a sélectionné un autre effet critique, illustrant la diversité des dangers liés aux PFAS : son impact sur la thyroïde, en tant que perturbateur endocrinien. L'effet critique, identifié par Butenhoff et al. (Butenhoff, 2009 [18]), est l'hyperplasie/hypertrophie de l'épithélium des follicules thyroïdiens chez le rat. Sur cette base, l'ATSDR a calculé un MRL oral intermédiaire de  $2 \cdot 10^{-5}$  mg/kg de poids corporel (pc) par jour. Malgré cela, l'ATSDR souligne, à travers la revue de nombreuses études épidémiologiques, que la relation entre les PFAS et les perturbations thyroïdiennes reste controversée. Les observations épidémiologiques ont mis en évidence une réduction des poids de naissance des nouveau nées associée à une sur-exposition aux PFAS (Engström, 2022 [19]).

#### b. Effets cancérigènes :

L'IARC (agence de l'OMS spécialisée dans l'évaluation des effets cancérigènes) avait classé en 2016 [20] le PFOA (acide perfluoro-octanoïque) en catégorie 2B (L'agent est peut-être cancérigène pour l'Homme) sur la base d'éléments de preuve limités tant chez l'Homme (cancers des testicules et du rein) que chez l'animal.



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L'IARC [20] prenait en considération deux études épidémiologiques relatives à l'exposition professionnelle (par inhalation) au PFOA : Steenland et Woskie (Steenland et Woskie, 2012 [21]) et Raleigh et al. (Raleigh et al., 2014 [22]). L'étude de Raleigh et al. (2014 [22]) ne conclut pas à l'existence d'une association entre les effets critiques observés et l'exposition au PFOA (en l'occurrence son sel d'ammonium). Toutefois, l'IARC considérait que, dans cette étude, l'incidence et la mortalité du cancer de la vessie sont associées à l'exposition au PFOA (sel d'ammonium), bien qu'aucun de ces paramètres ne conduise à une association statistiquement significative.

En 2023, L'IARC (Zahm et al., 2023 [6]) a revu sa position et classé le PFOA comme une substance cancérigène pour l'homme (Groupe 1). La classification du PFOA repose aujourd'hui sur des preuves suffisantes de cancers chez l'animal (exposé via l'alimentation) (foie et pancréas) et sur des preuves fortes relatives aux mécanismes pouvant être mis en œuvre chez l'Homme. Le lien entre le PFOA et l'apparition de carcinome des cellules rénales et de cancer des testicules reste limité. Le PFOS a été classé comme potentiellement cancérigène pour l'homme (Groupe 2B).

Au vu des processus industriels mettant en œuvre ces composés, il n'est pas aisé d'établir dans les études épidémiologiques la distinction entre exposition au PFOS/PFOA et autres PFAS.

En 2016, l'US-EPA (agence américaine en charge de l'environnement), dans un health advisory PFOA (US-EPA, 2016a [4]) pour l'eau de boisson, proposait un OSF (Oral Slope Factor / estimateur quantitatif du risque de cancers) de 0,07 (mg/kg  $pc \cdot j$ )<sup>-1</sup> dérivé de l'étude de Butenhoff (Butenhoff, 2012 [23]) analysant les tumeurs dans les cellules de Leydig chez le rat mâle. En 2024, (huit ans après), l'US-EPA (US-EPA, 2024a [7]) propose un nouvel OSF de 0.0293 (ng/kg p.c.·j)<sup>-1</sup> soit 29,300 (mg/kg p.c.·j)<sup>-1</sup>. Ce nouvel OSF est basé sur l'apparition de carcinome des cellules rénales mis en évidence par Shearer et al. (2021) [24]. Le nouvel OSF est près de 500.000 fois plus précautionneux.

Pour le PFOS, les données de l'étude de Buttenhoff et al. [23] ont notamment été utilisée par l'US-EPA pour produire un « Oral Slope-Facteur » : OSF de 39,5 (mg/kg p.c.·j)<sup>-1</sup>.

Le TFA n'apparaît ni mutagène (le test de Ames avec et sans activation métabolique est globalement négatif), ni clastogène. Le TFA n'est pas classé cancérogène.

#### c. Mécanismes d'action

#### i. Mécanisme médié par le récepteur PPAR-alpha

La principale cible des PFAS est le récepteur PPAR-  $\alpha$  (peroxisome proliferator-activated receptor – alpha) chez l'animal de laboratoire (rongeurs). Toutefois, il est nécessaire de signaler que d'une manière générale, les rats et les souris possèdent des récepteurs PPAR- $\alpha$  plus sensibles aux PFAS que ceux des primates (humains et non-humains). Les différences entre espèces s'expliquent notamment par l'expression du récepteur dans les tissus.

Le récepteur PPAR- $\alpha$  est un récepteur nucléaire présent dans de nombreux organes caractérisés par un taux élevé de catabolisme des acides gras (foie, rein, cœur ...). Ses ligands endogènes sont principalement les acides gras, ainsi que, dans une moindre mesure, certains dérivés des eicosanoïdes... Les gènes activés par le récepteur PPAR- $\alpha$  (après translocation nucléaire) sont responsables, entre autres, de l'absorption cellulaire des acides gras et de leur oxydation métabolique (Boelsterli, 2007 [25]; Landry, 2019 [26]).

Les congénères PFOA, PFOS, PFUnA, PFHpA et PFDoDA activent le récepteur PPAR-alpha.



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Les congénères sulfoniques sont réputés moins puissants que les congénères carboxyliques. La puissance augmente en fonction de la longueur de la chaîne fluorocarbonée.

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Les gènes dont l'expression est augmentée appartiennent au métabolisme des acides gras, à la biosynthèse des peroxysomes et à la structure et à l'organisation des protéasomes. Les gènes dont l'expression est diminuée interviennent notamment dans la réponse inflammatoire.

#### ii. Le mécanisme médié par d'autres voies

Les mécanismes indépendants de l'activation de PPAR-alpha peuvent être médiés par d'autres récepteurs nucléaires tels PPAR-gamma, CAR (constitutive androstane receptor) ou ER-alpha. Des réactions de stress oxydant mises en évidence en particulier par la présence de 8-OH-dG peuvent résulter soit d'une activation via Nrf-2 ou via PPAR-alpha.

#### d. Toxicocinétique

L'EFSA (2020 [13]) a réévalué la toxicocinétique des PFAS, en particulier chez l'Homme. L'exposition humaine est principalement due à l'ingestion d'aliments contaminés (eau de boisson contaminée, produits de la mer, etc.) ainsi que de l'inhalation de l'air intérieur. Les substances PFOS et PFOA sont majoritairement absorbées par voie orale, cette voie d'exposition étant plus significative que l'inhalation ou la voie transdermique (Sunderland, 2019 [27]). Dans certains cas, la modélisation de la toxicocinétique nécessite l'utilisation de logiciels PBPK.

Pour le PFOA et le PFOS, la distribution dans l'organisme se caractérise par une liaison à l'albumine plasmatique. Ces composés ne sont pas connus pour être métabolisés. Leur élimination se fait par les voies biliaire et urinaire. Par exemple, le PFOA subit une réabsorption tubulaire via des transporteurs d'anions organiques (OAT, pour "organic anion transporters"), tandis que la réabsorption biliaire joue également un rôle majeur. La demi-vie des PFOS et PFOA se mesure en années. Les données concernant les composés à chaînes courtes et ultra-courtes laissent supposer qu'ils présenteraient une demi-vie bien plus courte (quelques jours) (Fenton, 2021 [28], Boutonnet, 1999 [29]). Concernant les composés poly-fluorés comme le 8 :2 FTOH, Nabb et al. (Nabb et al., 2007 [30]) ont démontré à l'aide de microsomes humains la métabolisation du 8 :2 FTOH en composés perfluorés. Les observations de Nilsson et al. (Nilsson et al., 2013 [31]) suite à l'exposition de travailleurs fartant des skis **avec** de la cire contenant du 8 :2 FTOH confirment les résultats expérimentaux.

#### 3. Valeurs Toxicologiques de Référence (VTR) :

Les valeurs présentées dans ce guide sont issues des rapports rédigés par des institutions nationales et supra-nationales (cf. Tableau 1 à 3) :

| Tableau 1 : Organismes proposant des VTR pour les PFAS |   |  |  |  |  |
|--|---|--|--|--|--|
| ANSES  | Agence nationale de sécurité sanitaire de l'alimentation, de l'environnement et du travail (France) |  |  |  |  |
| ATSDR  | Agency for Toxic Substances and Disease Registry (États-Unis)                                       |  |  |  |  |
| EFSA   | European Food and Safety Authority  |  |  |  |  |
| IRIS   | Integrated Risk Information of Substances (États-Unis)  |  |  |  |  |



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| UBA            | Umweltbundesamt (Allemagne)   |
|----------------|---|
| BfR            | Bundesintitut für Risikobewertung (Allemagne)   |
| ОЕННА          | Office of Environmental Health Hazard Assessment (États-Unis)                                   |
| WHO            | World Health Organization   |
| RIVM           | Netherlands Environmental & Health Institute (Pays-Bas)   |
| MDHHS          | Michigan Department of Health and Human Services, Division of Environmental Health (États-Unis) |
| TCEQ           | Texas Commission on Environmental Quality (États-Unis)  |
| NJ-<br>DWQIHES | New Jersey Drinking Water Quality Institute Health Effects Sub-committee (États-Unis)           |
| SLU            | Swedish University of Agricultural Science (Suède)  |

Il peut exister par ailleurs, dans la littérature scientifique des « Points of Departures (POD) » et VTR plus récentes ou plus spécifiques. Le choix des VTR constitue une étape importante de l'EQRS car leur évolution est rapide en ce qui concerne les PFAS. Par ex., l'ANSES a publié en 2017 un guide PFAS comportant certaines VTR pour les PFAS (ANSES, 2017 [32]). Aujourd'hui, les VTR publiées sont devenues beaucoup plus sévères, comme le montre l'évolution des VTRs publiées par l'EFSA depuis 2015 [33] (cf. Tableau 2).

| Tableau 2 : Évolution des VTR orales pour des PFAS<br>publiées par l'EFSA de 2005 à 2020 (EFSA 2005 [33] et 2020 [13]) |                   |              |                              |  |  |  |
|--|-------------------|--------------|------------------------------|--|--|--|
| Année de<br>publication  | Voie d'exposition | Substance(s) | VTR                          |  |  |  |
| 2020   | Ingestion         | Σ4 PFAS      | TWI = 4,4 ng/kg pc ⋅ semaine |  |  |  |
| 2018   | Ingestion         | PFOA         | TWI = 6 ng/kg pc · semaine   |  |  |  |
| 2018   | Ingestion         | PFOS         | TWI = 13 ng/kg pc·semaine    |  |  |  |
| 2005   | Ingestion         | PFOA         | TDI = 1500 ng/kg pc∙jour     |  |  |  |

Il est important de prendre en compte que les administrations de certains Etats américains sont très avancés dans la recherche toxicologique et la publication de VTR concernant les PFAS, du fait de la forte présence sur leur territoire d'industries chimiques et pétrolières (avec une utilisation historique importante de mousses anti-incendie) ainsi que de vastes décharges industrielles (avec les pollutions environnementales associées). Il s'agit notamment des états du Texas (TCEQ, 2023 [34]), du Michigan (MDHHS, 2019 [35]) et de New Jersey (NJ-DW-QI, 2017 [36]). Ces VTR sont souvent les bases des publications de VTR pour les PFAS de l'US-EPA.

Une Dose Hebdomadaire Tolérable (DHT) de 4,4 ng/kg pc·semaine (ou une Dose Journalière





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Tolérable (DJT) de 0,63 ng/kg pc·j) pour la somme de 4 PFAS (PFOA, PFOS, PFNA et PFHxS) a été publiée par l'**EFSA**, le 17/09/2020 [13]. En 2020, des **facteurs d'équivalence de toxicité** (RFP : Relative Potency Factors) par rapport au PFOA ont aussi été publiés par Bil et al. (2020) [37], 2021 [47] et 2023 [12] pour tenir compte de la toxicité de mélanges de PFAS.

Afin d'assurer le bon choix des VTR des PFAS, il est recommandé d'appliquer des critères de choix scientifiques et non des critères nationaux. Le tableau 3 montre des critères de choix de VTR applicables, afin de prendre en compte les meilleures connaissances toxicologiques concernant les relations de dose à effet des PFAS.

| Tableau 3 : Critères de choix qualitatifs des VTR applicables dans les cas de présence de<br>plusieurs VTR (F. Karg, 2022a,b – 2024a-g [41 - 46]) |   |   |  |  |           |  |
|---|---|---|--|--|-----------|--|
|   | Toxicological Poforonco Doso (TPD)  |   | Appreciat  | ion  |           |  |
| N°  | Choice Criteria   | Favorable   | Correct  | Not<br>favorable   | Exclusion |  |
| 1   | Variability of indicated TRD  | (+/- 0%)  | ≤(+/-30%)  | > (+/-30%)   |           |  |
| 2   | Class (potential) Carcinogenic:<br>EC: Class 3<br>US-EPA: Class B2, C / IARC: Group 1 | 3 Organisms :<br>CE, US-EPA,<br>IARC, etc.                  | 2 organisms  | 1 organism   |           |  |
| 3   | Several Organisms shows similar TRD (+/-50%)  | 3 organisms   | 2 organisms  | 1 organism   |           |  |
| 4   | Age of base Study   | ≤15 y   | 15-25 у  | > 25 y   |           |  |
| 5   | Mechanistic toxicological basement<br>Study (for ex. Genotoxicity)                    | Epidemiology  | In vivo  | In-Vitro/In-<br>sillico  |           |  |
| 6   | Basement Study: Klimisch Quality<br>Criteria  | Class 1   | Class 2  | Class 3  | Class 3   |  |
| 7   | Verified Purity of Compound   | Yes   | < 95%  | No   |           |  |
| 8   | Excipient potentially toxic   | No  |  | Yes  |           |  |
| 9   | Presence of population without exposure (test witness)                                | Yes   |  | No   |           |  |
| 10  | General Quality Criteria (Klimisch) of toxicological effect studies                   | Standardized<br>Study (OCDE,<br>UE, US-EPA,<br>FDA, etc.)   | Standardized<br>Study without<br>Details, but<br>correctly<br>documented | Document<br>insufficient<br>for<br>evaluation,<br>systematic<br>deficiencies |           |  |
| 11  | Point of Departure (POD)  | Quantified<br>Epidemiological<br>Data, BMLD,<br>etc. (PBPK) | NOAEL<br>sensitive<br>NOAEL  | LOAEL<br>sensitive,<br>LOAEL,<br>Other                                       |           |  |
| 12  | Uncertainty (or Assessment) Factors   | 1 – 100   | > 100 - 1000   | > 1000 -<br>10000  | > 10000   |  |
| 13a   | Transpositions: Between Exposure<br>Pathways  | No  |  | Yes  |           |  |
| 13b   | Transpositions: Animal to Human   | No  | Yes  |  |           |  |
| 13c   | Transpositions: From in-Vitro   | No  |  | Yes  |           |  |
| 13d   | Transpositions: From in-Sillico   | No  |  | Yes  |           |  |
| 14  | Study time-representatively   | ≥ chronic (><br>180 d)                                      | Sub-chronic<br>(90 d) to<br>chronic (180d)                               | < subchronic<br>(< 90 d)   |           |  |
| 15  | Integration of bio-disponibility / Bio-<br>resorption capacity (ex.: DIN 19738)       | Yes   | Not known<br>(100%)  | Known, but<br>not<br>considered  |           |  |



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L'application de ces critères de choix, fin 2024, conduit à sélectionner, par ex., les VTR indiquées dans le Tableau 4 :

| Tableau 4 (1/2) : Exemple de choix des VTR en 2024pour les PFAS en appliquant des critères du tableau 3(E. Karg et al. 2023; 2024; [28, 46]) |               |   |                                |                                    |  |                                       |       |   |   |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
|--|---------------|---|--------------------------------|------------------------------------|--|---------------------------------------|-------|---|---|-----------|----|------------|--------------------------------|-------------------------|---|--------------------|--------------------------|----------------------------------|--------------------|--------------------|
| Subst.   | CAS<br>Nr.    | Cancero-<br>genic /<br>not<br>cancero-<br>gonic | Chronic<br>Exposure<br>pathway | toxicologica<br>Target<br>organ    | l value<br>Value                                   | Testing<br>Species /<br>Study<br>Type | Sigle | Security<br>Factor<br>& Origin              | Organi-<br>sation                                     |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
| PFBA   | 375-          | NC  | oral                           | Hepatic                            | 1 µg/kg/d  | Rate                                  | RfD   | POD <sub>HED</sub> / 900                    | TCEQ 2023<br>US-EPA<br>IRIS 2022                      |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
|  | 22 4          |   | inhalation                     | Hepatic                            | 3,5 µg/m³  | Rate                                  | RfC   | from oral<br>value                          | TCEQ 2023   |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
| PFPeA  | 2706-<br>90-3 | NC  | oral                           | Hepatic                            | 0,5<br>µg/kg/d                                     | Rate                                  | RfD   | POD <sub>HED</sub> / 90                     | TCEQ 2023   |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
| PFHxA  | 307-<br>24-4  | NC  | oral                           | Hepatic                            | 0,5<br>µg/kg/d                                     | Rate                                  | RfD   | POD <sub>HED</sub> / 90                     | TCEQ 2023<br>US-EPA<br>IRIS 2023                      |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
| PFHpA  | 375-<br>85-9  | NC  | oral                           | Hepatic                            | 25<br>ng/kg/d                                      | Rate                                  | DJT   | Extrapolation<br>of DJT of<br>Health Canada | ANSES<br>2017<br>TCEQ 2023                            |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
|  |               |   |                                | Hematologic                        | 0,86<br>ng/kg/d                                    | Rate                                  | TDI   | BMDL 5                                      | UBA 2023<br>BFR &<br>EFSA 2018                        |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
|  |               |   | oral                           | Hepatic,<br>Mammar,<br>Hematologic | 12<br>ng/kg/d                                      | Mice                                  | RfD   | LOAEL /<br>(81 * 300)                       | TCEQ 2023   |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
| PF0A 335-  | 335-<br>67-1  | 335-<br>67-1                                    | 335-<br>67-1                   | 335-<br>67-1                       | 335-<br>67-1                                       | 335-<br>67-1                          | . NC  | 35-<br>7-1                                  | NC  | No        | NO | NC         | 35-<br>7-1                     |                         | Immune,<br>developmental<br>and<br>cardiovascular | 0,03<br>ng/kg/d    | Epidemiologic            | RfD                              | Several<br>studies | US-EPA<br>2024 [7] |
|  |               |   | inhalation                     | Hepatic                            | 4,1 ng/m <sup>3</sup>                              | Rate                                  | RfC   | NOAEL /<br>(81 * 3 000)                     | TCEQ 2023   |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
|  | С             |   |                                | Testicular<br>tumors               | 2,52 x 10 <sup>-6</sup><br>(ng/kg/d) <sup>-1</sup> | Epidem-<br>iologic                    | SF    | -   | New Jersey<br>2017                                    |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
|  |               |   |                                |                                    |  |                                       |       |   |   |           | C  | С          | oral                           | Renal Cell<br>Carcinoma | 0,0293<br>(ng/kg/d) <sup>-1</sup>                 | Epidem-<br>iologic | SF                       | -                                | US-EPA<br>2024 [7] |                    |
| PFNA   | 375-<br>95-1  | NC  | oral                           | Hematologic                        | 2,5<br>ng/kg/d                                     | Mouse                                 | RfD   | NOAEL / 300                                 | US-EPA<br>IRIS 2019 /<br>New<br>Hampshire<br>DES 2019 |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
|  |               |   |                                |                                    |  |                                       |       |   |   |           |    | inhalation | Lung,<br>respiratory<br>system | 28 ng/m <sup>3</sup>    | Rate  | RfC                | NOAEL /<br>(81 * 30 000) | US-EPA<br>IRIS 2019<br>TCEQ 2023 |                    |                    |
|  |               |   | oral                           | Hepatic                            | 15<br>ng/kg/d                                      | Rate                                  | RfD   | LOAEL /<br>(81 * 1 000)                     | TCEQ 2023   |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
| PFDA   | 335-<br>76-2  | NC  | oral                           | Immune /<br>developmental          | 0,002<br>ng/kg/d                                   | Rate                                  | RFD   | BMDL / 30                                   | US-EPA<br>IRIS 2024                                   |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
|  |               |   | inhalation                     | Hepatic                            | 53 ng/m <sup>3</sup>                               | Rate                                  | RfC   | from oral<br>value                          | TCEQ 2023   |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
| PFBS   | 375-          | NC  | oral                           | Hematologic                        | 1,4<br>µg/kg/d                                     | Rate                                  | RfC   | NOAEL /<br>(142*300)                        | TCEQ 2023   |           |    |            |                                |                         |   |                    |                          |                                  |                    |                    |
|  | 73-5          |   |                                | inhalation                         | anu renai  | 4,9 µg/m <sup>3</sup>                 | Rate  | RfC   | from oral<br>value                                    | TCEQ 2023 |    |            |                                |                         |   |                    |                          |                                  |                    |                    |



| Tableau 4 (2/2) : Exemple de choix des VTR en 2024<br>pour les PFAS en appliquant des critères du tableau 3<br>(F. Karg et al. 2023a – 2024g [38 - 46]) |               |   |                             |               |                                  |                                       |       |  |  |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |
|---|---------------|---|-----------------------------|---------------|----------------------------------|---------------------------------------|-------|--|--|------|--------------|------|---|--|------------------------|------------|----------|------------------------------------|-----------|-----|-----------------------------------|-----------|
| Subst.  | CAS<br>Nr.    | Cancero-<br>genic /<br>not<br>cancero-<br>genic | Chronic toxicological value |               |                                  | Testing<br>Species /<br>Study<br>Type | Sigle | Security<br>Factor<br>& Origin   | Organi-<br>sation  |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |
| PEHyS   | 355-          | NC  | oral                        | Hematologic   | 3,8<br>ng/kg/d                   | Rate                                  | RfC   | LOAEL /<br>(263*300)   | TCEQ 2023  |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |
|   | 46-4          | No  | inhalation                  | and thyroïdal | 13 ng/m <sup>3</sup>             | Rate                                  | RfC   | from oral<br>value   | TCEQ 2023  |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |
| PFHpS   | 375-<br>92-8  | NC  | oral                        | Hepatic       | 0,43<br>ng/kg/d                  | Rate                                  | TDI   | Potency<br>Factor :<br>0,6-2   | UBA 2020,<br>EFSA 2018,<br>BfR 2018                          |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |
|   | 1763-<br>23-1 |   |                             | Hepatic       | 1,86<br>ng/kg/d                  | Monkey                                | TDI   | NOAEL  | UBA 2020<br>BfR & EFSA<br>2018                               |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |
| PFOS 1763-<br>23-1  |               | NC  | NC                          | NC            | NC                               | NC                                    | NC    | NC   | NC   | NC   | 763-<br>23-1 | oral | Decreased<br>birth weight<br>and increased<br>cholesterol | 0,1<br>ng/kg/d   | Human<br>epidemiologic | RfD        | POP / 10 | US-EPA<br>2024                     |           |     |                                   |           |
|   |               | 23-1  | 23-1                        | 23-1          | 23-1                             | 23-1                                  | 23-1  | 23-1   |  |      |              |      | inhalation  | Thyroïdal,<br>neurological<br>and fœtal<br>development | 81 ng/m <sup>3</sup>   | Rate       | RfC      | from oral<br>value<br>(23 ng/kg/j) | TCEQ 2023 |     |                                   |           |
|   |               | С   | oral                        | Hepatic       | 39,5<br>(mg/kg/d) <sup>-1</sup>  | Rate                                  | SF    | -  | US-EPA<br>2024   |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |
| PEOSA   | 754-          | /54- NC   | oral                        | ral Mammary   | 12<br>ng/kg/d                    | Mice                                  | RfD   | As PFOA:<br>NOAEL /<br>(81 * 300)  | TCEQ 2023  |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |
|   | 91-6          | 91-6  | 91-6                        | 91-6          | 91-6                             | 91-6                                  | 91-6  | 91-6   | 91-6   | 91-6 | 91-6         | 91-6 | 91-6  | 91-6   | 1-6                    | inhalation | glands   | 4,1 ng/m <sup>3</sup>              | Rate      | RfC | As PFOA<br>NOAEL/<br>(81 * 3 000) | TCEQ 2023 |
| 6:2-<br>FTOH  | 647-<br>42-7  | NC  | oral                        | Hepatotoxic   | 43<br>ng/kg/d                    | Rate                                  | RfD   | RPF based on<br>PFOA's RfD x<br>0,02   | RIVM / Bil<br>et al. 2020<br>& 2021                          |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |
|   |               |   | oral                        |               | 21,5<br>ng/kg/d                  | Rate                                  | RfD   | RPF based on<br>PFOA's RfD x<br>0,04   | RIVM / Bil<br>et al. 2020<br>& 2021                          |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |
| 8:2-<br>FTOH  | 678-<br>39-7  | NC  | oral                        | Hepatotoxic   | 1,5 x 10 <sup>3</sup><br>ng/kg/d | Rate                                  | RfD   | RfD<br>assimilated to<br>PFOA<br>transformation<br>Product 8:2-<br>FTOH &<br>Inhalation:<br>20 m <sup>3</sup> /d | SLU 2018<br>(Sweden)<br>(Ingestion<br>based on<br>EFSA 2008) |      |              |      |   |  |                        |            |          |                                    |           |     |                                   |           |

#### 4. Évaluation des risques sanitaires :

Comme indiqué, cette évaluation consiste à mettre en relation les doses de PFAS ingérées ou inhalées avec les VTR.

Dans le cas des alcools fluoro-téloméres (FTOH) et d'autres PFAS volatils (TFA, etc.), présents dans les sols, eaux souterraines et gaz du sol, il faudra aussi réaliser des investigations dans l'air ambiant des bâtiments destinés à des usages sensibles (ERP : Ecoles, Crèches, etc.) ou résidentiels., sur une base des seuils de quantification suffisamment bas et adapté. Les Seuils de quantification de la chimie analytique ne devraient pas être plus élevés que les VTR applicables (cf. Bil et al. 2020, 2021



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et 2023 [12, 37 et 47] et SLU 2017 [48]).

L'EQRS peut permettre de de fixer des Concentrations Maximales Acceptables (CMA) dans des milieux d'exposition. Les CMA sont couramment utilisées sous forme de Valeurs de contrôle de conformité sanitaire, afin de vérifier ou de co-élaborer des objectifs de mesures correctives, voire des objectifs de dépollution ou des valeurs limites recommandées ou réglementaires Ces évaluations sont réalisées substance par substance, des méthodes permettent d'intégrer des expositions combinées à de multiples PFAS par exemple en additionnant les augmentations de risques du fait de l'exposition (Excès de risque individuel ou ERI), par pour les effets sans seuil (le plus souvent cancérigènes, par ex. d'un ERI >  $10^{-5}$ ) ou les Quotient de danger ou dr risque (rapports niveau d'exposition sur VTR à seuil) pour les effets dits « à seuil » : Indice de Risque systémique de IR < 1 (= DJE / DJT : Dose Journalière d'Exposition par rapport à la Dose Journalière Tolérable).

L'évaluation des risques nécessite aussi une bonne prise en compte des précurseurs (PFAS polyfluorés) et de leur chimie environnementale, afin d'apprécier le potentiel de formation environnementale et dans l'organisme de PFAS per-fluorés stables. La Figure 1a ci-dessous donne un exemple de biotransformations de précurseurs poly-fluorés vers des PFAS per-fluorés stables. La Fig. 1b montre la Photolyse du 6:2-FTAB envers les PFAS ultrashorts, comme le PFPrA (Acide perfluoropropionique) et TFA (Acide trifluoroacetic), à côté des PFHxA, PFPeA and PFBA (Naveed, A. et al 2024) [80], particulièrement applicable dans les eaux superficielles et la chaine alimentaire associée.



Fig. 1 : Exemple de biotransformation de précurseurs (PFAS poly-fluorés) vers des PFAS perfluorés stables : le 8:2 FTOH est transformé en PFHxA, PFHpA et PFOA : Graphique modifié selon N. Wang et al. 2009 [49] et J. Liu & S. M. Avendaño, 2013 [50].



Fig. 1b : Photolyse du 6:2-FTAB envers les PFAS ultrashorts, comme le PFPrA (Perfluoro-propionic Acid) et TFA (Trifluoro-acetic-acid), à coté des PFHxA, PFPeA and PFBA (Naveed, A. et al 2024).

Les quelques VTR présentées dans ce guide sont issues des rapports rédigés par des institutions nationales et internationales (cf. le tableau 4). Pour sélectionner des VTR pour des PFAS, il est recommandé d'appliquer des critères de choix scientifiques et non des critères de gestion nationaux ( cf. le tableau 3). Concernant les EQRS (évaluations quantitatives des risques sanitaires), il faudra appliquer les étapes suivantes :

#### 4.1. Identification des scénarios et des voies d'exposition applicables :

Les voies d'exposition suivantes peuvent exister sur un site :

#### > Inhalation :

- $\cdot$  des polluants sous forme gazeuse et particulaires ,
- · des poussières sur lesquelles est adsorbé le polluant,
- · de vapeur d'eau de distribution contaminée lors de la prise d'une douche ou d'un bain.



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#### > Ingestion :

- · directe du sol (terres à l'extérieur d'un bâtiment) et de poussières (terres à l'intérieur d'un bâtiment),
- $\cdot$  des aliments auto-produits (fruits, légumes, etc.),
- · d'eau de distribution contaminée,
- $\cdot$  d'eau lors de la prise d'une douche ou d'un bain (y compris dans une eau superficielle).

#### Absorption cutanée :

- · de sol et de poussières,
- · de polluant par l'eau de distribution contaminée lors de la prise d'une douche ou d'un bain,
- · lors de la prise d'un bain dans les eaux superficielles (étang, rivière, eau souterraine pompée, etc.).

En principe des scénarios d'exposition et des voies d'exposition associées sont à identifier via un Schéma conceptuel (cf. Fig. 2), montrant des sources de pollution, des voies de transfert des polluants et les cibles concernées (adultes, enfants...).



Fig. 2 : Exemple d'un schéma conceptuel, afin de bien visualiser les voies d'expositions applicables par scénario d'exposition (F. Karg & L. Robin-Vigneron 2023a [38]).

Une attention particulière doit être apportée aux PFAS volatils, comme par ex. les alcools fluorotélomères (FTOH), en particulier le 6 :2-FTOH et le 8 :2-FTOH. Le tableau 5 montre des voies d'expositions par Scénario d'usage ou d'exposition d'un site.

#### Tableau 5 : Scénarios d'expositions et voies d'expositions associées





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| Scénario<br>Voie | o d'exposition sur<br>site<br>/<br>s d'exposition   | Industrie<br>et<br>Commerce | Parcs &<br>Loisirs /<br>Activités<br>sportives | Jardins<br>d'enfants | Résidentiel<br>collectif<br>et Crèches<br>& Ecoles | Agricole<br>et<br>produc-<br>tion des<br>aliments | Résidentiel<br>avec<br>jardins<br>individuels |
|------------------|---|-----------------------------|--|----------------------|--|---|---|
| Inkolo           | Respiration des<br>Vapeurs ou gaz :<br>Gaz du sol → Air<br>Ambient<br>(Intérieur &<br>Extérieur)          | Oui                         | Oui  | Oui                  | Oui  | Oui   | Oui   |
| tion             | Respiration des<br>poussières   | Oui                         | Oui  | Oui                  | Oui  | Oui   | Oui   |
|                  | Respiration de la<br>vapeur contaminé<br>pendant la douche<br>ou d'un bain<br>chaud*                      | (Oui)                       | (Oui)  | Non                  | Oui  | Non   | Oui   |
| Oral             | Ingestion passive<br>(Enfants &<br>Adultes) ou active<br>(Enfants) du sol<br>contaminé                    | Oui                         | Oui  | Oui                  | Oui  | Oui   | Oui   |
|                  | Aliments<br>autoproduits  | Non                         | Non  | Non                  | Non  | Oui   | Oui   |
|                  | Ingestion de l'eau<br>contaminée*   | (Oui)                       | (Oui)  | Non                  | Oui  | Oui   | Oui   |
|                  | Contact direct au<br>sol pollué   | Oui                         | Oui  | Oui                  | Oui  | Oui   | Oui   |
|                  | Contact cutané:<br>douche ou bain   | (Oui)                       | (Oui)  | Non                  | (J)  | Non   | Oui   |
| Dermal           | Contact cutané:<br>bain dans les eaux<br>superficielles ou<br>eaux souterraines<br>pompées<br>contaminées | Non                         | Oui  | Non                  | (Non)  | (Non)   | (Oui)   |

(\*) : par exemple dans le cas des conduites d'eau potable enterrées en sous-sol contaminé

#### 4.2. Quantifier les doses journalières d'exposition (DJE) :

Les doses journalières d'exposition - DJEing (voie orale), DJEinh (voie respiratoire), DJEcut (voie cutanée) - sont toutes exprimées en en mg polluant / kg poids corporel / jour [mg • kg<sup>-1</sup> • j<sup>-1</sup>] et dépendent :

- des concentrations en polluants dans les médias d'exposition (par ex. en mg/kg ou  $\mu$ g/l dans les aliments ou de l'eau potable ou en mg/kg ou  $\mu$ g/l dans les poussières ou eaux successibles de provoquer un contact cutané ou en  $\mu$ g/m<sup>3</sup> pour l'air ambiant);



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- Cm = Concentration en polluant dans le médium d'exposition : Csol [mg/kg], Ceau [mg/l], Caliment [mg/kg],
- Ca = Concentration en polluant dans l'air [mg/m<sup>3</sup>] intérieur et/ou extérieur,
- Cs = Concentration en polluant dans le sol [mg/kg] ou dans les eaux (mg/l)

Deux grandes démarches sont applicables pour déterminer les concentrations en polluants dans les médias d'exposition : les mesures directes (prélèvements et analyses) et les modélisations par simulation des transferts de polluants vers les médias d'exposition.

Les modélisations des transferts des polluants entre des médias (par ex. des eaux souterraines ou des sols vers des gaz du sol et de l'air ambiant ou vers des aliments) sont possibles et pourront être calibrées via des analyses des échantillons réelles (recommandé, si possible).

Concernant la prise en compte des concentrations des polluants dans les sols, les gaz du sol, l'air ambiant, les eaux souterraines et les eaux superficielles, il est possible d'appliquer, pour les expositions chroniques (supérieures à 6 mois) uniquement, des concentrations moyennes prenant en compte l'atténuation naturelle mesurée. Cette atténuation naturelle de dégradation des PFAS concerne uniquement la biotransformation des PFAS poly-fluorés mais pas les PFAS per-fluorés (cf. par ex. la Fig. 1).

- des budgets espace-temps (présence des personnes exposées dans le milieu pollué en tenant compte du nombre d'heures par jour, nombre de jours par an et nombre d'années),

| Ex   | = Exposition totale par adulte ou enfant [a]                                 |
|------|--|
| Ve   | = Années de la vie entière par adulte ou enfant [a]. En cas d'exposition aux |
|      | substances avec seuil : $Ve = Ex [a]$  |
| Fexa | = Fréquence d'exposition annuelle [j/365 j]                                  |

Fexi = Fréquence d'exposition journalière [hrs/24 hrs]

- des quantités d'aliments ou d'eau potable ingérés (en kg/j) et/ou du volume d'air inhalé par jour (en m<sup>3</sup>/j) et/ou de la quantité de sol ou de l'eau en contact avec la peau (par ex. en g/cm<sup>2</sup> de surface cutanée),

| Qing  | = Quantité ingérée de sol et/ou aliment [kg/j] et/ou d'eau [l/j], distincte entre les   |
|-------|---|
|       | adultes (Qinga) et les enfants (Qinge)  |
| Qinh  | = Quantité inhalée d'air $[m^3/j]$ , distincte entre adultes (Qinha) et enfants (Qinhe) |
| Fa    | = Facteur d'absorption (par biorésorption) du polluant (à défaut : 100 % = [1])         |
| Facut | = Facteur d'absorption d'un polluant (à défaut : 100 % = [1]) ou vitesse                |
|       | d'absorption [m/h] pour la voie cutanée   |

```
Fsp = Fraction de sol dans la poussière (à défaut : 100 \% = [1])
```

- Spex = Surface de peau exposée, distincte entre les adultes et des enfants  $[m^2]$
- Qsp = Quantité du sol (ou poussières) sur la peau  $[kg/m^2]$
- du poids corporel.
- P(a) = Poids corporel d'un adulte [70 kg]
- P(e) = Poids corporel d'un enfant [15 kg]

#### Les formules de la quantification des doses journalières d'exposition sont les suivantes :

Exposition orale par ingestion :  $DJEing = Cm \bullet Qing \bullet P^{-1} \bullet Fa \bullet Ex \bullet Ve^{-1} \bullet Fexa \bullet Fexj$ Exposition par inhalation \* :  $DJEinh = Ca \bullet Qinh \bullet P^{-1} \bullet Fa \bullet Ex \bullet Ve^{-1} \bullet Fexa \bullet Fexj$ 



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\* Il est possible d'évaluer l'exposition via l'air ambiant extérieur séparément de **l'exposition via** l'air ambient d'intérieur.

#### Exposition par contact cutané (sol, poussières, eaux) :

 $DJEcut = Cs \bullet Fsp \bullet Qing \bullet P^{-1} \bullet Facut \bullet Ex \bullet Ve^{-1} \bullet Fexa \bullet Fexj$ 

Les paramètres applicables sont à justifier. Une application des valeurs par défaut des logiciels d'évaluation des risques de type « Black-Box » sont à prescrire, s'ils ne sont pas justifiables de façon transparente pour le scénario d'exposition concerné.

#### 4.3. Sélection des VTR adaptées :

#### I: Les Effets sans seuil de dose :

- En général, il s'agit des effets cancérigènes, mutagènes ou teratogènes via un mode d'action génotoxique), à l'exeption des effets épigénétiques.
- □ Dans ce cas, les Doses Journalières d'Exposition (DJE) sont multipliées par la VTR, s(Excès de Risque Unitaire (ERU) dénommé également selon le organismes élaborateurs Slope Factor (SF) ou Unit Risk (UR), exprimés par ex. en [(mg/kg/j)<sup>-1</sup>], soit : ERU .[(mg/kg/j)<sup>-1</sup>]• DJE (mg/kg/j) = ERI (-) : Excès de Risque Individuel
- L'ERI doit rester inférieur à « 10<sup>-5</sup> », valeur de gestion des risques sans seuil , sinon le risque est considéré comme non-acceptable.

#### II : Les effets avec seuil de dose :

- Il s'agit des effets toxicologiques (neurotoxicité, hépatotoxicité, néphrotoxicité, etc.).
- Dans ce cas, les Doses Journalières d'Exposition (DJE) sont divisées par la VTR, sous forme de DJT (Dose journalière Tolérable), exprimée par ex. en [mg/kg/j], soit:
  DJE (mg/kg/j) / VTR (mg/kg/j) = QR (Quotient de Risque) ou QD (Quotient de Danger)
- Le QDQR doit rester inférieur à « 1 », sinon le risque est considéré comme non-acceptable.

Par ex. les VTR pour le PFOA existent pour les effet cancérigènes (ERU) et systémiques (DJT), cf. Tableau 4.

#### 5. Effets de mélange :

En général, les effets de mélange de polluants (ou « Combined Exposure to multiple Chemicals") sont considérés depuis 2009 par l'IPCS & OMS (WHO) [51] etc. via une additivité des doses d'exposition, si les effets toxicologiques sont similaires ou les mêmes. Les exceptions sont :

- les synergies (interaction) qui montrent des effets toxicologiques plus importants que l'additivité des doses ou
- les antagonismes (interaction) qui provoquent des effets toxicologiques moins importants que l'additivité des doses.

Selon l'IPCS & OMS (WHO) 2009 [51] etc., une différence doit être faite entre :

- l'exposition agrégée (« Aggregate Exposure ») aux polluants individuels par l'ensemble des voies d'exposition,



- l'exposition cumulée (« Cumulative Exposure ») évaluant un risque combiné par plusieurs polluants.

Dans le cas de l'évaluation d'une exposition cumulée, il faudra identifier les polluants montrant les mêmes mécanismes toxicologiques (ou organes cibles). Ces groupes de polluants sont appelés les « MOA: Common Toxic Mode of Action ».

L'addition des doses est prise en compte pour chaque groupe MOA montrant les mêmes mécanismes toxicologiques (ou organes cibles), par ex. via l'application suivante :

Quotient de Risque : QR= Exposition 1/DJT+ Exposition 2/DJT+ ....Excès de Risque Individuel : ERI= Exposition 1 • ERU + Exposition 2 • ERU + ....

Cette application est simplifiée, comme par ex. dans le cas des Facteurs d'équivalence de toxicité pour les Dioxines (PCDD/F) et les « Dioxine-like PCB », les HAP et aussi par des « Potency-Corrections » pour les PFAS, par ex. via les RPF : Relative Potency Factors, cf. ci-après (Tableau 6).

Afin de définir des valeurs guides pour la santé humaine pour un groupe de congénère aussi important (quelques milliers de molécules), il est d'usage de caractériser les effets toxiques des composés. Toutefois, pour des raisons éthiques et financières, l'expérimentation animale n'est pas possible sur un si grand ensemble de molécules (Cousins, 2020 [52]). D'autres approches, in vitro voire in silico, peuvent être envisagées pour récolter suffisamment d'informations afin établir des valeurs guides.

Les informations obtenues sur des composés pris individuellement ne suffisent pas pour déterminer leur action en mélange. En effet, les composés chimiques peuvent interagir lorsqu'ils sont combinés, produisant ainsi des effets synergiques ou antagonistes. À l'inverse, en l'absence d'interaction, et si les substances partagent un mécanisme de toxicité et une cible biologique similaires, un modèle additif (par exemple, l'addition des concentrations) peut être envisagé pour décrire l'effet du mélange (Heys, 2016 [53]).

L'approche d'équivalence toxique s'applique aux mélanges dont les effets sont additifs. Elle est utilisée, notamment, pour interpréter l'effet de certains mélanges d'organochlorés (comme les chlorodibenzo-p-dioxines, chloro-dibenzofuranes et polychloro-biphényls) qui partagent un mode d'action commun, médié par la liaison à un récepteur spécifique, le récepteur AhR (Guyton et al. 2018 [54]).

Étant donné que chaque congénère n'a pas la même efficacité, ils sont classés à l'aide de facteurs de puissance relative (Relative Potency Factors, RPF) par rapport à un congénère de référence, appelé composé index. Ce dernier est souvent le plus toxique ou celui dont les effets sont les mieux documentés. Pour les dioxines et furanes, la dioxine de Seveso (2,3,7,8-tétrachlorodibenzo-p-dioxine) est utilisée comme référence, avec un facteur de 1, et son facteur de toxicité est désigné sous le nom de Toxic Equivalency Factor (TEF).



L'usage de RPF reste toutefois soumis au respect de conditions strictes :

- 1. en application de la définition présentée ci-avant, les composés doivent partager un même mode d'action ;
- 2. l'effet critique identifié pour le composé de référence doit l'être également pour les autres composés. Autrement dit, un composé index présentant un effet critique neurotoxique ne peut servir de référence pour un autre composé dont la toxicité s'exprimerait principalement au niveau rénal, par exemple ;
- 3. idéalement, les composés devraient posséder des courbes de relation dose-réponse de même efficacité tout en se différenciant, bien évidemment, par des puissances différentes. En outre, les courbes devraient être congruentes, c'est-à-dire qu'elles ne se croisent pas. Ces conditions sont nécessaires à la comparaison des Benchmark doses (BMD) pour un même niveau de réponse (Benchmark Response : BMR).

Les chercheurs du RIVM (Bil et al., 2020, 2021 et 2023 [12, 37, 47]; Zeilmaker et al., 2018 [55]) proposent une approche RPF appliquée à la toxicité orale de 14 PFAS et de deux précurseurs (6:2 FTOH et 8:2 FTOH). Ils sélectionnent le PFOA (acide perfluoro-octanoïque [335-67-1]) comme composé index. Le PFOA est avec le PFOS (acide perfluorooctane-sulfonique [1763-23-1]), l'un des PFAS les plus étudiés. Parmi les effets toxiques attribués au PFOA (mais également au PFOS), ils identifient l'hypertrophie hépatique comme effet critique.

Le RPF est établi sur la base du rapport entre la BMD du PFOA et la BMD du congénère d'intérêt (équation 1).

Equation 1

## $\frac{BMD_{PFOA}}{BMD_i}$

La BMD correspond à un BMR équivalent à 5% d'augmentation du poids du foie (absolu et relatif) établi sur des données continues ou à un excès de risque de 10 % d'hypertrophie hépatique. Cette dernière expression du risque est établie sur base de données dichotomisées. L'excès de risque est ajusté pour les taux d'incidence pour un même effet.

En 2023, le RIVM (Zeilmaker et al. 2023 [56] a complété ses premiers travaux en appliquant cette méthode au TFA.

L'approche de Zeilmaker [56] et Bil et al. 2020, 2021 et 2023 [12, 37 et 47] débouche sur la proposition de RPF résumée dans le Tableau 6.





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| Tableau 6 : RPF selon Bil et al. 2020, 2021 et 2023<br>[12, 37 et 47] et Zeilmaker et al. (2023) [56] |             |                                |  |  |  |  |  |  |
|---|-------------|--------------------------------|--|--|--|--|--|--|
| Congénères  | Numéro CAS  | RPF                            |  |  |  |  |  |  |
| Acides sulfoniques  |             |                                |  |  |  |  |  |  |
| PFBS  | 375-73-5    | 0,001                          |  |  |  |  |  |  |
| PFPeS   | 2706-91-4   | $0,001 \le \text{RPF} \le 0,6$ |  |  |  |  |  |  |
| PFHxS   | 355-46-4    | 0,6                            |  |  |  |  |  |  |
| PFHpS   | 375-92-8    | $0,6 \le \text{RPF} \le 2$     |  |  |  |  |  |  |
| PFOS  | 1763-23-1   | 2                              |  |  |  |  |  |  |
| PFDS  | 335-77-3    | 2                              |  |  |  |  |  |  |
| Acides carboxyliqu  | es          |                                |  |  |  |  |  |  |
| TFA   | 76-05-1     | 0,002                          |  |  |  |  |  |  |
| PFBA  | 375-22-4    | 0,05                           |  |  |  |  |  |  |
| PFPeA   | 2706-90-3   | $0,01 \le \text{RPF} \le 0,05$ |  |  |  |  |  |  |
| PFHxA   | 307-24-4    | 0,01                           |  |  |  |  |  |  |
| PFHpA   | 375-85-9    | $0,01 \le \text{RPF} \le 1$    |  |  |  |  |  |  |
| PFOA  | 335-67-1    | 1                              |  |  |  |  |  |  |
| PFNA  | 375-95-1    | 10                             |  |  |  |  |  |  |
| PFDA  | 335-76-2    | $4 \le \text{RPF} \le 10$      |  |  |  |  |  |  |
| PFUnDA  | 2058-94-8   | 4                              |  |  |  |  |  |  |
| PFTrDA  | 72629-94-8  | 3                              |  |  |  |  |  |  |
| PFDoDA  | 307-55-1    | $0,3 \le \text{RPF} \le 3$     |  |  |  |  |  |  |
| PFTeDA  | 376-06-7    | 0,3                            |  |  |  |  |  |  |
| PFHxDA  | 67905-19-5  | 0,02                           |  |  |  |  |  |  |
| PFODA   | 16517-11-6  | 0,02                           |  |  |  |  |  |  |
| Ethers des acides carboxyliques   |             |                                |  |  |  |  |  |  |
| HFPO-DA   | 13252-13-6  | 0,06                           |  |  |  |  |  |  |
| ADONA   | 919005-14-4 | 0,03                           |  |  |  |  |  |  |
| Alcools télomérique   | es          |                                |  |  |  |  |  |  |
| 6:2 FTOH  | 647-42-7    | 0,02                           |  |  |  |  |  |  |
| 8·2 FTOH  | 678-39-7    | 0.04                           |  |  |  |  |  |  |

L'approche de Zeilmaker (Zeilmaker et al. 2023 [56] a été sélectionnée par l'Etat de Hawaï (State of Hawai - Department of Health, 2020; 2021 [57, 58]) afin d'établir certaines VTR orales (preliminary RfD oral) nécessaires à l'établissement des valeurs guides environnementales (eaux souterraines et sol).

L'emploi des RPF produits par Zeilmaker (Zeilmaker, 2018 et 2023 [48, 49]) et Bil (Bil et al., 2021 [40]) dans le cadre de l'évaluation des risques est fortement contesté par Rietjens (Rietjens, 2022 [59]). Rietjens et al. soutiennent l'usage de RPF dans les études de risques mais contestent la robustesse de ceux calculés par Bil (Bil et al. 2020, 2021 et 2023 [12, 37 et 47]). Rietjens et Bil s'opposent sur la sélection des données et sur l'impact du mode d'administration des PFAS chez les rongeurs. Bil et al. (Bil, 2022a [60]) rejettent ces critiques signalant que numériquement les valeurs ne diffèrent pas significativement en modifiant le jeu de données selon les recommandations de Rietjens.



Goodrum et al. (Goodrum et al., 2021 [61]) critiquent également l'approche de Bil et Zeilmaker en soulignant les différences dans le mode d'action des PFAS à chaînes courtes par rapport à ceux à chaînes longues (C>8). Les PFAS à chaines longues présenteraient la capacité de se fixer à un nombre plus important de récepteurs nucléaires (entre 6 et 16) que ceux à chaînes courtes qui seraient limités à deux récepteurs. Goodrum et al. contestent également l'existence d'une congruence dans les relations dose-réponse. La mise en œuvre de cette approche pour les PFAS à chaines ultra-courtes est également contestable.

## Par conséquent, l'application des RPF n'est pas recommandée (en 2025), sauf dans le cas exceptionnel argumenté.

Récemment, Bil et al. (Bil et al., 2022b [62]; Bil, 2023 [12]) ont publié une approche similaire pour le biomonitoring humain. Les RPF proposés sont résumés dans le tableau 7. Les RPF ont été établis sur base de modèles toxicocinétiques conçus à partir d'expérimentations chez le rat.

| Tableau 7 : RPF selon Bil et al, 2022b [62] pour le biomonitoring. |            |     |  |  |  |  |
|--|------------|-----|--|--|--|--|
| Congénères   | Numéro CAS | RPF |  |  |  |  |
| PFBS   | 375-73-5   | 0,2 |  |  |  |  |
| PFHxS  | 355-46-4   | 0,6 |  |  |  |  |
| PFOS   | 1763-23-1  | 3   |  |  |  |  |
| PFBA   | 375-22-4   | 2   |  |  |  |  |
| PFHxA  | 307-24-4   | 10  |  |  |  |  |
| PFOA   | 335-67-1   | 1   |  |  |  |  |
| PFNA   | 375-95-1   | 5   |  |  |  |  |
| PFDoDA   | 307-55-1   | 10  |  |  |  |  |
| HFPO-DA  | 13252-13-6 | 9   |  |  |  |  |

La plus grande inconnue dans les effets des mélanges par des PFAS concerne la grande partie des PFAS poly-fluorés. Il est recommandé d'appliquer les analyses des PFAS (28 – 70 molécules, cf. aussi l'Arrêté Ministériel en France du 20/06/2023) avant et après le « Top Assay ».

Le « Top Assay » (Total Oxidizable Precursor), selon Houtz et Sedlak : 2012 [63] transforme les PFAS poly-fluorés vers les PFAS perfluorés. Dans l'environnement, l'ensemble des PFAS poly-fluorés (Precurseurs) sont bio-transformés vers les PFAS per-fluorés stables. Via le « Top Assay » (cf. Fig. 3) il est possible d'intégrer dans l'évaluation des risques sanitaires immédiatement les PFAS per-fluorés, produits des bio-transformations dans un scénario d'exposition.

Par contre, il ne faut pas se contenter de réaliser l'évaluation des risques sanitaires uniquement sur les PFAS per-fluorés après le « Top Assay », car les effets avec-seuil de dose de certains PFAS poly-fluorés pourront être importants dans les expositions sub-chroniques et chroniques, comme par ex. concernant les PFAS volatils (FTOH : fluorotélomére-alcools, etc.) (Karg et al.2023a, 2024a-g, 2025 [38, 40 - 46, 68, 79]).




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Fig. 3 : Top Assay (Total Oxidizable Precursor) : Example de quantification des PFAS poly-fluorés via leurs acides carboxyliques perfluorés finaux correspondants après dégradation (Houtz et Sedlak : 2012 [63]), à prendre en compte dans une EQRS.

Certaines expositions aux mélanges sont à évaluer attentivement, et notamment entre le PFOA, PFOS, le bisphénol-A et le méthyle-parabène. Dans le cas d'une exposition aux mélanges de PFOA, bisphénol-A et méthyle-parabène provoque la prolifération des cellules épithéliales des seins par suppression de l'apoptose (Dairkee et al. 2018 [64] et Pesonen et al. 2024 [65].

L'exposition faible aux mélanges de PFOA et PFOS a montré un effet synergique, cancérigène en transformant des cellules épithéliales des seins (MCF-10A) en tumeurs malines phénotypiques ce qui permet la prolifération des métastases (Pierozan et al. 2023 [66] et Pesonen et al. 2024 [65]). Les auteurs ont aussi décrit les approches plus détaillées de l'EQRS, notamment avec la prise en compte large des PFAS poly-fluorés par Top Assay dans le Guide (fiches) de gestion des PFAS de la SFSE (Société Francophone de Santé et Environnement / Heilier et al. 2023 - 2024) [67] et F. Karg et al. 2023 - 2024 [68] et 2025 [79].



#### 6. Conclusion :

La plus grande inconnue dans les effets des mélanges par des PFAS concerne la grande partie des PFAS poly-fluorés. Il est recommandé d'appliquer les analyses des PFAS (28 – 70 molécules, cf. aussi l'Arrêté Ministériel en France du 20/06/2023) et la liste des PFAS-65 + 5 PFAS ultrashorts avant et après le « Top Assay » (Tableau 8).

Le « Top Assay » (Total Oxidizable Precursor), selon Houtz et Sedlak : 2012 [63] transforme les PFAS poly-fluorés vers les PFAS perfluorés. Dans l'environnement, l'ensemble des PFAS poly-fluorés (Precurseurs) sont bio-transformés vers les PFAS per-fluorés stables. Via le « Top Assay » (cf. Fig. 3) il est possible d'intégrer dans l'évaluation des risques sanitaires immédiatement les PFAS per-fluorés, produits des bio-transformations dans un scénario d'exposition.

Par contre, il ne faut pas se contenter de réaliser l'évaluation des risques sanitaires uniquement sur les PFAS per-fluorés après le « Top Assay », car les effets avec-seuil de dose de certains PFAS poly-fluorés pourront être importants dans les expositions sub-chroniques et chroniques, comme par ex. concernant les PFAS volatils (FTOH : fluorotélomére-alcools, etc.) (Karg et al.2023a, 2024a-g, 2025 [38, 40 - 46, 68, 79]).

Le choix scientifique des VTR, en utilisant des critères scientifiques décrit est à assurer. Pour certains PFAS importants et très présents en environnement des VTR pourront être définies, par ex. à partir des NOAEL, comme pour le 6 :2-FTAB (Capstone B) et le 6 :2-FTNO 5Capstone A).

Le Tableau 8 montre les PFAS à analyser en minimum dans les médias d'expositions (aliments, eau potable, eau de baignade, sols, poussières et air ambiant (sans oublient les gaz du sol concernant les PFAS volatils) (F. Karg et al. 2024 - 2025) [81 - 84].



| Tableau 8.1. Parametres PFAS a recommander pour les evaluations des risques sanitaires |
|--|
|--|

|   |             | Q  | ~ . ~       |     | Dir. CE         | AM                 |
|---|-------------|----|-------------|-----|-----------------|--------------------|
| PFAS  | Eaux        |    | CAS         | VTR | EP2020/<br>2184 | 20/06/23<br>France |
| PFBA (acide perfluorobutanoique)                            | ng/l        | 1  | 375-22-4    |     |                 |                    |
| PFPeA (acide perfluoropentanoique)                          | ng/l        | 5  | 2706-90-3   |     |                 |                    |
| PFHxA (acide perfluorohexanoique)                           | ng/l        | 1  | 307-24-4    |     |                 |                    |
| PFHpA (acide perfluoroheptanoique)                          | ng/l        | 1  | 375-85-9    |     |                 |                    |
| PFOA linéaire (acide perfluorooctanoique)                   | ng/l        | 1  | 335-67-1    |     |                 |                    |
| PFOA ramifié (acide perfluorooctanoique)                    | ng/l        | 1  | 335-67-1    |     |                 |                    |
| PFOA totale (acide perfluorooctanoique)                     | ng/l        | 1  | 335-67-1    |     |                 |                    |
| PFNA (acide perfluorononanoique)                            | ng/l        | 1  | 375-95-1    |     |                 |                    |
| PFDA (acide perfluorodecanoique)                            | ng/l        | 1  | 335-76-2    |     |                 |                    |
| PFUnDA (acide perfluoroundecanoique)                        | ng/l        | 1  | 2058-94-8   |     |                 |                    |
| PFDoDA (acide perfluorododecanoique)                        | ng/l        | 2  | 307-55-1    |     |                 |                    |
| PFTrDA (acide perfluorotridecanoique)                       | ng/l        | 1  | 72629-94-8  |     |                 |                    |
| PFTeDA (acide perfluorotetradecanoique)                     | ng/l        | 1  | 376-06-7    |     |                 |                    |
| PFHxDA (acide perfluorohexadecanoique)                      | ng/l        | 2  | 67905-19-5  |     |                 |                    |
| PFODA (acide perfluorooctadecanoique)                       | ng/l        | 1  | 16517-11-6  |     |                 |                    |
| PFBS (acide perfluorobutane sulfonique)                     | ng/l        | 1  | 375-73-5    |     |                 |                    |
| PFPeS (acide perfluoropentane sulfonique)                   | ng/l        | 1  | 2706-91-4   |     |                 |                    |
| PFHxS linéaire (acide perfluorohexane sulfonique)           | ng/l        | 1  | 355-46-4    |     |                 |                    |
| PFHxS ramifié (acide perfluorohexane sulfonique)            | ng/l        | 1  | 355-46-4    |     |                 |                    |
| PFHxS totale  | ng/l        | 1  | 355-46-4    |     |                 |                    |
| PFHpS (acide perfluoroheptane sulfonique)                   | ng/l        | 1  | 375-92-8    |     |                 |                    |
| PFOS linéaire (acide perfluorooctane sulfonique)            | ng/l        | 1  | 1763-23-1   |     |                 |                    |
| PFOS ramifié (acide perfluorooctane sulfonique)             | ng/l        | 1  | 1763-23-1   |     |                 |                    |
| PFOS totale (acide perfluorooctane sulfonique)              | ng/l        | 1  | 1763-23-1   |     |                 |                    |
| PFDS (acide perfluorodecane sulfonique)                     | ng/l        | 1  | 335-77-3    |     |                 |                    |
| 4:2 FTS (acide 4:2 fluorotelomer sulfonique) H4-PFOS        | ng/l        | 1  | 757124-72-4 |     |                 |                    |
| 6:2 FTS (acide 6:2 fluorotelomer sulfonique)                | ng/l        | 1  | 27619-97-2  |     |                 |                    |
| 8:2 FTS (acide 8:2 fluorotelomer sulfonique)                | ng/l        | 1  | 39108-34-4  |     |                 |                    |
| 10:2 FTS (acide 10:2 fluorotelomer sulfonique)              | ng/l        | 1  | 120226-60-0 |     |                 |                    |
| MePFOSAA (acide N-méthylperfluorooctane sulfonamide         |             | 1  | 2355-31-0   |     |                 |                    |
| acétique)   | ng/l        | 1  | 2333-31-9   |     |                 |                    |
| EtFOSAA (acide N-éthylperfluorooctane sulfonamide acétique) | ng/l        | 1  | 2991-50-6   |     |                 |                    |
| PFOSA linéaire (perfluoro-n-octanesulfonamide)              | ng/l        | 2  | 754-91-6    |     |                 |                    |
| PFOSA ramifié (perfluoro-n-octanesulfonamide)               | ng/l        | 2  | 754-91-6    |     |                 |                    |
| PFOSA totale (perfluoro-n-octanesulfonamide)                | ng/l        | 2  | 754-91-6    |     |                 |                    |
| MeFOSA linéaire (N-méthylperfluorooctanesulfonamide)        | ng/l        | 1  | 31506-32-8  |     |                 |                    |
| (MePFOSA)   | <u>e</u> /1 | 1  | 51500 52-0  |     |                 |                    |
| 6:2-FTAB (6:2 fluorotelomer sulfonamido propyl betaine)     |             |    | 34455-29-3  |     |                 |                    |
| Capstone B  | ng/l        | 10 | 01100 20 0  |     |                 |                    |



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| Tableau 8.2. : Paramètres PFAS à reco | mmander pour les éva | aluations des risques sanitaires |
|---------------------------------------|----------------------|----------------------------------|
|---------------------------------------|----------------------|----------------------------------|

| PFAS  | L<br>Ea | Q<br>ux | CAS          | VTR | Dir. CE<br>EP2020/<br>2184 | AM<br>20/06/23<br>France |
|---|---------|---------|--------------|-----|----------------------------|--------------------------|
| MeFOSA ramifié (N-méthylperfluoro-n-octanesulfonamide)        | ng/1    | 1       | 31506-32-8   |     |                            |                          |
| MeFOSA totale (N-méthylperfluoro-n-octanesulfonamide)         | 11g/1   |         | 31506-32-8   |     |                            |                          |
| (MePFOSA)   | ng/l    |         | (50.41.1     |     |                            |                          |
| 8:2 DIPAP (8:2 polyiluoroalkyl phosphate diester)             | ng/l    | 1       | 678-41-1     |     |                            |                          |
| HFPO-DA (acide nexalituoropropyleneoxide dimer) Gen X         | ng/l    | 1       | 13252-13-6   |     |                            |                          |
| EtFOSA lineaire (N-ethylperfluorooctanesulfonamide) (EtPFOSA) | ng/l    |         | 4151-50-2    |     |                            |                          |
| EtFOSA ramifie (N-ethylperfluorooctanesulfonamide) (EtPFOSA)  | ng/l    |         | 4151-50-2    |     |                            |                          |
| EtFOSA totale (N-ethylperfluorooctanesulfonamide) (EtPFOSA)   | ng/l    | 1       | 4151-50-2    |     |                            |                          |
| MeFBSAA (perfluorobutanesulfonamide(N-méthyl)acetate)         | ng/l    | 5       | 159381-10-9  |     |                            |                          |
| 5:3-FTCA: 5:3 acide carboxylique fluorotélomère)              | ng/l    | 1       | 914637-49-3  |     |                            |                          |
| 6-2-FTCA: 6:2 acide carboxylique fluorotélomère               | ng/l    | 5       | 53826-12-3   |     |                            |                          |
| 8:2 FTUCA (acide 2H-perfluoro-2-décenoique)                   | ng/l    | 1       | 70887-84-2   |     |                            |                          |
| DONA (acide 4,8-dioxa-3H-perfluorononanoique)ADONA            | ng/l    | 1       | 919005-14-4  |     |                            |                          |
| MeFBSA (n-méthylperfluorobutanesulfonamide)                   | ng/l    | 1       | 68298-12-4   |     |                            |                          |
| PFBSA (perfluorobutanesulfonamide)                            | ng/l    | 1       | 30334-69-1   |     |                            |                          |
| PFECHS (acide perfluoro-4-éthylcyclohexanesulfonique)         | ng/l    | 1       | 646-83-3     |     |                            |                          |
| PFNS (acide perfluorononane sulfonique)                       | ng/l    | 1       | 68259-12-1   |     |                            |                          |
| PFDoDS (acide perfluorododecane sulfonique)                   | ng/l    | 1       | 79780-39-5   |     |                            |                          |
| 6:2 diester de phosphate fluorotélomérique. 6:2 diPAP         | ng/l    | 10      | 57677-95-9   |     |                            |                          |
| 6:2 8:2 diester de phosphate fluorotélomérique. 6:2 8:2 diPAP | ng/l    | 10      | 943913-15-3  |     |                            |                          |
| PFHxSA (perfluorohexanesulfonamide)                           | ng/l    | 1       | 41997-13-1   |     |                            |                          |
| PFUnDS (acide perfluoroundecane sulfonique)                   | ng/l    | 2       | 749786-16-1  |     |                            |                          |
| PFTrDS (acide perfluorotridecane sulfonique)                  | ng/l    | 2       | 791563-89-8  |     |                            |                          |
| EtFOSE (2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol)     | ng/l    | 5       | 1691-99-2    |     |                            |                          |
| MeFOSE (2-(N-methylperfluoro-1-octanesulfonamido)-ethanol)    | ng/l    | 5       | 24448-09-7   |     |                            |                          |
| NFDHpA (Nonafluoro-3.6-dioxaheptanoic acid)                   | ng/l    | 1       | 151772-58-6  |     |                            |                          |
| PFMPA (Perfluoro-3-methoxypropanoic acid)                     | ng/l    | 1       | 377-73-1     |     |                            |                          |
| PFMBA (perfluoro-4-methoxybutanoic acid)                      | ng/l    | 1       | 863090-89-5  |     |                            |                          |
| C6O4 (Perfluoro([5-methoxy-1,3-dioxolan-4-yl]oxy)acetic acid) | ng/l    | 10      | 1190931-41-9 |     |                            |                          |
| 6:2-FTOH (6:2 fluorotelemer alcohol) FHET                     | ng/l    | 20      | 647-42-7     |     |                            |                          |
| 8:2-FTOH (8:2 fluorotelemer alcohol) FOET                     | ng/l    | 10      | 678-39-7     |     |                            |                          |
| PFAS Ultrashorts  |         |         |              |     |                            |                          |
| TFA (trifluoroacetic acid)                                    | ng/l    | 10      |              |     |                            |                          |
| PFPrA (perfluoropropanoic acid)                               | ng/l    | 10      |              |     |                            |                          |
| TEMS (trifluoromethanesulfonic_acid)                          | no/l    | 10      |              |     |                            |                          |
| PFFS (perfluoroethanesulfonic acid)                           | no/l    | 10      |              |     |                            |                          |
| DEDrS (perfluoropropageulfonic acid)                          | ng/l    | 10      |              |     |                            |                          |
| rirris (perhabitopropanesanonic acid)                         | ng/i    | 10      |              |     |                            |                          |

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# Air Emissions, transport and vapor intrusion in buildings. From conceptual diagram to feedback on indoor/outdoor air concentration measurements of PFAS

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#### Résumé

Les PFAS sont une famille de produits chimiques artificiels considérés comme des Polluants Organiques Persistants en raison de leur dégradation lente et de leur omniprésence dans tous les compartiments de l'environnement. Les composés PFAS sont suspectés d'avoir des effets cancérigènes et présentent des risques pour la santé. Le ministère de la Transition Ecologique et de la Cohésion des Territoires a présenté en janvier 2023 un plan d'action pour réduire ces risques et améliorer la connaissance des effets des PFAS sur la santé et l'environnement. Ce plan d'action est complété en avril 2024 avec la volonté de renforcer les dispositifs de surveillance.

Ginger LECES est le premier laboratoire à avoir reçu l'accréditation par le Comité français d'accréditation (COFRAC) pour déterminer les concentrations en PFAS dans les rejets atmosphériques selon la méthode OTM-45. Des développements sont en cours pour les mesures en rejet d'autres PFAS (très volatils : OTM-50, non polaires : OTM-55).

Par ailleurs, au regard de la grande variabilité des molécules, de leurs caractéristiques physicochimiques et de leur rémanence, la question des méthodes appropriées pour la surveillance environnementale des impacts se pose.

Enfin, vis-à-vis des environnements intérieurs de bâtiments, la multiplicité des sources de dégradation de la qualité de l'air (sols pollués, air atmosphérique, revêtements et produits de consommation courante) pose également aujourd'hui la question des méthodes de caractérisation.

La communication proposée s'articule ainsi autour du schéma conceptuel mettant en regard les sources de PFAS, les vecteurs de migration et les populations susceptibles d'être exposées. Dans un premier temps, au niveau des sources, les formes physiques des PFAS (répartition phase particulaire et phase gazeuse) mesurés sur les rejets atmosphériques de multiples industriels seront mises en regard des mesures dans l'air et les dépôts atmosphériques. Ces résultats permettent d'apporter un éclairage sur les méthodes de mesures dans l'environnement. Dans un second temps, nous nous s'intéresserons à la qualité de l'air intérieur et plus largement la qualité des environnements intérieurs. Après la présentation des multiples sources de dégradation (air atmosphérique, sources internes : revêtements, produits de consommations et sols/ nappes pollués), les impacts pouvant être issus de sols ou nappes contaminées seront évalués à travers la modélisation des transferts (vapor intrusion) pour différentes situations. Les concentrations dans l'air intérieur des différents FTOH volatils seront alors mises en regard des concentrations mesurées. Les axes d'amélioration des connaissances nécessaires seront proposés en conclusion.





#### From AOF to PFAS identification: An integrated approach

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In France, since July 2023, the analysis of Adsorbable Organic Fluorine (AOF) has become mandatory in wastewater for many industries to monitor the presence of PFAS in industrial discharges.

AOF analysis is as an essential first step in detecting potential PFAS contamination, providing a rapid and comprehensive indication of the presence of organic fluorine in wastewater.

However, on its own, this measurement does not identify the specific molecules responsible for contamination or differentiate between various PFAS classes. This can made the difference for those industries that are working on PFAS decontamination.

To transform raw AOF data into actionable insights for environmental monitoring and risk management, our case study shows an integrated approach that combines AOF with advanced screening techniques.

In the presented case study, wastewater samples, positive for AOF analysis, underwent a preliminary treatment designed to separate low molecular weight substances: this preparation step allowed the analysis to focus on the most critical chemical species from a contamination and risk assessment perspective.

Subsequently, a suspect screening analysis was conducted to compare the detected compounds against a library of over 1,000 PFAS belonging to 40 different PFAS families. After this step, a non-targeted analysis was performed, capable of identifying characteristic PFAS functional groups such as  $CF_2$  and  $CF_3$ . This step allowed for the detection of unexpected or next-generation PFAS, which may evade traditional screening methods.

Integrating these approaches with AOF analysis transforms an aggregate measurement into detailed and targeted insights into PFAS contamination in wastewater. This advanced methodology not only enlarge the possibilities of identification of specific compounds responsible for pollution but also provides critical data to enhance environmental monitoring and risk management strategies, contributing to a more comprehensive assessment of PFAS presence in the environment.

## Mesure de 7 composés PFAS critiques dans le sérum humain en utilisant une préparation d'échantillon par SPE dispersive en pipette avant une analyse par LC-MS/MS.

#### Measurement of 7 critical PFAS compounds from human serum utilizing dispersive in-pipette SPE sample prep prior to LC-MS/MS Analysis

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#### Introduction:

PFAS (per- and polyfluoroalkyl substances) testing in clinical research is crucial due to their environmental contamination and associated health risks, including immune disruption and cancer. The National Academies of Sciences, Engineering, and Medicine, NASEM, "Guidance on PFAS Testing and Health Outcomes" report emphasize the need for biomonitoring of seven key PFAS compounds: PFOA, PFOS, PFNA, PFHxS, PFDA, PFUnA, and PFDoA. These substances are persistent and bioaccumulative, necessitating rigorous testing to investigate exposure pathways and health implications. Understanding these factors is vital for evaluating interventions to reduce PFAS exposure and for informing public health strategies and regulatory policies aimed at mitigating their impact on human health and the environment.

#### Methods:

For the analysis of seven key PFAS analytes (PFOA, PFOS, PFNA, PFHxS, PFDA, PFUnA, and PFDoA) as highlighted by the NASEM report, human serum samples were spiked with stable isotopically labeled internal standards and vortexed, with a portion reserved as a control. Analytes were quantified against matrix-matched and solvent prepared calibrators. Matrix-matched calibration curve was prepared using the same serum lot. Samples underwent solvent-assisted protein precipitation with cold 1% ammonium formate in methanol, followed by solid-phase extraction using a zirconia-coated silica in-pipette adsorbent to remove known-matrix contaminants such as phospholipids. Finally, samples were analyzed directly by LC-MS/MS without further concentration or dilution.

#### **Preliminary data:**

Automation of sample preparation was achieved using a liquid handler, enhancing reproducibility, and allowing simultaneous preparation of 96 samples using in-pipette dispersive adsorbent. This approach eliminates the need for positive or negative pressure systems, making it more economical. Calibration curves prepared with matrix-matched samples and solvent-based methods showed similar results across seven analytes, with only one concentration for a single analyte differing. This demonstrates the effectiveness of the developed sample preparation in mitigating matrix effects, enabling future studies to utilize solvent-prepared calibration curves.

Reproducibility tests for matrix-prepared samples at 0.5 ng/mL showed accuracy percent standard deviations below 6% for all analytes, except for PFUnA, which had a 15% deviation, correlating with its higher concentration in serum. For serum samples spiked at 1.25 ng/mL of each analyte (PFOA, PFOS,

PFNA, PFHxS, PFDA, PFUnA, and PFDoA), relative recoveries ranged from 77.2% to 101.8%, while absolute recoveries varied from 72.8% to 92.6%.

The chromatography method employed parameters established in previous EPA 1633 method development, utilizing a PFAS-tested C18 delay column (50 mm x 3.0 mm, 2.7  $\mu$ m) before the LC autoinjector, followed by a PFAS-tested C18 column for separation (100 mm x 2.1 mm, 2.7  $\mu$ m).

This methodology ensures accurate and reliable detection of PFAS compounds in complex biological matrices, facilitating further research on PFAS exposure and health implications.

#### **Novel Aspect:**

PFAS analysis from human serum using non-pressure SPE mediated sample preparation prior to LC-MS.

#### Human Health Risk Assessment of PFAS on the site of an old paper manufacture: Stakes and Challenges

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In the context of the development of the site of a former paper manufacture, a quantitative health risk assessment study was carried out for different development scenarios. From very sensitive (residential with garden / green space or nursery with garden) to less sensitive (tertiary, industrial or green space), five exposure scenarios were evaluated. The exposure routes taken into account cover all the possibilities of these scenarios, in the current state of knowledge on PFAS:

- Ingestion of soil, dust, self-produced vegetables,
- Inhalation of gas and dust,
- Skin contact with soil and dust,
- Tap water not considered (lack of data about PFAS permeation) and groundwater not used for consumption or irrigation.

PFAS analyses were performed on soil, soil gas and groundwater samples. The analysis of 66 PFAS was carried out on those media, followed by Top Assays on water and soil samples, in order to optimize the number of perfluorinated molecules taken into account. These tests make it possible to optimize the consideration of PFAS molecules, by transforming polyfluorinated molecules into perfluorinated molecules into generated molecules the concentrations of perfluorinated molecules integrated into the assessment. These tests represent a predictable degradation over time.

This step of sampling and labor analysis generated significant uncertainties that need to be identified for a realistic approach to risk assessment:

- There are a lot of biases during sampling, which can interfere with the results (floor coverings, use of pesticides, containers, PPE, etc.),
- The number of molecules that can be analysed in "routine" (66 molecules) is relatively small compared to the estimated 10,000 molecules or more,
- The laboratory's uncertainty about the analyses is 30% to 40%,
- The reproducibility of the analyses is not optimal.

These uncertainties have been taken into account as much as possible in the performance of the risk calculations. The measured concentrations were increased by the percentage of laboratory uncertainties and the results of the Top Assays provided higher concentrations of perfluorinated molecules and short chains. Taking into account the uncertainties led to multiple quantifications with and without percentage of uncertainties for the five scenarios, before and after Top Assay, taking into account arithmetic means, maximum concentrations, etc.

The toxicity of PFAS molecules is also a major source of uncertainty. The same molecule can have toxicological values of totally different orders of magnitude, depending on the reference organisation (database). For example, for PFOA, more than 10 databases offer toxicological values ranging from 0.03 ng/kg/day to 100 ng/kg/day for systemic risk. In our study, the most recent and best documented toxicological values were taken into account.

The existence or absence of toxicological value also limits the number of PFAS molecules taken into account in the risk assessment study: among 66 molecules analysed, only 27 could be considered for a risk quantification. The results of the Top Assays made it possible to include more substances, as

polyfluorinated substances converted into perfluorinated substances could be taken into account in the 27, which reduced this uncertainty.

The results obtained were interpreted with regard to the limitations mentioned above, and led to highly unacceptable risks for the three most sensitive scenarios (residential with or without a private garden and nursery with garden). The main exposure pathways involved in unacceptable risks are the ingestion of self-produced vegetables and the ingestion of soil and dust.

In order to map the areas of health risk, a calculation of maximum acceptable concentrations (MAC), based on health risk assessment, was carried out for each molecule taken into account in the study, for the soil and groundwater environments and for each exposure scenario. Maps showing the areas where these MAC were exceeded were produced, an example below:

#### Residential scenario with private garden:

Areas where PFAS are exceeding maximal acceptable concentrations based on health risk assessment



This health risk assessment study has provided a good understanding of the risks associated with PFAS, despite the uncertainties and gaps. This study made it possible to map areas where health risks are exceeded, according to the development scenario, thus offering secure management and informed choices for future development.

## Évaluation Quantitative des Risques Sanitaires liés aux PFAS sur le site d'une ancienne fabrique de papier : Enjeux et Défis

## Human Health Risk Assessment of PFAS on the site of an old paper manufacture: Stakes and Challenges

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Dans le cadre d'un projet d'aménagement du site d'une ancienne fabrique de papier, une étude d'évaluation quantitative des risques sanitaires a été réalisée pour différents scénarios d'aménagement. De très sensibles (résidentiel avec jardin / espace vert ou crèche avec jardin) à moins sensibles (tertiaire, industriel ou espace vert), cinq scénarios d'exposition ont été évalués. Les voies d'exposition prises en compte couvrent l'ensemble des possibilités de ces scénarios, en l'état actuel des connaissances sur les PFAS :

- Ingestion de sols, de poussières, de légumes autoproduits,
- Inhalation de gaz et poussières,
- Contact cutané avec les sols et poussières,
- Eau du robinet non considérée (absence de données de perméation des PFAS à travers le PEHD) et eaux souterraines non utilisées pour la consommation ou l'irrigation.

Les analyses de PFAS ont été réalisées sur des échantillons de sols, de gaz du sol et d'eaux souterraines. L'analyse de 66 PFAS a été réalisée sur ces milieux, suivie de Top Assays sur des échantillons d'eaux et de sols, afin d'optimiser la prise en compte des molécules perfluorées. Ces essais permettent en effet d'optimiser la prise en compte des molécules PFAS, en transformant par oxydation des polyfluorés en perfluorés, ce qui augmente in-fine les concentrations en molécules perfluorées intégrées dans les analyses. Ces essais réalisent en accéléré une dégradation prévisible dans le temps.

Cette étape du prélèvement et de l'analyse au laboratoire a généré des incertitudes importantes qu'il est nécessaire d'identifier pour une approche réaliste et éclairée de l'évaluation des risques :

- Il existe beaucoup de biais lors des prélèvements, qui peuvent interférer sur les résultats (revêtements de sol, usage de pesticides, contenants, EPI...)
- Le nombre de molécules analysables en « routine » : 66 molécules est relativement faible comparées aux 10 000 ou plus existantes,
- L'incertitude du laboratoire sur les analyses est de 30% à 40%,
- La reproductibilité des analyses n'est pas optimale.

Ces incertitudes ont été prises en compte au maximum dans la réalisation des calculs de risques. Les concentrations mesurées ont été augmentées du pourcentage d'incertitudes du laboratoire et les résultats des Top Assays ont fourni des concentrations plus importantes en molécules perfluorées et en chaines courtes. La prise en compte des incertitudes a conduit à réaliser pour les cinq scénarios, des quantifications multiples avec et sans pourcentage d'incertitudes, avant et après Top Assay, en considérant les moyennes arithmétiques, les concentrations maximales, etc...

La toxicité des molécules PFAS est également une grande source d'incertitudes. Une même molécule peut présenter des valeurs toxicologiques d'ordres de grandeur totalement différents, selon l'organisme de référence. Par ex., pour le PFOA, plus de 10 bases de données proposent des valeurs toxicologiques allant de 0,03 ng/kg/j à 100 ng/kg/j pour le risque systémique. Dans notre étude, les valeurs toxicologiques les plus récentes et les mieux documentées ont été prises en compte.

L'existence ou non de valeur toxicologique limite également la prise en compte des molécules PFAS dans l'étude d'évaluation des risques : sur 66 molécules analysées, seules 27 ont pu faire l'objet d'une quantification des risques. Les résultats des Top Assays ont permis d'intégrer davantage de substances,

car les polyfluorés transformés en perfluorés ont pu être pris en compte dans les 27, ce qui a permis de réduire cette incertitude.

Les résultats obtenus ont été interprétés au regard des limites évoquées ci-avant, et ont conduit à des risques fortement inacceptables pour les trois scénarios les plus sensibles (résidentiel avec ou sans jardin privatif et crèche avec jardin). Les voies d'exposition principalement impliquées dans les risques inacceptables sont l'ingestion de légumes autoproduits et l'ingestion de sols et poussières.

Afin d'identifier les zones à risques sanitaires, un calcul de concentrations maximales admissibles (CMA) a été effectué pour chaque molécule prise en compte dans l'étude, pour les milieux sols et eaux souterraines et pour chaque scénario d'exposition. Des cartographies montrant les zones de dépassement de ces CMA ont été réalisées, un exemple ci-dessous :

#### Scénario résidentiel avec jardin privatif :

Zones de dépassement des concentrations maximales admissibles basées sur des risques sanitaires.



Cette évaluation des risques sanitaires très complète a permis une bonne appréhension des risques liés aux PFAS, en dépit des incertitudes et des lacunes de connaissance sur ces polluants. Les cartographies découlant de cette étude permettent d'identifier les zones à risques sanitaires en fonction de l'aménagement prévu, offrant ainsi une gestion sécurisée et des choix éclairés pour l'aménagement futur.

### Freshwater is a resource that can no longer be taken for granted – we need a circular transformation to take place, but it is compromised/endangered by PFAS

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PFAS is an increasing problem in the EU and the world. The chemicals are pervasive pollutants detected in water, soil, and food across Europe, posing serious environmental and health risks. Long-term exposure to these substances is linked to various health issues, including cancer and developmental disorders, highlighting the urgent need for effective remediation solutions.

The price of buying 1,000 grams of a common PFAS chemical now stands at less than 10 euros – a fraction of the price of making sure that it does not end up in our environment, our food, or our drinking water. Today's waste management system is not designed to handle PFAS, nor do the current market prices for waste management services reflect the costs for extra PFAS cleaning. The waste management infrastructure is nevertheless connected to the urban wastewater infrastructure (waste management plants send their wastewater through the sewer systems). Landfills with limited degrees of groundwater protection, therefore, risks leaking PFAS into the society.

The presence of PFAS in products would, however, likely be reduced if producers who add PFAS to their products would be forced to bear the full cleaning costs. Such a liability mechanism would also increase the speed of detoxifying the ecosystems from PFAS. This is, however, not the case today. Instead, we are moving towards a scenario where the already limited amount of freshwater is becoming contaminated, which requires increasingly advanced treatment efforts.

PFAS is present in the current waste flow and will most likely be present for decades to come. In this speech, I will explain how Ragn-Sells is working in several ways to remove PFAS from the cycles and prevent it from spreading from our facilities. I will talk about how we advocate for stricter requirements on sampling and standardised measurement methods and actively work with our customers to obtain information about the risk that their waste contains PFAS and to push for the "polluter-pays-principle" – those who profit from using PFAS must foot the bill. This requires new legislation that imposes requirements on manufacturers.

I will touch on Ragn-Sell's efforts to

- Coordinate knowledge, actions, and approaches to manage PFAS in a consolidated manner and continuously evaluate its effectiveness.
- Advocate for stricter requirements on sampling and standardized measurement methods.
- Initiate the development of a solution to remove PFAS from problematic hotspots which will be further discussed by Chromafora on 19 June.
- Push for an EU ban on the use of PFAS in products to ensure circular practices.

A contribution from Ragn-Sells Chief Sustainability Officer Pär Larshans, who could not attend the conference, will also be included in the presentation.

## Serum levels of Per- and Polyfluoroalkyl Substances in relation to the length of residency in United States

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Key words: PFAS, Immigrant population, Environmental exposure, Residency duration, United States, Public Health.

#### ABSTRACT :

Immigrants in the United States may experience varying levels of exposure to perfluoroalkyl substances (PFAS) depending on their duration of residency and their region of origin. PFAS are persistent environmental pollutants linked to adverse health outcomes, yet little is known about exposure levels among immigrant populations. This cross-sectional study utilized data from the National Health and Nutrition Examination Survey (NHANES) 2003-2018 to assess serum PFAS concentrations among adults by length of U.S. residency and race/ethnicity (used as proxy for origin). Several PFAS compounds were analyzed using ANCOVA and general linear models. U.S.born adults represented 74.9% (n=8,272) of the sample. Overall, PFAS levels increased with time in the U.S., converging towards levels observed in U.S.-born individuals-, consistent with the long biological half-lives of certain PFAS compounds. Significant differences in SPFAS were found for immigrants residing less than 5 years (geometric mean ratio [GMR]: 0.73, 95% CI: 0.64-0.84, P<0.0001), with smaller differences in those residing 5-10 years (GMR: 0.84, 95% CI: 0.77-0.91, P<0.0001) or 10-15 years (GMR: 0.84, 95% CI: 0.77-0.93, P=0.0004), compared to U.S.-born. The pace of convergence varied by race/ethnicity, reflecting both environmental exposure in the U.S. and likely differences in exposure prior to migration. These results emphasize the need to consider duration of residency and background when evaluating chemical exposure disparities. The study highlights the need for public health interventions to address PFAS exposure among immigrants, especially those newly arrived, considering their potential differential exposure risks.

## Development of a PBPK model for the female mouse from birth to gestation, lactation to offspring: application to a PFOA oral exposure

## Développement d'un modèle PBPK pour la souris femelle de sa naissance à la gestation et allaitement du souriceau : application à une exposition orale au PFOA

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Keywords: PBPK model, mice, neurodevelopment, PFAS, pregnancy

#### Innovative nature of the proposed work:

A generic female mouse PBPK model was developed to include different life stages within a single model. This model, specific for females, considers physiological changes during growth, pregnancy and lactation, including fetal and offspring development. The model was therefore developed to predict the mice fetal and neonatal internal exposure resulting from oral exposure of dams during the pregestational, gestational and lactation periods. The model has been parametrized for PFOA, considering exposures during gestation and lactation, and has been validated with observed concentrations in different matrices. This model can support the different PFOA kinetics between a single or repeated oral doses.

#### <u>Aims:</u>

This work aims to: (i) develop a PBPK model for a female mouse and offspring; (ii) predict internal concentration for PFOA exposure.

#### Abstract:

PFOA has been found widespread in human serum, breast milk and placental cord blood, suggesting fetal and lactational exposures, causing neurodevelopmental disruption in mice (Loccisano et al., 2012). Mice are commonly used in neurodevelopmental studies, but the observed effects are rarely linked to internal concentrations of substances in the offspring's brain. It is therefore relevant to consider pregestational and maternal chemical exposures to better characterize fetal and lactational exposure, particularly during the critical window of early brain development. Physiologically Based Pharmacokinetic (PBPK) models allow to predict the toxicokinetic behavior of xenobiotics in an organism based on chemical properties and mouse physiological properties. While there are a few PBPK models for PFOA, none have been developed female mice, and none allow for continuous exposure from pre-gestation to lactation. We have developed a mouse PBPK model to determine the PFOA internal concentration in the offspring's brain resulting from maternal oral exposure. This model comprises six compartments for dams and five for the offspring including the brain compartment (Figure 1). The model also describes the dam's growth from birth and pup from the early embryonic

stages, as well as the changes in organ volumes and blood flows. The model has been parametrized for PFOA using parameter values from literature and updated to the mouse by calibrating the urinary constant parameter value. Predictions from the model align closely with observations from Fenton et al. (2009) and Hinderliter et al. (2005) in various biological matrices, including blood, mammary glands/milk, placenta, and offspring blood, with most predictions showing a 3-fold variation around the observed values. The model can predict the internal PFOA kinetics in the offspring brain throughout the neurodevelopmental window.



Figure 1 Structure of the generic pregnancy PBPK model for mouse. RP: Rapidly perfused, SP: Slowly perfused.

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#### Proposal of emission limit values for per- and polyfluoroalkylated substances (PFAS) based on the study of toxicological reference values (TRVs)

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• Objectives and approach

This study aimed to propose emission limit values (ELVs) for perfluoroalkylated and polyfluoroalkylated substances (PFAS) in industrial effluents emitted to the environment, based on expected health effects on the general population.

The approach is based on the study of toxicological reference values (TRVs) and applies the INERIS guidelines to carry out an integrated approach for management of the emissions of chemical substance from industrial facilities (Ineris-200357-2563482-v1.0, 2021).

The TRVs were selected according to the principles of the ministerial note DGS/EA1/DGPR/2014/307 issued on October 31<sup>st</sup> /2014, which relates to the procedures for selecting toxicological reference values during health risk assessments.

The criteria considered for the acceptability of the health risk are those prescribed by the circular of august 09<sup>th</sup> 2013. They are namely:

- for a substance with a threshold TRV: the acceptability criterion consists in a hazard quotient (HQ) inferior to 1 (HQ < 1).
- For a substance with non-threshold TRV: the criterion of acceptability consists in an excess individual risk (EIR) below 10<sup>-5</sup>.

Once the risk levels were fixated, the associated concentrations of pollutants emitted into the environment were then deduced. Concentration values compatible with health risk control objectives were compared with "benchmark" values applicable to PFAS taken from existing regulations. Technical and economic factors were also analyzed in order to establish ELVs.

• Substances considered

The substances studied correspond to the 28 PFAS listed in paragraphs 2 and 3 of article 3 the ministerial order of June 20, 2023, issued by the French ministry in charge of the environment, relating to the analysis of PFAS in aqueous discharges from installations classified for environmental protection (ICPE) under the category of Authorization. Because of its persistence in the environment, trifluoroacetic acid (TFA) is also considered.

• Exposure scenario

Ineris guidelines prescribe a construction of an exposure scenario based on overall conservative assumptions. Therefore, the exposure scenario in this study was constructed accordingly.

The emission source considered is an ICPE facility with liquid and gaseous discharges with constant characteristics throughout a year. After emissions in watercourses, dilution and mixture of PFAS in the water were considered complete and homogenous. All PFAS emitted into the water were considered bioavailable and therefore absorbed from drinking water and integrated by fish and other biota. Additionally, PFAS inputs to soils through irrigation and atmospheric deposition take place over 30 years (average duration of residence in France).

As for drinking water, all the water consumed by the exposed populations is considered to originate from a contaminated watercourse. Potential PFAS abatement through water treatment processes

during potabilization is not taken into account. Likewise, all the fish and dairy products consumed by the target populations are considered to come from contaminated sources.

As a study case, the human target considered for the quantification of human health risks was a child aged between 1 and 3 years old.

• Calculation models

The equations used to model pollutant transfers in environmental matrices are taken from document DRC-08-94882-16675C issued by INERIS (2010) – « Equations for modeling exposures linked to soil contamination or emissions from an industrial facility». These equations allow the modeling of transfers of PFAS through the food chain.

As for the generic data applied for human exposure, they are mostly taken from document issued by INERIS (2017), DRC-14-141968-11173C.

• Results (proposed threshold values)

Generic ELV levels for the pollutants studied are presented in the table below.

| Abbreviation  | CAS Number | Proposed ELV for an<br>industrial facility (mg/L) |
|---------------|------------|---|
| PFPeA         | 2706-90-3  | 0.1   |
| PFBA          | 375-22-4   | 0.1   |
| PFHxA         | 307-24-4   | 0.1   |
| PFHpA         | 375-85-9   | 0.001   |
| PFOA          | 335-67-1   | 0.0001  |
| PFNA          | 375-95-1   | 0.0004  |
| PFDA          | 335-76-2   | 0.001   |
| PFUnDA; PFUnA | 2058-94-8  | 0.001   |
| PFDoDA; PFDoA | 307-55-1   | 0.001   |
| PFTrDA; PFTrA | 72629-94-8 | 0.001   |
| PFBS          | 375-73-5   | 0.250   |
| PFHxS         | 355-46-4   | 0.0005  |
| PFOS          | 1763-23-1  | 0.0001  |
| PFDS          | 335-77-3   | 0.001   |
| TFA           | 76-05-1    | 1.0   |

In the case of PFOS and PFOA and for industrial plants located in areas subject to pressures from the point of view of the health of residents (sensitive areas), the following threshold values are proposed.

| Abbreviation | CAS Number | Proposed ELV for a plant<br>(mg/L) |
|--------------|------------|------------------------------------|
| PFOA         | 335-67-1   | 0.0000012                          |
| PFOS         | 1763-23-1  | 0.000009                           |

For these additional ELVs, only health risk considerations are taken into account, without considering technical-economic data.

• Outlook

The application of the deduced threshold values must consider the specific characteristics of each site, including PFAS background concentrations in the receiving water bodies. In addition, concentrations

must be accompanied by compatible mass and volumetric flow values, enabling environmental levels to be kept consistent with existing quality standards and an acceptable level of health risk.

On the other hand, it should be noted that among the list of PFAS initially studied, several of them do not have toxicological values available in the scientific literature that can be applied in a health risk study. In France, the organism Anses, in charge of food security, is currently conducting a project to establish and update TRVs for PFASs. The results are expected to be published in 2025. When available, these new data could lead to an update of the results of this study.

#### PFAS Immobilisation in Soil: How Long is Long Enough?

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#### **Innovative Nature**

Immobilisation is now considered a viable and cost-effective option for the management of PFAS contaminated soils. Long-term stability is the number one question that remains a key consideration to ensure peace of mind for future generations. Because we can only *simulate* what happens over decades, the data being produced by current test methods, and infield monitoring to date, is critical to answering the question; "How Long is Long Enough?". New data will be shown here.

#### Background/Objectives

Immobilisation of PFAS in soil is now considered a viable and cost-effective remediation approach. It involves *locking up* the contaminants in the soil enabling reuse of the treated soil onsite. Consistent with a Circular Economy approach, this allows construction projects to proceed without the need to halt the project to address 'contaminated site' issues.



#### Figure 1: Immobilisation of PFAS in Soil using Sorbents

This approach uses sorbents to reduce the leaching of PFAS into ground water and surface water reducing the risk to human health and the environment. It is particularly suitable for managing large soil volumes containing relatively low PFAS levels, where other thermal and washing techniques are not viable from a cost or capacity viewpoint.

Although the PFAS chemicals are rendered 'immobile' by this process, they still remain in the soil and so proving the long-term stability of the process is critical for giving peace of mind to future generations that the PFAS will not re-leach.

The question of robustness is particularly relevant where treated soil is to be reused on site as part of a circular economy approach which not only reduced costs, but also enables the beneficially reuse of otherwise healthy soil as a resource, avoiding unsustainable landfill disposal.

#### Approach/Activities

Independent data from Europe, USA and Australia will be presented that shows the stability of immobilised soils across a wide range of environmental conditions including pH, competing ions, freeze/thaw, wet/dry and repeated leaching events. A number of techniques for simulating long-term stability will be explored including the likes of DIN, the USEPA LEAF methods, MEP and TCLP-based methods.

Field-scale data from independent projects in Europe and the USA that have been monitored for up to 5 years will be presented as evidence of 'real-time' data from the field to validate lab-scale

simulations. Bioavailability data showing reduced uptake of PFAS into plants and earthworms will also be summarised.



Figure 2: Field data from a space base in North Dakota, USA, demonstrating that PFAS immobilisation using RemBind remains stable for over 17 months.

Divine et al. 2025. Field Demonstration of In Situ Stabilization (ISS) of PFAS in Soil with RemBind. J. Haz Mat., in review.

#### **Results/Lessons Learned**

The presentation of independent data from various projects around the world will put a case forward that immobilisation is, when applied correctly, a robust technique across a wide range of environmental conditions, as evidenced by both lab-scale simulations and some field demonstrations after 5 years of monitoring. Future considerations for developing better methods for testing the robustness of the immobilisation solution will be discussed, as will the importance of the sorbent/soil mixing process. Data showing the mixing efficiencies across a treated soil stockpile will be presented.

#### Characterizing PFAS in air emissions; monitoring methods & remediation challenges

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As the Flemish reference laboratory for environmental analyses, VITO represents the link between the Flemish government and the environmental laboratories in Belgium. In response to findings of elevated per- and polyfluoroalkyl compounds (PFAS) levels in various environmental media in Flanders, and the growing awareness that inhalation represents a significant human exposure route to PFAS, VITO was asked by the Flemish government to develop monitoring methods to quantify PFAS in the air compartment. Since 2021, VITO developed monitoring methods for the quantification of PFAS in emissions (Hofman et al., 2025b, Hofman et al., 2025a), ambient air and depositions (Peters et al., 2023).



Figure 1 Monitoring methods for PFAS in air emissions, ambient air and depositions

With regard to emissions, VITO derived a methodology from the US-EPA OTM-45 method published in 2021 (EPA, 2021, EPA, 2025), resulting in the first available regulatory method for the quantification of PFAS in guided emissions, into force in Flanders since 2024 (LUC, 2025). This method consists of isokinetic flue gas sampling with a heated or cooled probe and subsequent PFAS collection in a sampling train holding filter, XAD2 sorbents and water (impingers) fractions. Collected samples are extracted and analysed with LC-MS/MS (LUC, 2025). This method was validated extensively during an interlaboratory comparison in 2024, yielding 42 quantitative compounds with apparent compound recovery 70-120% and measurement uncertainty (k=2) <50% (Hofman et al., 2025a). Over the years, VITO gathered field experience by measuring ~50 polar PFAS compounds (C4-18) on a variety of stacks and industries, and at ambient background and hotspot locations throughout Flanders to explore

atmospheric PFAS prevalence, composition and gradients in Flanders. Today, analytical scope of these methods is extended to ultra-short chain (<C4) polar PFAS and non-polar PFAS compounds (canister + GC), identified using non-target screening techniques (DART-MS, HR-MS) on air and emission samples. Non-target results namely identified a range of ultra-short chain compounds (PFPrA, TFMS, TFA), poly-or H-substituted PFAS and long-chain PFCA's (up to C20) in emission samples and samples from residual streams (scrubber water, AC filters, bottom ashes,...).

Results indicate that a variety of "novel" PFAS compounds, beyond legacy or known PFAS, are being emitted and present in the ambient air. Although source contributions are generally lower than the temporary assessment framework applied in Flanders (0.3 ng/m<sup>3</sup> for the sum of PFOA, PFNA, PFHXS and PFOS (Vanhooren et al., 2024)), a lot of unknowns remain about the health risks of newly emerging compounds, byproducts and so-called products of incomplete combustion (PICs) (Weitz et al., 2024, EPA, 2024). We stress the urgent standardisation need for monitoring methods of PFAS in air emissions, in order to obtain robust and comparable results of emitted PFAS, prevailing concentration levels and impacts from abatement technologies. Remediation techniques should be evaluated holistically, both from an analytical (total PFAS (EOF/AOF), non-target analysis) and monitoring (representativity, residual streams/mass balance) perspective. Moreover, the environmental fate of PFAS emissions should be evaluated by comparing the contribution from guided and diffuse emission sources to atmospheric PFAS levels and resulting dry and wet deposition fluxes.

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#### Breaking the Cycle: The Power of Activated Carbon in PFAS Removal and Destruction

Johan Craeye, CTO, DESOTEC Johan.craeye@desotec.com Tel: +32/486.150.154

#### Abstract:

Per- and polyfluoroalkyl substances (PFAS) are a pressing global challenge due to their persistence in the environment and adverse health effects. France and Belgium were among the first countries to tackle this issue. DESOTEC, an international environmental services company, provides sustainable mobile filtration solutions based on activated carbon, a proven and effective method for PFAS removal from liquid and gaseous streams. Recognized as a Best Available Technology (BAT) under the Stockholm Convention (March 2021), activated carbon offers a versatile and robust approach to addressing PFAS contamination.

#### Case study:

A Belgian chemicals producer had to bring down the PFAS in its wastewater from 300 ng/l to 100 ng/l. Following some pilot tests, DESOTEC installed four filters after the client's water purification system. The first two are designed to adsorb organic pollutants other than PFAS. This avoided the saturation of the other two filters downstream, which are tailored to capture PFAS. As a result of our intervention, the company became fully compliant with the new lower PFAS limits.

After bringing back the spent carbon to its facilities, DESOTEC measured the adsorbed PFAS level using a method developed internally yet validated by authorities. Being below the POPs regulation limits, the PFAS-containing carbon was thermally treated, reactivating the carbon for reuse while destroying PFAS below detectable limits.

#### Activated carbon for PFAS removal:

Activated carbon's effectiveness is due to its highly porous structure and extensive surface area, enabling strong adsorption of PFAS molecules. However, the performance of any activated carbonbased filtration system depends on several key factors, including activated carbon characteristics, PFAS concentration and chemical properties, contact time and filter design, water matrix composition and filter configuration and monitoring.

#### What happens after the PFAS capture?

Beyond filtration, DESOTEC provides a comprehensive service encompassing filter takeback and rigorous adherence to regulatory standards. Saturated activated carbon undergoes detailed analysis to quantify PFAS content, ensuring compliance with the Persistent Organic Pollutants (POP) directive. This directive specifies strict limits for PFOS, PFOA, and PFHxS concentrations on spent carbon eligible for thermal reactivation, guaranteeing both regulatory alignment and operational integrity.

#### A new method to measure PFAS level adsorbed on activated carbon:

To address the limitations of traditional measurement methods, DESOTEC collaborated with VITO, a leading independent research institute, to develop an advanced extraction and analysis methodology for accurate PFAS quantification on spent activated carbon. This methodology enhances reliability and regulatory compliance, giving operators confidence in their waste management practices. It consists of a multi-step extraction procedure to optimize PFAS recovery from the spent carbon, followed by liquid chromatography–mass spectrometry (LC-MS) analysis.

#### Safe reactivation of PFAS-loaded spent activated carbon:

Once spent carbon is assessed, and meets all acceptance criteria, it is processed through thermal reactivation, an environmentally sustainable solution. During reactivation, under the right conditions, PFAS molecules are effectively destroyed through pyrolysis.

All along the reactivation process, PFAS molecules were closely monitored via targeted analysis:

- Levels were determined on the spent carbon going into the furnace
- All outgoing streams from the process (reactivated carbon, stack gasses and flue gas salts) were sampled

In addition, CF4 was monitored in the stack as a product of incomplete combustion.

DESOTEC's research, using these extensive analytics, demonstrates that PFAS level is below detection limit on the reactivated carbon, and in the stack gasses, further supporting this circular economy approach.

In parallel, the flue gas treatment systems integrated in our reactivation facilities, neutralize mineralization byproducts such as hydrogen fluoride and sulfur dioxide, ensuring safe and compliant operation throughout the process.

#### Conclusion:

This presentation will explore the interplay of these factors in optimizing PFAS removal and destruction using activated carbon. We will discuss the parameters influencing adsorption performance, the dual-filter approach, and the advanced PFAS measurement technique developed jointly by DESOTEC and VITO lab. Additionally, we will share detailed findings from our reactivation research, demonstrating the efficacy of PFAS destruction and the environmental advantages of carbon reuse. Through these insights, DESOTEC underscores its commitment to delivering sustainable, effective solutions to one of the most critical environmental challenges of our time.







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## L'EXPERTISE AU PROFIT DE L'ÉLIMINATION DES PFAS

Technologie de filtration avancée Charbon actif Résines échangeuses d'ions



#### Jeudi 19 juin 2025

09h00 Accueil des participants

#### Présidents :

• Dr. Rahul Singh, Chercheur associé au département d'Ingénierie mécanique, des Procédés et de l'Environnement - Université de Wismar, Allemagne

• Dr. Frank Karg, SFSE & ARET, Expert judiciaire, Directeur scientifique - HPC International, France & Allemagne

#### Technologies de traitement pour l'eau, les sols, les sédiments, l'air & les émissions

#### 09h30

Oxydation électrochimique pour l'élimination des PFAS dans l'eau contaminée Peter Eddowes, Chercheur principal en électrochimie - Arvia Water Technologies, Royaume-Uni

#### 10h00

Traitements in-situ des PFAS par lavage via des Bio-polymères protéiniques – Etudes de Cas en Belgique, Allemagne, Pays Bas et Suède

• Stephan Hüttmann, Directeur général - Sensatec, Allemagne

• Dr. Frank Karg, SFSE & ARET, Expert judiciaire, Directeur scientifique - HPC International, France & Allemagne

10h30 Pause-café & thé

#### 11h00

Aperçu technique de la technologie d'élimination des PFAS Selpaxt Dr. Karin Löfstrand, Responsable de la R&D - Chromafora, Suède

#### 11h30

Étude de la dégradation des PFAS par faisceau d'électrons dans l'eau et adsorbés sur une matrice de charbon actif

Stéphane Lucas, Directeur de l'innovation - Ion Beam Application / IBA, Belgique
#### 12h00

Évaluation de l'influence du vieillissement des sols contaminés par les PFAS sur l'efficacité du lavage : Défis et optimisations Mahshid Shahbazi Sehrani, Université de Nouvelle-Galles du Sud (UNSW), Australie

#### 12h30

Déjeuner

#### 13h45

Élimination durable des PFAS à des niveaux de contamination élevés et complexes Jürgen Buhl, Directeur des ventes - Cornelsen Umwelttechnologie, Allemagne

#### 14h15

Comparaison entre les boues activées conventionnelles et le bioréacteur à membrane pour l'élimination de la DCO des eaux usées polluées par des PFAS Valeria Mezzanotte, Professeur associé - Université de Milan-Bicocca, Italie

#### 14h45

Evaluation de méthodes de traitement pour l'élimination des PFAS présents dans les concentrats d'osmose inverse et de nanofiltration en traitement d'eau potable *Raphaëlle Du Besset, Ingénieur de recherche - SUEZ-CIRSEE, France* 

#### 15h15

Pause-café & thé

15h45

Fractionnement par moussage, un traitement efficient des PFAS Frédéric Bossert, Directeur d'agence adjoint - Züblin Umwelttechnik, France

#### Recherche et développement (R&D)

#### 16h15

Combinaison du regroupement chimique (Chemical clustering), de l'utilisation des modèles QSARS (Quantitative Structure Activity Relation) pour la cartographie chimique et toxicologique des PFAS *Philippe Oudin, Gérant - Semaco Environnement, France* 

#### 16h45

Étude des conditions expérimentales de défluoration du PFOA à l'aide d'un mélange DMSO/NaOH Raphaël Tur, Thésard - Colas Environnement / BRGM, France

#### 17h15

PFAS et ressources en eau : comment déterminer la meilleure des solutions de traitement ? Romain Gandré, Ingénieur Procédé - Veolia, France

#### 17h45

Fin de la troisième journée



#### Thursday, June 19, 2025

09:00 Welcoming participants

#### **Presidents:**

Dr. Rahul Singh, Research Associate, Department of Mechnical, Process & Environmental Engineering - University of Wismar, Germany
Dr. Frank Karg, SFSE & ARET, Court Expert, Scientific Director - HPC International, France & Germany

#### Treatment Technologies for Water, Soils, Sediments, Air & Emissions

09:30

Electrochemical Oxidation for the Elimination of PFAS from Contaminated Water Peter Eddowes, Senior Electrochemical Researcher - Arvia Water Technologies, United Kingdom

#### 10:00

Treatment of soil by in-situ PFAS washing with proteinic Bio-polymers – Feasibility studies from Belgium, Germany, the Netherlands and Sweden

• Stephan Hüttmann, Managing director - Sensatec, Germany

• Dr. Frank Karg, SFSE & ARET, Court Expert, Scientific Director - HPC International, France & Germany

10:30 Coffee & Tea break

11:00

Technical insight to Selpaxt PFAS removal technology Dr. Karin Löfstrand, Head of R&D - Chromafora, Sweden

#### 11:30

Study of PFAS degradation by e-beam in water and adsorbed on activated carbon matrix *Stéphane Lucas, Chief Innovation Officer - Ion Beam Application / IBA, Belgique* 

#### 12:00

Assessing the Influence of PFAS-Contaminated Soil Aging on Washing Efficiency: Challenges and Optimisations Mahshid Shahbazi Sehrani, The University of New South Wales (UNSW), Australia

#### 12:30

Lunch

#### 13:45

Sustainable removal of PFAS at high and complex contamination levels *Jürgen Buhl, Sales Director - Cornelsen Umwelttechnologie, Germany* 

#### 14:15

Comparison between conventional activated sludge and membrane bioreactor for the removal of COD from PFAS polluted wastewater Valeria Mezzanotte, Associate Professor - University of Milan-Bicocca, Italy

#### 14:45

Evaluation of treatment methods for the removal of PFAS from reverse osmosis and nanofiltration concentrates in drinking water treatment *Raphaëlle Du Besset, Research Engineer - SUEZ-CIRSEE, France* 

#### 15:15

Coffee & Tea break

15:45

Foam fractionation for efficient PFAS treatment Frédéric Bossert, Deputy Branch Manager - Züblin Umwelttechnik, France

#### Research & Development (R&D)

#### 16:15

Combination of chemical clustering and Quantitative Structure Activity Relation (QSARS) models for chemical and toxicological mapping of PFASs *Philippe Oudin, Managing Director - Semaco Environnement, France* 

#### 16:45

Investigation of experimental conditions of PFOA defluorination using DMSO/NaOH mixture *Raphaël Tur, PhD student - Colas Environnement / BRGM, France* 

#### 17:15

PFAS and water resources: How to determine the best treatment solution? *Romain Gandré, Process engineer - Veolia, France* 

#### 17:45

End of the third day

#### Electrochemical Oxidation for the Elimination of PFAS from Contaminated Water

Peter Eddowes, Senior Electrochemical Researcher - Arvia

Arvia Water Technologies presents an advanced and sustainable solution for addressing per- and polyfluoroalkyl substances (PFAS), also known as "forever chemicals," through innovative electrochemical oxidation technology. PFAS are notoriously persistent in the environment due to their robust carbon-fluorine bonds, accumulating in water sources, wildlife, and humans. Their widespread occurrence, originating from industrial processes, firefighting foams, household products, and landfill leachates, poses significant health risks, including cancers, liver diseases, thyroid disorders, and immune dysfunction.

PFAS separation methods have critical limitations. Adsorption processes such as GAC require frequent and costly media replacements but could effectively be used as a polishing step to reach regulatory targets on treated water that contains low concentrations. Membrane filtration concentrates contaminants without destroying them but generates a clean permeate. There are increased regulatory restrictions on conventional incinerators and some now refuse PFAS-containing waste due to operational conditions. Specialised incinerators are required operating at >1000°C with sophisticated gas scrubbing systems to manage toxic emissions.

Arvia's Nyex<sup>™</sup> electrochemical oxidation technology provides an efficient, sustainable PFAS destruction process. Central to this technology is the proprietary Nyex.3 electrode, featuring granular media with high surface area and the ability to generate hydroxyl radicals—one of the most potent oxidizing agents known. These radicals effectively cleave PFAS molecules producing carbon dioxide and fluoride.

Arvia addresses significant operational challenges inherent in electrochemical PFAS treatment. First, other electrodes deteriorate rapidly in high-fluoride environments generated by PFAS oxidation. However, Nyex.3 electrodes demonstrate superior fluoride resistance, maintaining stability and efficiency. Second, achieving destruction at low concentrations requires enhanced mass transfer efficiencies, facilitated by Nyex.3's three-dimensional electrode structure, substantially increasing surface contact and treatment effectiveness at low concentrations compared to flat plate electrodes.

The efficacy of Arvia's approach has been validated through comprehensive real-world applications. Using the Florenox<sup>™</sup> system, incorporating Nyex.3 electrodes, Arvia has consistently demonstrated over 99% removal efficiencies for high-concentration PFAS solutions, even amidst complex mixtures and solvent presence. For scalability, Arvia strategically integrates Florenox<sup>™</sup> with complementary separation technologies such as reverse osmosis, foam fractionation and adsorbent resin regeneration solutions supported by collaborations with innovative startups and academic institutions.

In summary, Arvia Water Technologies is spearheading the sustainable and scalable elimination of PFAS through electrochemical oxidation, effectively overcoming the limitations of traditional methods, ensuring regulatory compliance, and promoting environmental safety. For further information, please visit www.arviatechnology.com or contact us at: info@arviatechnology.com



International PFAS Congress – Paris 17 - 20 June 2025 HPC INTERNATIONAL SAS

## Traitements in-situ des PFAS par lavage via des Bio-polymères protéiniques – Etudes de Cas en Belgique, Allemagne, Pays Bas et Suède.

## Treatment of soil by in-situ PFAS washing with proteinic Bio-polymers – Feasibility studies from Belgium, Germany, the Netherlands and Sweden"

Stephan Hüttmann<sup>1</sup> / SENSATEC &

Frank KARG<sup>2</sup> / HPC INTERNATIONAL

(1) CEO of SENSATEC – Kiel / Germany,
 (2) CEO / HPC INTERNATIONAL SAS, Dr. Alfred-Herrhausen-Allee 12, 47228 Duisburg – Germany and HPC International SAS, H. de Recherche / Centre Médical de Perharidy, 29680 Roscoff, France Email: <u>frank.karg@hpc-international.com</u> / <u>Phone</u>: +33 607 346 916

#### Résumé

À l'heure actuelle, aucune technologie de dépollution in-situ n'est applicable aux contaminations par les PFAS dans les zones saturées et non saturées. Les stratégies de dépollution mises en œuvre appliquent principalement des méthodes très coûteuses telles que l'excavation des sols pollués (& mise en décharge, lavage hors site et traitement thermique) ou le P&T (pump-and-treat) très coûteux en tant que technologie de dépollution hydraulique des eaux souterraines.

En raison de leur grande solubilité, de leur stabilité chimique, de leur mobilité dans l'environnement et de leur bioaccumulation, les substances alkylées poly- et perfluorées (PFAS) représentent une préoccupation importante en ce qui concerne leur impact potentiel sur l'environnement et la santé. Dans toute l'Europe (Belgique, France, Allemagne, Italie et autres pays), de nombreux sites contaminés par des PFAS dans le sol, les eaux souterraines, les eaux de surface, etc. ont été identifiés, dont certains couvrent de vastes zones, comme la contamination de la couche arable de plus de 800 hectares dans la région de Rastatt (Baden-Württemberg, GER), où des boues de compostage de papier et de stations d'épuration contenant des PFAS ont été utilisées comme fertilisants. Un autre traitement de référence de lavage in-situ est **l'Aéroport de Kiel en Allemagne**.

L'objectif des traitements par lavage in-situ des zones non-saturées et saturés (aquifères) de la coopération d'HPC INTERNATIONAL avec Sensatec est le développement et la mise en œuvre d'un traitement alternatif de réhabilitation in-situ et sur site des PFAS des zones saturées et non saturées et des sols excavés, en utilisant un processus de lavage avec des composés de bio-polymères protéiniques biodégradables et des bêta-

1



cyclodextrines (cf. Fig. 1). Les ingrédients biopolymères tensioactifs spécialement développés sont utilisés pour séparer correctement les PFAS du sol.

Le lixiviat de lavage a également une action positive sur les eaux souterraines et est ensuite pompé de l'aquifère au moyen d'un système de lavage hydraulique (cf. Fig. 1).

Cette technologie permet l'extraction in-situ et/ou sur place et l'élimination ultérieure des PFAS monomères dans les sols superficiels et profonds sans recourir à des méthodes destructrices telles que l'excavation. Cette approche ne nécessite pas la séparation des différentes fractions granulométriques du sol.

Au cours de la mise au point du processus, l'efficacité de cette approche technique de dépollution est d'abord testée par une étude de faisabilité en laboratoire, puis par une application pilote sur le terrain. La première étape consiste à effectuer des essais à petite échelle sur des colonnes de percolation de sol, puis dans des lysimètres de sol et, après quelques semaines (3 à 4), dans le cadre d'une application pilote sur le terrain.

Les essais en colonne et en lysimètres ainsi que l'essai pilote sur site ont permis d'établir une liste plus large de paramètres relatifs aux PFAS, ainsi que le TOF et le Top Assay pour couvrir un maximum de PFAS perfluorés transformables en PFAS polyfluorés, afin de quantifier l'efficacité de la technologie d'assainissement.

Les acides carboxyliques perfluorés à chaîne courte (comme le PFBA, le PFPeA, etc.) peuvent être lessivés de des sols en utilisant simplement de l'eau comme ingrédient de lavage. Les acides carboxyliques à chaîne plus longue > C8 (PFOA, PFNA, PFDA, PFUnDA, etc.) et les acides sulfoniques (PFOS, PFNS, etc.) sont moins mobiles. La technologie de lavage avec des bio-polymères protéiniques prouve clairement que ces composés peuvent être mobilisés au moyen des biotensides spécialement développés. Des tests lysimétriques ont montré qu'une réduction de 95 % de la concentration en PFAS dans le sol est obtenue en appliquant la nouvelle technologie développée. Des essais sur le terrain ont permis de réduire les concentrations de PFAS de plus de 80 % en l'espace de trois semaines.



En comparaison avec l'analyse coût-avantage, les traitements de lavage in situ sont environ 30 fois moins chers que les excavations ou le P&T : Pump and Treat. La technologie est également applicable aux PFAS volatiles, comme les FTOH (alcools fluorotélomères, etc.).



Fig. 1 : Lavage du sol in situ pour la décontamination des PFAS avec des bio-polymères protéiques ou des bêta-cyclodextrines.

Le processus développé offre la possibilité de traiter les sols contenant des PFAS in situ dans la zone saturée et non saturée en mobilisant les PFAS et en traitant ensuite les eaux souterraines à l'aide d'une technologie de pointe. Une autre application possible de la méthode développée qui est actuellement appliquée sur le terrain est le traitement ex-situ des sols excavés contenant des PFAS (sur site et hors site) en utilisant les biopolymères protéiniques par lavage dans une technologie cyclique.

Les objectifs de traitement du lavage in situ visent à garantir la conformité des lixiviats et des conditions sanitaires, conformément aux objectifs d'un Plan de Gestion et des CMA (Concentrations Admissibles Maximales) d'une EQRS ou d'une ARR préventive ou prédictive, afin d'assurer la conformité sanitaire, pour une utilisation future du site.



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L'application pratique est illustrée dans la Fig. 2, comme s'était réalisé par ex. sur le site de l'Aéroport de Kiel en Allemagne.



Fig. 4 : Principe de l'application spécifique au site du lavage in situ (élution) des PFAS (Sensatec).

Si des zones insuffisamment perméables sont présentes dans le sous-sol (par exemple, des couches riches en argile), une perméabilisation préparatoire peut être réalisée par la technique TSE avec affleurement au sable (cf. Fig. 5).



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Fig. 5 : Technique d'injection TSE pour une éventuelle préparation de la perméabilité avant le lavage in situ (si nécessaire).

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#### SELPAXT<sup>™</sup> PFAS removal technology – Technical insight

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SELPAXT<sup>™</sup> is a membrane-based PFAS removal technology developed by Chromafora AB. The technique utilizing Surfactant-Ligand Assisted Membrane Filtration (SAMF) to selectively capture both long- and short-chain PFAS. Unlike adsorption-based methods, SELPAXT provides high and consistent removal rates with automatic regeneration, minimizing operational downtime and waste generation.

#### **Technical Overview**

The SELPAXT process operates similarly to any conventional ultrafiltration system but with the addition of proprietary SELPAXT chemicals. PFAS are selectively retained and concentrated by the membrane barrier. The PFAS, together with the SELPAXT chemicals, are concentrated into a small volume of approximately 0.1% of the water being processed. The concentrate, along with minor co-contaminants, is intermittently discharged from the membrane system during the regeneration cycle. Periodically, the system pauses production for an automatic clean-in-place (CIP) procedure to maintain optimal performance. The system operates at low pressure, reducing energy demand compared to RO or thermal methods. The process is scalable and works well in both bench scale as well as full scale.

#### **Performance and Demonstrated Results**

SELPAXT greatly reduces both short- and long-chain PFAS from several types of water, without being negatively affected by co-contaminants. The reduction efficiency is generally above 90%, even for C4-C6 PFAS. In addition, bench scale testing of removal efficiency for C1-C3 PFAS shows promising results, reaching 20-99% reduction.

Complex waters such as landfill leachate, firefighting residue, industrial wastewater and storm water from airports are among the different waters tested and show reductions of >80% for all analyzed C4-C10 PFAS substances as illustrated in Figure 1 below for a landfill leachate.



#### RECYCLING FACILITY - PFAS reduction, landfill leachate:



Figure 1: PFAS reduction in landfill leachate.

#### Conclusion

SELPAXT provides consistent and efficient PFAS removal without adsorption media exhaustion or frequent regeneration cycles. Its scalability, automatic operation, and resilience to cocontaminants make it a robust solution for challenging PFAS-contaminated water streams.

#### Study of PFAS degradation by e-beam in water and adsorbed on activated carbon matrix

Stéphane Lucas IBA Chief Research Officer (CRO) <u>Stephane.Lucas@iba-group.com</u> Mobile: +32 498 97.52.82

#### Introduction:

PFAS (per- and polyfluoroalkyl substances) are persistent environmental pollutants posing significant health risks. Traditional methods like incineration and activated carbon adsorption for PFAS treatment have notable limitations. Incineration is energy-intensive and can lead to incomplete degradation, releasing harmful by-products. Activated carbon, while effective in adsorbing PFAS, does not destroy them, requiring further disposal.

High energy electron beam treatment offers a compelling alternative, leveraging accelerated electrons to break down PFAS molecules efficiently. This method operates at ambient temperatures, ensuring energy efficiency, minimizes hazardous by-products, and achieves effective degradation, making it a promising, sustainable solution for PFAS remediation. This study explores the feasibility of high-energy electron beams (e-beam) for treating contaminated carbon filters and enabling their regeneration. Additionally, it evaluates the impact of e-beam treatment on filter integrity post-irradiation.

#### Materials and Methods:

Two types of samples were prepared: first tridistilled water spiked with either PFOA or PFOS at 500 ng/L, second Filtrasorb <sup>®</sup> 400-M activated carbon, 8g contaminated by 400 mL of a solution of PFOA or PFOS at 1mg/L, for 48 hours with stirring.

Different pH (neutral/alkaline) and dissolved oxygen concentration were tested.

Samples were irradiated at x different doses within 250 kGy to 2 MGy using a TT300 Rhodotron $^{\circ}$  10 MeV.

The irradiation levels were checked with alanine pellets.

After irradiation, PFASs have been analyzed in the water samples by Solid Phase Extraction (SPE) followed by LC-MS/MS (AquityIclass (LC) and Orbitab Q Extactive (MS/MS).

Irradiated activated carbon samples and reference were analyzed by SEM (Scanning Electron Microscope (SEM) FEG 7610F) and XRD (Inel equipment with a curved detector (120°, 4000 cells) to assess the impact of irradiation on the integrity of activated carbon.

A radio-kinetic model (cf figure 1) developed using Matlab<sup>®</sup> software, based on ordinary differential equations (ODE) related to the equations involved in the degradation mechanism of PFOA or PFOS by aqueous electron and OH• radicals, was developed to predict optimal degradation conditions.

#### **Results:**

Following the interaction of the beam electrons with water, free radicals and aqueous electrons are produced (water radiolysis). These aqueous electrons will launch the PFAS degradation mechanism [1]. Byproducts of irradiation include short-chain PFAS.

A constrained optimization of the rate constant for the reaction of the different degradation products with electron lead to a fit of the model with the experimental results (concentration of PFAS by-products as a function of the applied dose) as presented in Figure 1.

An alkaline pH with a low dissolved oxygen concentration appears to promote the degradation of PFOA and PFOS. This confirms the data presented in the literature [1], [2].



Figure 1: Radio-kinetic model fit (line) with experimental data (symbols).

#### **Discussion and Conclusion:**

The electron beam degradation process of PFOA and PFOS is a complex process that may involve different degradation pathways [2]. In order to increase treatment efficiency, optimal irradiation conditions must enable the main degradation pathway to be promoted. These different pathways can be included in the radio-kinetic model to predict the concentration of secondary pathway by-products and try to find conditions to minimize it.

The rate constants of the rate-limiting degradation reactions can be very different depending on the length of the chain. For example, for PFBA (C4 chain), the rate constant is much lower than for longer chain PFAS. The use of the radio-kinetic model allows the fast and easy exploration of irradiation parameter space to search for a plausible optimum irradiation condition. This allows reducing the number of experiments to do and to focus the experiment plan in the most promising region of parameter space.

This model is also accompanied by a clustering study based on the structure of the compounds, their physicochemical properties and their conformations in order to identify families of compounds which would present the same irradiation conditions to achieve the most effective treatment. Based on these clusters, PFAS representatives per group can be identified and used to guide the selection of compounds to be tested in order to feed the radio-kinetic model with relevant data to broaden the field of predictions to the different groups.

Currently, there are thousands of different PFAS compounds, it would be difficult to test them all exhaustively, so the use of modeling and clustering approaches seems to be suitable. This predictive modeling could be extended to other PFAS compounds in the future.

This approach seems to be promising in the search for optimal irradiation conditions. Based on these results, IBA is in parallel, working on an E-beam PFAS destruction solution based on an industrial accelerator, and that a treatment of ~10 kt of carbon per year is anticipated based on these results

Acknowledgments: We would like to thank the Aerial Research center for providing the Feerix facility

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## Assessing the Influence of PFAS-Contaminated Soil Aging on Washing Efficiency: Challenges and Optimisations (Impact of Soil Aging on PFAS Washing Efficiency)

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#### 1. Introduction

Per- and polyfluoroalkyl substances (PFAS) are a class of synthetic fluorinated chemicals noted for their persistence, bioaccumulation, and toxicity [1], [2], [3]. Among them, PFOS and PFOA are particularly concerning, contributing to approximately 80% of identified contamination sites globally [4]. PFHxS is an unusual exception in that its half-life is greater than both longer and shorter chain equivalents such as PFOS or PFBS[5]. Their widespread distribution, often via atmospheric deposition, and complex interactions with soil properties complicate remediation efforts[6], [7], [8], [9], [10].

While several treatment methods have been proposed, including soil washing, most studies assess their effectiveness using freshly spiked soils, which may not reflect real-world conditions [7], [11]. Limited research has addressed PFAS removal from aged or field-contaminated soils [12], [13]. However, the influence of soil aging on PFAS mobility and treatment efficiency remains underexplored.

This study addresses this gap by evaluating how aging affects PFAS removal efficiency in soil washing. We compare treatment performance in fresh, artificially aged, and field-aged soils, with and without washing agents, to inform more realistic and scalable remediation strategies.

#### 2. Methodology

#### 2.1. Soil Preparation

Clean soil was artificially contaminated using a methanolic solution containing 40  $\mu$ L PFOS, 18  $\mu$ L PFOA, and 30  $\mu$ L PFHxS stock solution (each at 100 mg/L) to simulate moderately low concentrations representative of real-world contamination. After evaporation and homogenisation, final concentrations reached 9.95(±0.15)  $\mu$ g/kg for PFOS, 3.63(±0.09)  $\mu$ g/kg for PFOA, and 4.61(±0.03)  $\mu$ g/kg for PFHxS.

#### 2.2. Aging and Experimental Design

Batch soil washing tests were conducted on three soil types: freshly spiked (FS), artificially aged (AS; stored 14 weeks under dark, dry, and cool conditions), and field-aged (AF; collected from a contaminated site with similar properties). A 10:1 water-to-soil ratio was used, with samples agitated at 200 rpm for 24 hours using a shaker. Two washing agents were tested: a proprietary surfactant (Surfactant 1) at the manufacturer's recommended dose, and Surfactant 2 based on its reported efficacy. All experiments were performed in triplicate.

#### 2.3. PFAS Quantification and Quality Control

PFAS concentrations were measured using UHPLC-MS/MS. Quality control was ensured by using procedural blanks and calibration verification standards after every ten samples.

#### 3. Results and discussion

#### 3.1. Baseline Washing Performance Without Additives

Figure 1a presents the results of washing without washing agents. While PFHxS removal remained high (up to 93.8% in AF soil), the removal efficiencies for PFOS and PFOA were notably lower in aged soils, highlighting the limited effectiveness of washing without washing agents. These findings confirm that soil ageing increases PFAS retention, particularly for PFOS, likely due to stronger sorption interactions.

#### 3.2. Enhanced Washing Using Chemical Additives

To improve PFAS removal, washing agents were introduced. According to figure 2, Surfactant 2 is a broadly effective washing agent, especially for aged soils. Surfactant 1 can be beneficial, particularly for PFOS and PFHxS in moderately aged soils, but is less effective for PFOA. Figure 1b compares the performance of two washing agents in AS soil. Surfactant 2 showed greater enhancement, especially for PFOA, compared to Surfactant 1. This difference is attributed to their distinct desorption mechanisms: Surfactant 2 removes PFAS by forming stable inclusion complexes, which more effectively disrupt PFAS–soil binding than the micelle-based solubilisation

mechanism of Surfactant 1. Additionally, Surfactant 2 maintains high removal efficiency in aged soils due to its low soil affinity and chemical stability in complex matrices.

The effectiveness of washing agents varies by PFAS type and soil aging, highlighting the need for tailored remediation strategies.







(1%w/w) (b)

#### 4. Conclusion

Soil ageing significantly reduces PFAS removal efficiency during washing without washing agents, particularly for PFOS (declining from 84.1% to 45.7%) and PFOA (from 95% to 60.5%). PFHxS, however, remains more extractable, with removal efficiencies exceeding 90% even in field-aged soils. Among the additives tested, Surfactant 2 achieved up to 90.8% PFOS and 66.9% PFOA removal in aged soils, clearly outperforming Surfactant 1. These findings underscore the importance of accounting for soil ageing in remediation planning. Strong performance of Surfactant 2 highlights its potential as a more effective and field-relevant washing agent for aged PFAS-contaminated soils. Future work should explore its optimisation and scalability in real-world applications.

#### 5. Acknowledgements

This work was supported by the CRC-P (Cooperative Research Centres Projects), Australian government. **References** 

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#### Sustainable Removal of PFAS at high and complex contamination levels

(Élimination durable des PFAS à des niveaux de contamination élevés et complexes) Jurgen BUHL (Sales Director), Cornelsen Group, Germany, buhl@cornelsen.group

#### Background

In some areas, challenging levels of PFAS are found in water. Sources of such water are, for example, the decontamination of extinguishing systems in vehicles or buildings if fluorine-free foams are subsequently to be filled into the system, as well as industrial wastewater or landfill leachate. High concentrations of PFAS, sometimes in combination with other components from the extinguishing foam or increased levels of dissolved carbon (DOC) or a complex water chemistry, make the treatment of such water very difficult or impossible for conventional techniques such as granulated activated carbon (GAC) or ion exchangers (IX).

#### Method

The analysis of the premix of an extinguishing agent commonly used in Germany revealed high levels of other fluoro-organic compounds (some precursors) as well as cationic and anionic surfactants in addition to PFAS. In the treatment of such a liquid, a pretreatment with an agent that specifically forms flocs with PFAS was compared with a classic treatment of the water using GAC and IX in a column system.

In another case, the floc-forming agent was used to test whether PFAS can be specifically removed from landfill leachate with complex chemism.

#### Result

The test with the premix showed that around 99% of the PFAS were removed by the formation of flocs and their filtration. However, very high proportions of the other fluoro-organic compounds and the anionic surfactants were also eliminated.

The comparative analysis made it clear that the approach without floc-forming pre-treatment initially achieves good cleaning performance for GAC and IX. However, the effluent values then increase unpredictably quickly and approach the level of the PFAS concentrations in the influent.

A combination of pre-treatment with floc formation and post-treatment with GAC/IX showed consistently low concentrations of PFAS in the effluent.

One leachate was characterized by 134 ppm TOC, 3790 ppm Cl and 4.4 ppb PFAS (e.g. PFOA, PFHxS, PFOS). An addition of the flocculating agent at a dosing rate of 100 mg/l resulted in a PFAS removal rate of 93.7% after filtration. A comparable removal rate for PFAS was also achieved for the concentrate from an RO system using the flocculating agent.

#### Applications

The results from the treatment of premix have led to fire engines now being decontaminated on a large scale. A rinsing solution with the floc-forming agent helps to remove the PFAS from the tank and pipe system on the one hand and to treat the resulting rinsing water on the other. PFAS values of less than one microgram are usually achieved as residual values in the vehicle's AFFF tank.

In addition, stationary extinguishing systems in warehouses or production buildings in Germany are currently also being intensively decontaminated. Initially, the work focused on the extinguishing centre of the system (AFFF tank, concentrate pipe), but now the work also includes the branched lines of the sprinkler system, where up to 130 m<sup>3</sup> of premix has already been encountered. Premix solutions and the resulting rinsing water often show PFAS values of >1,000 µg/l. The liquids are treated on site and then drained into the sewer.

As the pre-treatment of such rinsing liquid removes the majority of the PFAS load by forming and filtering PFAS-containing flocs, the need for GAC/IX for the post-treatment of such water is significantly reduced. Very low effluent values can be reliably achieved when treating the water. In addition, this pre-treatment significantly reduces the volume of PFAS-containing residues.

## COMPARISON BETWEEN CONVENTIONAL ACTIVATED SLUDGE AND MEMBRANE BIOREACTOR FOR THE REMOVAL OF COD FROM PFAS POLLUTED WASTEWATER

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Keywords: Municipal wastewater, Aqueous Film Forming Foam (AFFF), Biological treatment

#### Introduction

In the framework of a LIFE project (LIFE CAPTURE, 2022) a research was carried out to test and compare the performances of two kinds of biological processes to treat PFAS contaminated wastewater.

The project aims to test two treatment trains to remove PFAS from polluted soils and groundwater. In the treatment train for soil, PFAS are expected to be removed by Soil Foam Fractionation (SFF) and the resulting effluent to undergo biological treatment, removing oxidant demanding components leached from soil, and advanced chemical oxidation/photoelectric catalysis, followed by adsorption on activated carbon to remove PFASs. To this purpose, while SFF is not yet put in place, the first period of the research was dedicated to optimize at lab-scale the biological treatment of PFAS polluted wastewater in order to define the working conditions to be applied for the treatment of SFF effluent.

The reason to test the effect of the presence of PFAS on biological oxidation was the knowledge that such molecules can affect the growth and metabolism of bacteria (Shittu et al., 2023).

Till now very few papers have been published on the effects of PFASs on biological processes. However, this is a key issue in view of the recent European UWWTD imposing strict limits for contaminants of emerging concern (CEC), and the gradual adoption of quaternary treatments for wastewater treatment plants to obtain a minimum removal of 80% of selected CEC for the overall treatment train.

A commercial Aqueous Film Forming Foam (AFFF), containing PFAS, was used to artificially contaminate a municipal wastewater. AFFF containing fluorosurfactants are used to suppress fires of flammable liquids for aviation, military applications, refineries, oil rigs, and in general for fire and rescue services and are important sources of PFAS pollution all over the world (Myers et al., 2014) and, in particular, in the European sites where the results of Life project will be tested at pilot scale. They are made of water, a foam-forming agent, and PFAS, used as surfactants. They work by smothering the fire, depriving it of oxygen. Further, as they help cooling the fire, they can prevent re-ignition. Over 100 PFASs have been used in the formulation of AFFF (D'Agostino and Mabury, 2014).

#### Materials and methods

Tests were carried out using two lab-scale plants, provided by Idea Bioprocess Technology srls (Dalmine, BG, IT) on specific design, having a total volume of 4.5 L and a working volume of 2 L. Each reactor is equipped with probes for continuous monitoring of dissolved oxygen, pH and temperature (Hamilton, Reno, Nevada - USA). The two plants differ essentially for the separation of sludge which is settled in activated sludge (AS) and retained by a microfiltration membrane in MBR. This leads to different Hydraulic Retention Time and Sludge Retention Time and generates a clearer effluent, due to filtration (Figure 1).

Air bubbling was used to ensure that dissolved oxygen was not limiting during the trial. The reactors were fed in continuous with 8 hours hydraulic retention time (HRT) using independent peristaltic pumps. The effluent from each reactor was collected in dedicated tanks by gravity through thin Plexiglas tubes. To maintain the

concentration of Total Suspended Solids (TSS) between 1.5 and 2 g L-1 the activated sludge was recirculated daily.



Figure 1 – Lab-scale plants to compare conventional activated sludge system, on the left, and biomembrane reactor, on the right

The influent was a primary clarified municipal sewage, collected from a municipal wastewater treatment plant in Milano, spiked with AFFF at growing doses. Monitoring concerned COD, ammonia and nitrate Nitrogen.

AFFF was provided by the produced an analyzed for the concentration of selected PFAS. The analytical characterization of AFFF is reported in Table 1.

| Per- and Polyfluoroalkyl Substances (PFAS) | Concentration (mg L <sup>-1</sup> ) |
|--|-------------------------------------|
| 4:2-fluorotelomer sulfonate (4:2-FTS)      | 0.013                               |
| 6:2-fluorotelomer sulfonate (6:2-FTS)      | 8.274                               |
| 8:2-fluorotelomer sulfonate (8:2-FTS)      | <0.010                              |
| Perfluorobutanoic acid (PFBA)              | 0.046                               |
| Perfluoropentanoic acid (PFPeA)            | 0.011                               |
| Perfluorobutanesulfonic acid (PFBS)        | <0.010                              |
| Perfluoroesanoic acid (PFHxA)              | 0.132                               |
| Perfluoroeptanoic acid (PFHpA)             | <0.010                              |
| Perfluorooctanoic acid (PFOA)              | <0.010                              |
| Perfluorononanoic acid (PFNA)              | <0.010                              |
| Perfluorooctanesulfonic acid (PFOS)        | <0.010                              |
| Perfluorodecanoic acid (PFDeA)             | <0.010                              |
| Perfluoroundecanoic acid (PFUdA)           | <0.010                              |
| Perfluorotridecanoic acid (PFTrDA)         | <0.010                              |
| Perfluorotetradecanoic acid (PFTDA)        | <0.010                              |

Table 1 – Concentration of different PFASs in the AFFF spiked in wastewater

| Perfluorododecanoic acid (PFDoA)   | <0.010 |
|------------------------------------|--------|
| Perfluoroesansulfonic acid (PFHxS) | <0.010 |

The doses to be spiked were defined based on preliminary data concerning the PFAS extraction efficiency of foam fractionation in contaminated soils and on the concentration of the most abundant compound (6:2-fluorotelomer sulfonate, 6:2-FTS) in the AFFF used for the experimental trials and were 5, 10 and 20 mL/L of sewage. In the first period (blank) the plants were fed only on primary settled municipal sewage.

Real-time qPCR were performed to study the effect of growing doses of AFFF on the microbial community.

#### Results

The results show that COD removal efficiencies did not decrease with increasing AFFF doses both in AS and in MBR. The starting COD in the influent was quite low (as the influent was the effluent from primary settling) in the blank period, when no AFFF was added, and COD removal rate was quite constant, while the variability in terms of percent removal efficiency was higher. With the addition of AFFF the influent COD increased (approximately 12 mg/mL of AFFF) and the removal efficiency also tended to increase, as shown in Figure 2, except for the second dose (10 mL AFFF/L) period, during which the influent COD was lower and more variable (COD variations are also related to climatic events affecting the wastewater concentration).



Figure 2 – COD and NH<sub>4</sub>-N removal in the different experimental phases. (A) Percent efficiency, (B) Removal rate (mg/L/day)

Such observation agrees with the strong linear correlation between the influent COD (CODin) and the COD percent removal efficiency and removal rate (mg/L/day). Such result demonstrates that, at the tested doses, AFFF has no negative effect on COD removal, as also reported by Gonzalez et al. (2021) who worked with lower PFAS concentrations (maximum 2 mL/L of AFFF) with a 24 h HRT in 30 L sequencing batch reactors (SBR) under controlled conditions (T 15°C).

Some variations were observed both with time and with AFFF dose in the composition of the microbial community in both reactors, as shown in Figure 3, but this did not affect the good performance of COD removal.

On the contrary, a difference between the two processes can be observed for nitrification. The addition of AFFF had a significant impact on NH<sub>4</sub>-N removal, especially in the AS reactor. Such difference could be attributed to the long sludge retention time in the MBR, allowing for a gradual acclimation of ammonia oxidizing bacteria.



Figure 3 - Principal component analysis of the composition of bacterial populations in AS (Conventional Activated Sludge) and MBR (Membrane Bioreactor).

#### Conclusions

The conclusions drawn for the lab-scale experiments can be summarized as follows:

- No inhibition by AFFF was observed on COD removal in the lab-scale biological processes fed on primary clarified municipal sewage
- The microbial community changed but the bacteria maintained or improved their initial activity level for COD removal
- Nitrification was inhibited at the higher AFFF doses in the AS but not in the MBR system. However, expected NH<sub>4</sub>-N concentration in foams from SFF are very low and would not affect the performance of any further treatment.
- MBR was slightly more efficient for COD and Nitrogen removal at higher AFFF concentrations. This could lead to address towards MBR technology for liquids with high PFAS concentrations, but MBR present different management issues correlated with the formation of foam, due to the strong aeration needed to avoid the membrane clogging.

The absence of negative effects is particularly important in view of the application of the recent European UWWTD which will gradually impose the adoption of quaternary treatments to wastewater treatment plants processing more than 150,000 PE or, in specifically critical cases, also in smaller plants. Of course, a good performance of biological process is a key issue for a good performance of quaternary treatments.

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## Evaluation of treatment methods for the removal of PFAS from reverse osmosis and nanofiltration concentrates in drinking water treatment

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#### SUMMARY

Pollution by PFAS is significant and worldwide: it includes thousands of compounds present in all segments of the environment (water, soil, air). Because of their proven toxicity to humans, drinking water regulations are being introduced in many countries, including France, with a wide variety and concentration of PFAS involved.

Membrane technologies are currently the most effective for eliminating a wide range of PFAS (particularly those poorly retained by conventional treatments such as adsorption), but this technology has the disadvantage of having to manage its reject, the concentrate.

There are solutions for treating these concentrates before they are released into the environment, and we need to refine our knowledge of the performance of these solutions and the factors influencing them. This study enables us to position technically adapted solutions for the treatment of membrane concentrates, and thus to propose complete treatment processes that (1) remove PFAS from drinking water and (2) protect the quality of the resource.

Like the drinking water treatment line, the selection of the best treatment line must be determined on a case-by-case basis according to technical, economic and environmental criteria.

**KEYWORDS**: Low Pressure Reverse Osmosis, Nanofiltration, PFAS, Activated Carbon, Resin, Brine, Foam fractionation

#### Context

PFAS are organic contaminants present worldwide, in both wastewater and drinking water, due to their stability in the environment. In view of concerns about their effects on the environment and health, many countries have established regulations for these compounds or are working towards doing so. For example, in France, the limit for the sum of 20 PFAS below 100 ng/L is considered for drinking water, following the transposition of the European Directive. In addition, a limit for the sum of 4 PFAS below 20 ng/L has been suggested by the high council for public health. Regulations concerning the type of PFAS and their concentrations in discharges are currently being studied. In addition to long-chain PFAS, short-chain PFAS are increasingly present in the environment because of long-chain PFAS degradation and new product industrial development: they are less acutely toxic, but more soluble in water and-often more persistent. As a result, water resources are contaminated by a wide variety of PFASs, with different carbon chain lengths, properties, and treatability. The main treatments dedicated to eliminating them for drinking water application, are adsorbent materials, ion exchange resins, nanofiltration or reverse osmosis membranes. The most effective technologies for eliminating a range of PFAS with varying carbon chain lengths are nanofiltration or reverse osmosis membranes. However, this process generates a reject, the concentrate containing all the PFAS removed by the membrane at a concentration 5 to 10 times higher (see Figure 1). Discharge of these concentrates into the environment, is not a satisfactory option, even though it is not currently restricted by current regulations in France. Few studies have been dedicated to the treatment and the fate of membrane concentrates. As a result, concentrate management is still a major obstacle to the full-scale application of membrane technologies for PFAS treatment. The aim of this project is therefore to evaluate a range of technical and economic solutions for eliminating PFAS from concentrates prior to discharge into the environment. In the absence of regulations, the project: (i) focused on the list of PFAS targeted in the ministerial decree dedicated to the monitoring of ICPE (Installations Classées pour la Protection de l'Environnement) discharges; (ii) positioned the concentration versus drinking water limits and NQE values post-dilution (being discussed in the revision project of the Water Framework Directive).

#### Methodology & results

Treatment tests were carried out on a set of 17 PFAS from the ministerial decree on PFAS analysis for ICPE discharges (see Table 1). The tests were carried out at laboratory and pilot scale on real concentrates, produced by a pilot plant from groundwater and subsequently spiked with PFAS. Total PFAS concentration was around 6-10  $\mu$ g/L. PFAS were analyzed by direct injection high-performance liquid chromatography-mass spectrometry using a protocol adapted from draft standard prEN 17892:2022. The tests were used to qualify PFAS removal performance for various treatments, based on (i) ion exchange (PFAS specific resin), (ii) adsorption (activated carbon and other media), and (iii) physical (foam fractionation) (see figure 1).

Preliminary results show a strong impact of PFAS physico-chemical characteristics (mass, charge, sulfonic or carboxylic acid form...), with very low removal efficiencies for short-chain PFAS and excellent efficiencies for long-chain PFAS. Intermediate PFASs show variable results. Tests on the influence of matrix, organic matter content (in the range 5-15 mg/L as TOC level) and salinity (in the range of 1500 - 3000  $\mu$ S) were also carried out. Main parameters impacting the performance of PFAS removal were also studied (type of media, media dosage, contact time, type of membrane, air/water ratio....) for each process.

Results produced from lab-scale tests will be confirmed during a next step through a pilot-scale experimentation. These tests will confirm the performances, optimize the operating conditions and support the selection of the best available technology for concentrate treatment.

| Name            | N°CAS       | Nb C | RPF (*) (eq PFOA) |
|-----------------|-------------|------|-------------------|
| PFBA            | 375-22-4    | C4   | 0,05              |
| PFPeA           | 2706-90-3   | C5   | 0,03              |
| PFHxA           | 307-24-4    | C6   | 0,01              |
| PFHpA           | 375-85-9    | C7   | 0,505             |
| PFOA            | 335-67-1    | C8   | 1                 |
| PFNA            | 375-95-1    | C9   | 10                |
| PFDA            | 335-76-2    | C10  | 7                 |
| PFUnDA ; PFUnA  | 2058-94-8   | C11  | 4                 |
| PFDoDA ; PFDoA  | 307-55-1    | C12  | 3                 |
| PFBS            | 375-73-5    | C4   | 0,001             |
| PFPeS           | 2706-91-4   | C5   | 0,3005            |
| PFHxS           | 355-46-4    | C6   | 0,6               |
| PFHpS           | 375-92-8    | C7   | 1,3               |
| PFOS            | 1763-23-1   | C8   | 2                 |
| PFDS            | 335-77-3    | C10  | 2                 |
| HFPO-DA - Gen X | 13252-13-6  | C6   | 0,06              |
| ADONA           | 919005-14-4 | C7   | 0,03              |

Table 1: List of PFAS spiked

(\*) RFP : Ratio Potency Factor





Table 2: Examples of laboratory scale elimination performances of PFAS in brine treatment

| PFAS (by size)               | Powdered Activated<br>Carbon<br>(Jar-test / Dose = 50<br>ppm / CT = 30 min) | <b>PFAS specific resin</b><br>(Isotherm / Dose =<br>500 ppm / 24 h) | Foam Fractionation |
|------------------------------|---|---|--------------------|
| XS<br>(MW < 300 g/mol)       | <20%  | 40 - 80%  | <20%               |
| S<br>(MW [300-400]<br>g/mol) | 0 - 60%   | >70%  | 10-90%             |
| M<br>(MW [400-500]<br>g/mol) | 40 - 90%  | >90%  | 80 - 99%           |
| L<br>(MW > 500 g/mol)        | >80%  | >95%  | >95%               |

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#### Foam fractionation for efficient PFAS treatment

Frédéric Bossert (Deputy branch manager), Hans-Georg Edel (Manager R&D)

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#### **Introduction**

PFAS contamination in aqueous media differs in terms of the composition and concentration of the individual compounds, depending on the cause of the damage. In order to ensure efficient and cost-effective, but also sustainable removal of PFAS, specific processes or combinations of processes must therefore be selected for each individual case and appropriate systems designed. In particular, the varying qualities of groundwater, leachate, process water or fire extinguishing water must be considered.

Since 2009, Züblin Umwelttechnik GmbH has planned, built and operated around 70 customised PFAS water and soil treatment plants at various locations in Germany, Switzerland, France and Italy [1, 2, 3].

#### Foam fractionation

A patent application for the foam fractionation process was filed back in 1918. Up to now, it has mainly been used to separate surfactants and proteins in the metal and food industries and in fish farming. The removal of PFAS from aqueous media using foam fractionation is a comparatively new application. Intensive aeration produces a foam, to whose air-water interface the PFAS adsorptively attach. The separated foam contains high concentrations of PFAS, which must be disposed of respectively destroyed in high-temperature incineration.

As part of an internal R&D project, the foam fractionation of leachate, groundwater, fire extinguishing water and soil washing water with high PFAS contents in different compositions was investigated using a laboratory plant and a pilot plant (*Fig. 1*). The process is robust and particularly suitable for long-chain PFAS; short-chain PFAS can be partially removed by adding a surfactant (*Fig. 1*). The results and experience to date show that foam fractionation can be used efficiently and economically for high PFAS concentrations alone or in combination with activated carbon.



**Fig. 1**: PFAS foam fractionation, leachate treatment Bavaria (left), residual contents  $\Sigma PFAS_{13}$  after foam fractionation without/with addition of surfactant (right) [2]

#### Efficient treatment processes

The selection of an efficient process for treatment of water containing PFAS depends largely on the PFAS content present. If only low concentrations in the range of approx. 0.1-10  $\mu$ g/L are present, activated carbon adsorption as a basic technology is the most efficient and cost-effective purification process.



**Fig. 2**: Process stages for cleaning water with a high PFAS content, schematic: Gravel filter  $\rightarrow$  Foam fractionation  $\rightarrow$  Activated carbon adsorption  $\rightarrow$  Ion exchange [2]

At PFAS-contaminated sites, groundwater from hotspots, leachate, fire extinguishing water and process water can contain high PFAS levels in the range of 50-500  $\mu$ g/L, sometimes even higher. In these cases, activated carbon adsorption is uneconomical due to the high consumption of operating materials. Instead, a process combination of foam fractionation as the main treatment stage and downstream fine cleaning using activated carbon filters and/or ion exchangers is recommended (*Fig. 2, 3*). In this way, the low purification targets for PFAS [4] are not only met safely but also economically.



**Fig. 3**: Apparatus for efficient PFAS removal: Foam fractionation (left), Activated carbon adsorption (centre), Ion exchange (right)

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#### Investigation of experimental conditions of PFOA defluorination using DMSO/NaOH mixture

Raphael Tur<sup>1,2</sup>, Stéphanie Betelu<sup>1</sup>, Romain Rodrigues<sup>1</sup>, Stéfan Colombano<sup>1</sup>, Dorian Davarzani<sup>1</sup>, Sébastien Bristeau<sup>1</sup>, Julien Grandclément<sup>2</sup>, Arnaud Perrault<sup>2</sup>, Julie Lions<sup>1</sup>, Eric Van Hullebusch<sup>3</sup>, Ioannis Ignatiadis<sup>1</sup> <sup>1</sup> BRGM, French geological survey, Orléans, France <sup>2</sup> Colas Environnement, Lyon, France <sup>3</sup> IPGP, Institut de Physique du Globe de Paris, Paris, France

Corresponding author email: <a href="mailto:raphael.tur15@gmail.com">raphael.tur15@gmail.com</a>

Keywords: PFOA, Dimethyl sulfoxide, NaOH, defluorination, by-products, pathways

Goal: Investigation and optimization of a new PFAS removal technology for potential field application

#### ABSTRACT

Per- and polyfluoroalkyl substances, PFAS, are a large group of around 5,000 synthetic chemical compounds widely used in industrial and consumer applications since the 1950s, most usually where extremely low surface energy or surface tension and/or durable water- and oil-repellence is needed, i.e. fire-fighting foams, surface treatment of textiles. Their persistent nature results in diffuse pollution issues in the environment and adverse health effects.

Trang et al. investigated low-temperature thermal treatment (80-120°C) of perfluorooctanoic acid, PFOA (36 g/L), using a mixture of dimethyl sulfoxide, DMSO (145/1 DMSO/PFCA molar ratio), sodium hydroxide, NaOH (30/1 NaOH/PFOA molar ratio) and milliQ water (8/1 DMSO/water volume ratio). Based on density functional theory calculations and by-products analyses, the authors proposed PFOA degradation mechanism during which the joint action of temperature, aprotic, polar and nucleophile solvent results in fluoride ions, F<sup>-</sup> (90% vs total F provided by PFOA in 24 hours), carbonate, formate, oxalate, glycolate, tartronate and trifluoroacetic acid CF<sub>3</sub>COOH (TFA). We pursued the optimization of the degradation process with the ultimate goal of proposing a practical solution for addressing PFOA environmental contamination and its supposed main degradation by-product, TFA.

PFOA doping concentration ( $C_0$  = 893 mg/L) was selected to decrease the dilution factor to analyse PFOA and its by-products at the µg/L level, while keeping optimum conditions for measuring fluoride by potentiometric titration, at the mg/L level, in the range from 20 mg/L to 20 µg/L. The degradation kinetics of PFOA was investigated over a period of six days at 120°C. The influence of the DMSO/water volume ratio as well as the NaOH/PFOA molar ratio on the treatment efficiency was studied to minimize DMSO volume while optimizing PFOA and by-products degradation.

PFOA defluorination process was verified by Ultra-High-Pressure Liquid Chromatograph coupled with Mass Spectrometer (UPLC-MS), potentiometric titration and SEM-EDS. UPLC-MS analysis was used to quantify PFOA, perfluoroheptanoic acid (PFHPa), perfluorohexanoic acid (PFHxA), perfluoropentanoic acid (PFPeA), perfluorobutanoic acid (PFBA), perfluoropropanoic acid (PFPrA), trifluoroacetic acid (TFA) and qualify by-products of the following masses:  $325 \pm 0.5$ ,  $307 \pm 0.5$ ,  $275 \pm 0.5$ ,  $257 \pm 0.5$ ,  $229 \pm 0.5$ ,  $225 \pm 0.5$ ,  $207 \pm 0.5$ ,  $175 \pm 0.5$  and  $157 \pm 0.5$ . Fluoride selective electrode was used for F<sup>-</sup> analysis. SEM-EDS allowed us to characterize NaF on dried surface of a PFOA/DMSO/NaOH mixture ( $20 \mu$ L) after interaction.

Experiments conducted at 120°C highlighted a complete PFOA removal after six hours, a total PFAS mineralization after twelve hours of reaction ( $\geq$  96 % of fluoride) and the necessity of maintaining at least four times larger the DMSO/H2O volume and sixty times to one higher the NaOH/PFOA molar ratio to ensure a complete PFAS defluorination while minimizing by-products. In a partial PFAS removal condition, with only a NaOH/PFOA molar ratio at 31:1, by-products were identified and PFHpA, PFHxA, PFPeA fell below the limit of quantification after 30 minutes; PFBA after 12 hours, while PFPrA, TFA and by-products with masses 325 ± 0.5, 275 ± 0.5, 229 ± 0.5, 225 ± 0.5 and 175 ± 0.5 were declining versus time. Furthermore, in this condition, the by-products with masses 307 ± 0.5, 257 ± 0.5, 207 ± 0.5 and 157 ± 0.5

were identified as persistent by-products and three PFAS defluorination pathways were proposed to illustrate the PFAS removal mechanism in the DMSO/NaOH system.

**Acknowledgements:** This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 101036449. S. Borensztajn done SEM-EDS analyses.

#### References:

(1) Trang, B.; Li, Y.; Xue, X.S.; Ateia, M.; Houk, K.N.; Dichtel, W.R. Science 2022, 377, 839.

#### PFAS AND WATER RESOURCES: HOW TO DETERMINE THE BEST TREATMENT SOLUTION?

#### Romain GANDRÉ Veolia Research & Innovation, Aubervilliers, IDF, France

#### <u>ABSTRACT</u>

Per- and polyfluoroalkyl compounds (PFAS) are a wide range of molecules extraordinarily stable in the environment, making them difficult to treat (especially short-chain PFAS), and also to degrade given their strong chemical structure. Thus, Veolia has developed a lab-scale test methodology in order to determine the best adsorption treatment line, one of the most effective and easy-to-deploy solutions. This method has been compared with others widely used, such as the RSSCT, as well as with industrial real-life, demonstrating in the end its reliability and robustness in predicting PFAS treatment performance and designing the processes needed to tackle this fundamental challenge.

#### **INTRODUCTION**

This document sums up the work carried out to demonstrate the applicability of the methodology developed by Veolia, called Diabolo, for the definition of targeted PFAS treatment trains based on adsorption. A comparison with other accelerated methods (*e.g.* RSSCT) to generate the necessary and sufficient data for industrial scale-up was also conducted to position the latter in terms of reliability with regard to industrial reality.

#### METHODOLOGY/ PROCESS

The experimental approach developed at lab-scale to address the treatment of PFAS, called Diabolo, consists of three phases:

- Phase 1: Screening This phase consists of a rapid comparison of several adsorbents. A ranking is established according to their affinity for the targeted PFAS and their ability to treat them, leading to the choice of the most promising for the next phases, which are the core of the methodology.
- Phase 2: Isotherms Thermodynamic aspect This phase involves comparing adsorbents previously selected under 'ideal' conditions to explore their theoretical maximum adsorption capacities towards the targeted PFAS, allowing the maximum lifetimes (or doses) of the adsorbents to be estimated.
- Phase 3: Mini-Column Kinetic aspect This phase continues the evaluation of adsorbents in a fixedbed configuration under conditions that are more representative of the reality of an industrial unit. It enables a more realistic estimate to be made of the expected lifetimes of adsorbents as a function of the real rate of use of their adsorption capacities, taking into account the contact time, since not all media active sites are accessible to contaminants in an industrial context.

All the data generated along the different phases are used as input for physical models (Freundlich and Langmuir adsorption models, molecular diffusion model, etc...), allowing, in the end, to draw up the breakthrough curves of the adsorbents towards the targeted PFAS in real water matrices. Besides, by performing these tests in batches to determine these key parameters (adsorption law and kinetics), the Diabolo methodology allows for the simulation of long-term field or pilot testing in a much shorter time frame. Diabolo has been compared with industrial scale, and the RSSCT (Rapid Small Scale Column Test) accelerated method.

RSSCTs are widely used to predict the removal of PFAS by adsorbents. The water passes through a small column of ground adsorbents (unlike Diabolo, where the adsorbent is not ground). The principle is based on the fact that by grinding the adsorbents, the breakthrough of PFAS will be observed faster. The generated

data are then extrapolated to the industrial scale by using a factor calculated according to several parameters from the full-scale unit and the design of the RSSCT column.

All of the presented work has been conducted on a drinking water matrix, more specifically on an LPRO (Low Pressure Reverse Osmosis) concentrate spiked with eleven (11) PFAS chosen among the short and long-chain PFCAs and PFSAs families (sum of 11 PFAS around 120 µg PFAS-11/L).

#### **RESULTS/ OUTCOMES**

A large number of adsorbents were evaluated and selected from the following families: powdered activated carbons (PAC), granular activated carbons (GAC), micrograin activated carbons (MGAC), and anion exchange resins (AER). The results presented here will focus on the best GAC evaluated in the first phase. Adsorption isotherms for each of the PFAS were carried out for the best GAC, allowing preliminary estimates of lifetimes to be made. Lastly, mini-column tests were performed to determine the mass transfer coefficients for each of the PFAS. The breakthrough curve for the sum of the PFAS (PFAS-11) was finally drawn up for different contact times. These predicted breakthrough curves were compared with those obtained from the RSSCT tests, and then both with the measured PFAS breakthrough curve obtained with the industrial pilot.

A comparison of the two lab-scale accelerated methodologies for predicting PFAS treatment performance by adsorption showed a good correlation with the industrial reality using the continuous column pilot unit for the Diabolo approach and a poor correlation with the RSSCT methodology. Indeed, the predictions of the PFBA breakthrough curves with Diabolo methodology have an uncertainty evaluated at around 20% for PFBA, and around 50% with RSSCT.

Thus, beware of the RSSCT methodology, for which other REXs (based on over 35 studies) of comparison with full-scale have demonstrated prediction errors of over 20% more than 90% of the time. This method requires calibration of the data and adjustment of the extrapolation factor with full-scale operational data to gain significantly in accuracy.

Finally, the development of this Diabolo methodology has also enabled us to evaluate multi-stage treatment processes to address the problem of PFAS in water matrices, such as a GAC stage followed by an AER, or very promising alternative media followed by a GAC stage, for instance.

#### **CONCLUSION**

All the work presented here has demonstrated the reliability of the Diabolo methodology for predicting the performance of PFAS treatment processes in water matrices, making it possible in a very short period to generate sufficient data to assess the renewal rates of adsorbents while recommending the best contact times to be applied in the design of process units. The responsiveness that the PFAS issue requires from water managers and services is thus fulfilled with such laboratory methods available, whose robustness has been proven, while bearing in mind the uncertainty associated with them. In this way, the treatment lines to be deployed will be implemented even faster and more securely.



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# Vendredi 20 juin 2025

09h00 Accueil des participants

#### **Présidents :**

• Stéfan Colombano, Ingénieur/Chercheur - BRGM, France

• Dr. Rahul Singh, Chercheur associé au département d'Ingénierie mécanique, des Procédés et de l'Environnement - Université de Wismar, Allemagne

• Dr. Frank Karg, SFSE & ARET, Expert judiciaire, Directeur scientifique - HPC International, France & Allemagne

#### Gestion générale des PFAS en Environnement & Santé publique

#### 09h30

Approche couplée terrain et modélisation, application à une contamination PFAS *Jérôme Texier, Ingénieur R&D - XSEM, France* 

#### 10h00

Panoplie des technologies des traitements de dépollution des sols, eaux et de l'air existants concernant les PFAS *Frank Karg, Directeur Scientifique - HPC International, Allemagne & France* 

10h30 Pause-café & thé

#### 11h00

Exposition aux PFAS : Approche One Health dans la zone anthropisée de Fos-Berre. Étude de la contamination et de la toxicité des PFAS dans les eaux de consommation et développements analytiques en vue de la biosurveillance lichénique des contaminations atmosphériques

• Maëlys Dijoux, Doctorante - ABO-ERG Environnement / Aix-Marseille Université, France

• Melissa Denton, Institut Méditerranéen de Biodiversité et d'Écologie Marine et Continentale (IMBE), France

#### 11h30

Relargage des substances per et polyfluoroalkylées (PFAS) des sols vers les eaux souterraines : importance de la famille de PFAS et de la substance Julien Michel-Malfait, Ingénieur d'études et de recherche - Institut National de l'Environnement Industriel et des Risques (INERIS), France

#### 12h00

Dégradation des PFAS pendant l'incinération des déchets dangereux : Une étude à l'échelle pilote avec une approche analytique complète *Gaël Muckensturm, Ingénieur de recherche - Veolia Recherche et Innovation, France* 

#### 12h30

Déjeuner

#### 13h30

Retours d'Expérience sur le remplacement des émulseurs fluorés dans différents systèmes anti incendies : de la caractérisation aux traitements des déchets Matthieu Hirrien, Responsable Cellule Design, Développement et Expertise - SARPI ThinkTech, France

#### 14h00

Deux études de cas illustrant la dépollution in situ de concentrations très élevées de PFAS dans la nappe en zones sources Mariangela Donati, Responsable de région - Regenesis, Italie

#### 14h30

Effet des caractéristiques du sol et des produits chimiques mobilisateurs sur la désorption de l'acide perfluoroalkyle (PFAA) lors du rinçage du sol *Arne Vangansbeke, Doctorant - KU Leuven, Belgique* 

15h00 Pause-café & thé

#### Recherche et développement (R&D)

#### 15h30

Remédiation des sols contaminés par les PFAS à l'aide de biopolymers - Traitement in situ d'un site aéroportuaire contaminé par l'AFFF Anja Wilken, Directeur de projet - Sensatec, Allemagne

#### 16h00

Traitement des PFAS des eaux usées industrielles par procédé de réduction avancée et électrooxydation Baptiste Mathon, Ingénieur R&D - Treewater, France

#### 16h30

Étude comparative de filières de traitement en vue de l'élaboration d'une solution adaptée aux caractéristiques de la pollution aux PFAS dans la ressource en eau et à la production d'eau potable

• Stéphanie Dugenest, Ingénieur référent Eau et Déchet - Antea, France

• Laure Brichet, Chargée de mission des ressources en eau – Agglomération du Grand Annecy, France

#### 17h00

Fin de la quatrième journée / Fin de la conférence



# Friday, June 20, 2025

09:00 Welcoming participants

#### **Presidents:**

• Stéfan Colombano, Engineer/Researcher - BRGM, France

• Dr. Rahul Singh, Research Associate, Department of Mechanical, Process & Environmental

Engineering - University of Wismar, Germany

• Dr. Frank Karg, SFSE & ARET, Court Expert, Scientific Director - HPC International, France & Germany

### **General management of PFAS in Environment & Public Health**

#### 09:30

Coupled field and modeling approach, application to PFAS contamination *Jérôme Texier, R&D Engineer - XSEM, France* 

#### 10:00

Overview of existing PFAS treatment technologies concerning soils, water and air *Frank Karg, CEO & Scientific Director - HPC International, Germany & France* 

10:30 Coffee & Tea break

#### 11:00

Exposure to PFAS: One Health approach in the anthropised area of Fos-Berre. Study of contamination and toxicity of PFAS in drinking water and analytical developments for lichen biomonitoring of atmospheric contamination

- Maëlys Dijoux, Ph.D Student ABO-ERG Environnement / Aix-Marseille University, France
- Melissa Denton, Research engineer in biological techniques Mediterranean Institute of Biodiversity and Marine and Continental Ecology (IMBE), France

#### 11:30

Release of Per- and Polyfluoroalkyl Substances (PFAS) from Soil to Groundwater: Importance of PFAS Family and substance

Julien Michel-Malfait, Study and Research Engineer - French National Institute for Industrial Environment and Risks (INERIS), France

#### 12:00

PFAS degradation during hazardous waste incineration: A pilot-scale study with comprehensive analytical approach *Gaël Muckensturm, R&D engineer - Veolia Research and Innovation, France* 

#### 12:30

Lunch

#### 13:30

Returns of experience for the replacement of fluorinated afff in different firefighting systems : from characterization to waste treatment *Matthieu Hirrien, Design, Development and Expertise Manager - SARPI ThinkTech, France* 

#### 14:00

Two case studies demonstrating in situ groundwater PFAS remediation within high concentration source areas Mariangela Donati, District Manager - Regenesis, Italy

#### 14:30

Effect of soil characteristics and mobilizing chemicals on perfluoroalkyl acid (PFAA) desorption during soil flushing Arne Vangansbeke, PhD Student - KU Leuven, Belgium

15:00 Coffee & Tea break

#### Research & Development (R&D)

#### 15:30

Biopolymer based remediation of PFAS contaminated soils - In Situ Treatment of an AFFF Contaminated Airport Site Anja Wilken, Head of Project Management - Sensatec, Germany

#### 16:00

Treatment of PFAS from industrial wastewater using advanced reduction and electro-oxidation processes Baptiste Mathon, R&D Engineer - Treewater, France

#### 16:30

Comparative analysis of treatment processes to develop a solution adapted to the characteristics of PFAS pollution in water resources

- Stéphanie Dugenest, Water and waste Engineer Antea France
- Laure Brichet, Water Resources Officer Greater Annecy Agglomeration, France

17:00 End of the fourth day End of conference



#### 5th International Congress, 17, 18, 19 and 20 June 2025 - Paris, France

# Coupled field and modeling approach, application to PFAS contamination Approche couplée terrain et modélisation, application à une contamination PFAS

#### Jérôme TEXIER<sup>1</sup>, Sara PETITJEAN<sup>1</sup>, Loïc ESNAULT<sup>1</sup>

<sup>1</sup> XSEM Environnement, 1225 Rte de l'Enfant, 13290 Aix-en-Provence <u>contact@xsem.fr</u>

Key-words: PFAS, transfer pathways, modelling, saturated zone, unsaturated zone, feedback, remediation

**Objectives:** To provide feedback on the strategy used to identify the risk of PFAS pollution transfer to both unsaturated and saturated zones, and on the choice of remediation methods. Highlight the methodological approach based on numerical modeling of the transfers in the unsaturated zone and then in the saturated zone.

**Innovative aspect of the project:** This work is based on a real remediation case of a pilot site contaminated using fire-fighting foams, in an uncertain regulatory context (absence of thresholds for soils). The strategy adopted is based on modeling the risk of off-site transfer and assessing sensitive targets to define operational treatment thresholds.

#### Summary:

PFAS are ubiquitous industrial pollutants, presenting serious health and environmental risks. Their persistence and capacity to disseminate in groundwater and surface water make them a major concern. We present a case study based on the remediation of a PFAS-contaminated site using fire-fighting foam. The focus will be on characterizing and modeling the transfer pathways of PFAS from the surface to the soil and groundwater and determining the appropriate remediation strategy. Indeed, in the absence of soil regulations and thresholds, an assessment of the risk of soil contamination transfer must be carried out to identify the consequences of a transfer and adapt the proposed remediation methods.

Soil borings were used to characterize migration within heterogeneous media (sandy and clayey) and to estimate a significant stock of PFAS contaminant in the unsaturated zone. At the same time, groundwater monitoring showed that the groundwater was already contaminated, despite the presence of a significant fraction of the stock in the soils. Numerical modelling was realized to evaluate the fate of this PFAS stock, in particular the mobility of short-chain PFAS and their potential to migrate off-site, where sensitive targets were identified. The results indicate a risk of dissemination of a dissolved pollution plume. Given the mobile nature of the pollutants identified, and the fact that a part of the contamination has already reached the groundwater, it is important to assess the treatment options available. The question then emerged to concentrate treatment solely on the groundwater or also intervene in the unsaturated zone. However, simply treating groundwater could involve a long pumping and treatment period and an increased risk of off-site dispersion, underlining the importance of integrating the soils remediation into the overall strategy.

This feedback demonstrates the value of a methodological approach based on a combination of field data acquisition and numerical modelling. As part of the management of sites contaminated by PFAS, this method makes it possible to identify suitable treatment methods and strategies, discuss critical parameters such as transfer time and risk, and choose thresholds values.



# Panoplie des technologies des traitements de dépollution des sols, eaux et de l'air existants concernant les PFAS

# **Overview of existing PFAS treatment technologies concerning Soil, Water and Air**

### Frank KARG / HPC INTERNATIONAL

CEO / HPC INTERNATIONAL SAS, Dr. Alfred-Herrhausen-Allee 12, 47228 Duisburg – Germany and HPC International SAS, H. de Recherche / Centre Médical de Perharidy, 29680 Roscoff, France Email: <u>frank.karg@hpc-international.com</u> / <u>Phone</u>: +33 607 346 916

# Abstract

Several PFAS Treatment technologies are available today, as for ex. "old fashion" and not really sustainable technologies, like Excavation and thermal treatments or P&T: Pump and Treat for Groundwater

Nowadays, more sustainable technologies for PFAS contaminated Site Remediation are available, as for ex. in-situ or on-site Washing with Biopolymer Proteins, which are used in an Washing circle with regeneration of the washing ingredients.

The following Table shows a general resume of those technologies, with their advantages and inconvenients:

| PFAS: Dépollutions des sol, des sediments et des eaux (souterraines, lixiviats, etc.) Frank KARG |                                |   |                 |                    |                        |  |
|--|--------------------------------|---|-----------------|--------------------|------------------------|--|
| Matrice  | Technologie                    |   | Advantage       | Inconvénients      | Remarques              |  |
| 1. Soil  | On-site & off-site:            | ~ | Rapid Site      | Very Expensive.    | Lots of Landfills      |  |
|  | Excavation & Confinement       | 1 1 2                                   | Remediation.    | Needs excava-      | don't accept           |  |
|  | / Landfill (Dig & Dump)        | C.D.Y.                                  |                 | tion. No sustain-  | PFAS contamin-         |  |
|  |                                | AT AT AS                                |                 | able solution.     | ated soils (>50 mg/kg) |  |
| 2. Soil  | On-site:                       |   | Rapid cut-off   | Site use restric-  | Lots of                |  |
|  | Capping / Surface Sealing      |   | concerning      | tions /Servitudes  | Authorities            |  |
|  | to avoid Leaching to Ground-   |   | Exposure Path-  | No sustainable     | don't accept           |  |
|  | water                          |   | ways & Leachate | solution.          | this solution.         |  |
|  |                                |   | to Groundwater  |                    |                        |  |
| 3. Soil  | In-situ:                       | Caller State THE                        | In-situ-Site    | Relatively         | Feasibility            |  |
|  | Excavation & Treatment:        |   | Remediation     | Expensive by       | Study is need-         |  |
|  | Physical, Chemical, Thermic-   | The Part of                             |                 | Electricity Costs. | ed concerning          |  |
|  | ally (Washing, ISCO, Thermal   |   |                 | Gas treatment is   | sub-soil condi-        |  |
|  | Destruction, etc.)             | A Walt                                  |                 | needed.            | tions.                 |  |
| 4. Soil  | On-site, off-site or in-situ   | (here)                                  | Avoid leachate  | Very Expensive.    | Feasibility            |  |
|  | Stabilization with cationic    |   | to Groundwater. | Needs              | Study is need-         |  |
|  | clays, Zeolites, etc. to avoid |   |                 | potentially        | ed concerning          |  |
|  | Leaching to Groundwater        | Boil TT Augers                          |                 | excavation.        | Stabilization          |  |
|  |                                | Binder Noction<br>Nozzles               |                 |                    | ingredients.           |  |

1



| PFAS: Dépollutions des sol, des sediments et des eaux (souterraines, lixiviats, etc.) Frank KARG |   |   |  |  |   |
|--|---|---|--|--|---|
| Matrice  | Technologie   |   | Advantage  | Inconvénients  | Remarques   |
| 5. Soil  | On-site & off-Site:<br><u>Thermal Desorption</u> up to<br>1 200 °C:<br>JACOBS – William DiGuiseppi  |   | Relatively rapid<br>Site<br>Remediation.                                       | Expensive.<br>Needs excava-<br>tion. No sustain-<br>able solution  | Lots of<br>Authorities<br>don't accept<br>PFAS soils.                       |
| 6. Soil  | et al.<br>Off-site:<br><a> <u>Incineration</u> up to<br/>1 200 °C</a>   |   | Relatively rapid<br>Site<br>Remediation.                                       | (off-gas, CO <sub>2</sub> ).<br>Very Expensive.<br>Needs excavation.<br>No sustain-able<br>solution (off-gas,<br>PFDD-F Emissions) | Lots of Inciner-<br>ators don't<br>accept PFAS<br>contaminated<br>soils.    |
| 7. Soil  | In-Situ:<br><u>Immobilization</u> with colloidal<br>activated carbon (CAC):<br>Plume Stop/ Regenesis or MattCARE                          | Liquid colloidal carbon<br>soil treatment (18 PFASs)  | In-situ-Site<br>Remediation by<br>injection of CAC.                            | A feasibility stu-<br>dy must show<br>the needed ad-<br>sorption capacity<br>& aquifer needs                                       | Feasibility<br>Study is need-<br>ed concerning<br>sub-soil condi-<br>tions. |
| 8. Soil &<br>Aquifer   | In-situ & ex-situ:<br>➤ In-situ soil Washing with<br>proteinic Bio-polymers,<br>beta-Cyclodextrines, etc.:<br>Sensatec, HPC-INTERNATIONAL | A factor in the factor in | Avoid excava-<br>tion. Appliable<br>for saturated &<br>non-saturated<br>Zones. | Needs technical-<br>economic<br>Feasibility Study.   | Washing<br>detergents or<br>Agents must be<br>tested.                       |

| PFAS: Soil -Water and Aquifer Remediation Technologies (Wastewater, Leachate, Groundwater, etc.) Frank KARG |   |  |  |  |  |
|---|---|--|--|--|--|
| Matrice   | Technologie   |  | Advantage  | Inconvénients  | Remarques  |
| 9. Soil   | In-Situ & ex-situ:<br><u>Immobilization</u> with Clays  | Natural Modified<br>Zeolites   | In-situ-Site<br>Remediation by   | A feasibility stu-<br>dy must show   | Feasibility<br>Study is need-  |
|   | and Zeolites:<br>CETCO & ZEOCEL and by DND<br>Biotech –Italy  |  | injection of<br>Clays or Zeolites  | the needed ad-<br>sorption capacity<br>& aquifer needs   | ed concerning<br>sub-soil condi-<br>tions.   |
| 10. Water   | Ex-situ:<br>Extraction by biodegradable<br>Floculant & Precipitation:<br>PerfluorAd /<br>Cornelsen-TRS,   | PerfluorAd*  | Efficient Water<br>treatment,<br>useable also for<br>soil & sediment<br>washing.                   | Needs P&T &<br>technical-econo-<br>mic Feasibility<br>Study (pH, salinity,<br>co-pollutants + GAC<br>Polishing ?). | Different<br>Agents must be<br>tested.   |
| 11. Water   | <ul> <li>Ex-situ:</li> <li>Modified Clay / Adsorbens:<br/>CETCO &amp; ETEC2: Fluorosorb</li> </ul>  | FLUORO-SORB'<br>adsorbent<br>SS<br>PFAS Treatment Solution Proven To Be Up<br>To 0-0 HIMES MORE EFFECTIVE Than GAC | Efficient Water<br>treatment.  | Needs P&T &<br>technical-econo-<br>mic Feasibility<br>Study (pH, salinity,<br>co-pollutants).                      | Different<br>Agents must be<br>tested.   |
| 12. Water   | Ex-situ:<br>TP 108 DW (+for short PFAS) & Lewatit <sup>®</sup> K 6362 (Wastewater)<br>& Lewatit <sup>®</sup> M 62 WS/LAWESS, Sorbix A3F resin & Sorbix<br>LC4 & Sorbix RePure LC (FTEC, Purofine <sup>®</sup> PFA-694 & Puro-<br>fine <sup>®</sup> - PFA694E/ Pyrolite & ECOLAB, Amberite PSR 2Plus /<br>DuPont, Calles 2302(Calgon Carbon Corp., SIR-110-HP/<br>Resin Tech Inc., Fluoro-Sorb <sup>®</sup> / CETCO, RemBind <sup>™</sup> / ZILTEC |  | Efficient Water<br>treatment if pH<br>is not alkaline.<br>Depends on salini-<br>ty, co-pollutants. | Needs P&T &<br>technical-econo-<br>mic Feasibility<br>Study: pH, salinity,<br>co-pollutants, GAC<br>needs, ?       | Different Agents<br>and IX (mixtures)<br>must be tested.<br>A: Single used IX:<br>$\rightarrow$ Incineration need<br>B: Regenerable IX<br>$\rightarrow$ With alkaline pH |



| PFAS: Dépollutions des eaux (souterraines, lixiviats, etc.) |                               |   |                  |                    |                               |  |
|---|-------------------------------|---|------------------|--------------------|-------------------------------|--|
| Matrice   | Technologie                   |   | Advantage        | Inconvénients      | Remarques                     |  |
| 13. Water   | Ex-situ:                      | SAFF <sup>®</sup>   | Efficient Water  | Needs technical-   | Different                     |  |
|   | Foam Fractioning / Air-Water  |   | treatment,       | economic           | Agents must be                |  |
|   | Interface Foam / Concen-      | ····· ····  | useable also for | Feasibility Study. | tested.                       |  |
|   | tration in Bubble Interfaces: |   | soil & sediment  | Principally for    |                               |  |
|   | ALLONIA, SAFF®, etc.          | Sum ye  | washing.         | PFAS > C6.         |                               |  |
| 14. Water   | In-situ:                      |   | Potentially      | Needs P&T &        | Only one                      |  |
|   | Downhole Foam Fractioning:    |   | Efficient Water  | technical-econo-   | provider in                   |  |
|   | OPEC Systems / Australia      |   | treatment.       | mic Feasibility    | 2024                          |  |
|   |                               | Alb. 18. Prinzip des In. site Schwarzhakterijerung Verfahrens. 1. Komprinsiels Luft wird an der   |                  | Study.             |                               |  |
|   |                               | bisses are solven the program of the solution of the soluti |                  |                    |                               |  |
| 15. Water   | Ex-situ:                      | 3 <sup>2</sup> 8 0.   | Mineralization   | Experimental       | Creation of hydraed           |  |
|   | Photolysis by UV/             |   | via hydrated OH- | Status in 2024.    | duction by adding a           |  |
|   | $\frac{H_2O_2}{Advanced}$     |   | Radicals.        | Needs technical-   | reducing agent (iod-          |  |
|   | Reduction Process             | New Storter<br>their optic  |                  | eco-nomic          | ides, thiosulfate,            |  |
|   | (ARP): Tree Water             | UV/Nitrilotriacetic acid Javed et al., 2020   |                  | Feasiblity Study.  | sulfite, etc.).               |  |
| 16. Water   | Ex-situ:                      | Concentrate   | Efficient Water  | Needs P&T &        | 10mS·cm <sup>-1</sup> -       |  |
|   | <u>Reverse Osmosis (RO):</u>  | Perforated Product Take   | treatment in     | technical-econo-   | total organic carbon          |  |
|   | (Wastewater):                 | Parel Wyter<br>Carity   | dependance of    | mic Feasibility    | concentration of              |  |
|   | SIMPEC (+SARPI-VEOLIA)        | Fend<br>Selection   | organic load.    | Study (organic     | 1100 mg·L <sup>-1</sup> could |  |
|   |                               | Permeate Carrier<br>Material  |                  | Imoad etc.)        | be treated by RO              |  |

| PEAS: Dépallutions des eaux (souterraines, liviviate, etc.) |                                 |   |                                   |                           |   |  |
|---|---------------------------------|---|-----------------------------------|---------------------------|---|--|
| PFAS: Depollutions des eaux (souterrain                     |                                 |   | es, intiviats, etc.)              |                           | Frank KARG  |  |
| watrice   | lechnologie                     |   | Aavantage                         | Inconvenients             | кетarques   |  |
| 17. Water   | Ex-situ (+ in-situ injections): | A A   | Efficient Water                   | Needs technical-          | Different   |  |
|   | Activated Carbon (GAC),         |   | treatment de-                     | economic Feasi-           | combinations  |  |
|   | Powdered Activated Carbon       |   | pending of pollu-                 | bility Study: pH,         | (+ Adsorption   |  |
|   | (PAC) & Biochars: Purolite,     |   | tant Cocktail <mark>(ultra</mark> | salinity, co-             | Resins ?) must  |  |
|   | etc. (+Sensatec)                |   | short PFAS ?)                     | needs?                    | be tested.  |  |
| 18. Water   | Ex-situ:                        |   | Efficient Water                   | Needs technical-          | Advanced  |  |
|   | Low Temperature Mineraliza-     | Deno  | treatment in Di-                  | economic Feasibil-        | Research Status:  |  |
|   | tion – Decarboxylation &        | F T T T T T T T T T T T T T T T T T T T   | methyle-sulfoxide,                | ity Study. Commer-        | Brittany TRANG  |  |
|   | Defluorination (80° - 120°C -   | 2 Bit Off,CO <sub>2</sub> 107 a Bi gyrunne mark.<br>Authentic standard degrades<br>Fu.s. Overview of degradation pathways identified in this study.   | useable for soil &                | cial Availability not     | et al. 2022   |  |
|   | Heating with NaOH & DMSO Solv.  | Healing PFCAs in point particle solveres such as DMBD decatebooities item to trity-performationes.<br>Then this instruction was performed in the presence of nuscles (in the PFCA memory output) is<br>fluoreacted, and nonhommated carbon-containing products. The trity-performaniation is underward the same<br>approximate memory of nuscle bases teamory output.   | sediment washing.                 | granted.                  | (https://www.science.org/doi/10.1<br>126/science.abm8868 ). |  |
| 19. Water   | Ex-situ:                        | Radicals<br>NTD Reartise species  | Efficient Water                   | Needs technical-          | Advanced  |  |
|   | Non-Thermal Plasma (NTP)        | ELECTRICITY   | treatment under                   | economic Feasibi-         | Research Status   |  |
|   | Destruction:                    | ↑ Photons v <sup>2</sup> d <sup>ar</sup>  | conditions.                       | lity Study (Co-poll-      | with pilots   |  |
|   | Tectero BV / Belgium            |   |                                   | uants, alkanity?).        | (2024).   |  |
|   | LSPM / Sorbonne                 |   |                                   | Electricity Costs ?       | . ,   |  |
| 20. Water   | Ex-situ:                        | Bow out   | Efficient PFAS                    | Needs technical-          | Usable for  |  |
|   | Photo-electrocatalytic          | AC sayshy DC for this TO, coldy/st v  | Destruction &                     | economic Feasi-           | contaminated  |  |
|   | Advanced Oxidation (PEC)        |   | Mineralization                    | bility Study: pH,         | Groundwater   |  |
|   | Processes: Capture – Italia     | Reactor, ASI 3161,<br>outerhis 55<br>30 With water D/ Jame  | Process (> 99 %).                 | salinity, co-pollu-       | and for WWTP  |  |
|   |                                 | Elsevicit     Purop     Purop | ( , -                             | tants. Pilot exist(2024). | (Wastewater Treatment Pants).                               |  |

For exemple the Fig. 1-5 shows the actual most Cost effective on-site and in-situ Treatment technologies for soil in the non-saturated and the saturated zones.





Fig. 1 : In-situ Washing of PFAS with proteinic Bio-polymers



Fig. 2 : Practical application of in-situ Washing of PFAS with proteinic Bio-polymers





Fig. 3 : Prior sub-soil permeabilization with TSE Injections for practical application of in-situ Washing of PFAS with proteinic Bio-polymers



Fig. 4: In-situ Immobilization of PFAS via colloidal Activated Carbon.



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## Exposure to PFAS: One Health approach in the anthropised area of Fos-Berre Study of contamination and toxicity of PFAS in drinking water and analytical developments for lichen biomonitoring of atmospheric contamination

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#### I. MATISSE project

The MATISSE (Multi-scale PFAS conTamInation Study in induStrial arEas) project, financed by the A\*midex Foundation (Aix-Marseille University) and ABO Group, was designed to study the contamination levels and impacts of PFAS on human and environmental health in the highly anthropised area of Fos-Berre (France). This region hosts many petrochemical and chemical industries, as well as Classified Installations for Environmental Protection (PCI-French Environment Code) that will be soon required to analyse PFAS in their effluents. In order to provide valuable and complementary information, the presence of PFAS will be investigated in different environmental compartments of this area (drinking and groundwater, soils, lichens) through a one-health approach, to determine contamination levels and pathways (multi-matrix transfers), in parallel with ecotoxicological and toxicological impacts of PFAS cocktails at environmental concentrations.

In order to carry out this project on a regional scale, MATISSE is led by a multidisciplinary consortium gathering the Environmental Chemistry Laboratory (LCE-Aix Marseille University), the Mediterranean Institute of Biodiversity and marine and continental Ecology (IMBE-Aix Marseille University), the Ecocitizen Institute for the Knowledge of Pollutions (IECP), and the ABO-ERG ENVIRONNEMENT company.

#### II. Lichens analytical development

The evaluation of atmospheric contamination can be performed by using passive biosamplers such as lichens. Thanks to their lack of a root system, their stationary location and their ubiquitous presence in almost all environments and supports, they are representative and integrative of the atmospheric contamination levels. An extraction method for 49 PFAS in lichens was developed based on the literature. A solid/liquid extraction method was developed on spiked freeze-dried and milled lichen (0.2 g), with 3 consecutive extractions. Extraction, purification and concentration methods were chosen according to the best extraction recovery and the lowest remaining matrix effects. Contamination from the procedure steps was also evaluated. The best extraction method involved 3 consecutive extractions with 25.7 mM NH<sub>4</sub>OH in methanol via ultrasonication (20 kHz, 15 min) followed by purification on Envi-Carb<sup>®</sup> cartridges. This method yielded extraction recoveries of around 70 to 120%. Reducing the concentration of purified extracts diminished the initial strong matrix effect. Samples were centrifuged to avoid the loss of PFAS through filtration and were analysed by LC-MS/MS coupled to a Sciex QqTrap 7500. Analytes were separated on a C18 column eluted by a methanol/water mixture and analysed through negative electrospray ionisation. In the final method, isotopically labelled surrogates were added to follow the extraction process and internal standards allowed the correction of matrix effects and the quantification of targeted compounds.

#### III. Drinking water monitoring

An analytical method has been developed to investigate the PFAS content (ISO 21675, PCI regulation, DWD 2020/2184) in 28 tap waters and 2 groundwaters collected from the Crau Water Table and the Durance River in the highly industrialised area of Fos-Berre. Water samples were collected in prewashed high-density polyethylene (HDPE) bottles, as recommended by the ISO 21675 regulation, and field blanks were filled on site with ultrapure water and exposed to the sampling environment. Ammonium acetate was added at 1 g.L<sup>-1</sup> as a preservation agent. Samples were transported at around 4 °C and stored frozen (-20 °C) until analysis by LC-MS/MS occurring within 2 weeks after collection. Solvent blanks were run before and regularly within each batch to investigate the instrumental background as well as potential carry-over contamination. The precision and accuracy of the method were confirmed for each run and monitored by the regular analysis of calibration points. Method accuracy has been verified by analysing a Certified Reference Material (IRMM-428 Water) and by having duplicated samples analysed by a certified laboratory. PFAS were detected and quantified in 29 of the 30 samples. Among the 35 PFAS investigated, 17 were quantified in at least 1 sample. PFHxA was the most quantified in the samples, followed by linear PFOS, PFOA and branched PFOS. Despite preventive measures, some contamination was found in the field blanks and was handled by result post-treatment. The sum of total PFAS was below the 2020/2184 DWD limit (500 ng.L<sup>-1</sup>) for all samples, and the sum of the 20 regulated PFAS was above the 100 ng.L<sup>-1</sup> limit for only 1 sample, with a concentration of 119 ng.L<sup>-1</sup>. The differences in contamination levels and profiles over the 28 sites indicated an influence of the water catchment location, its surrounding (e.g. industries) and the groundwater flow, with more contamination downstream of the water table.

#### IV. Eco- and toxicological Risk Assessment

Studied PFAS were chosen amongst the 17 according to their occurrence, amount and chemical structure. To conduct preliminary assays regarding potential PFAS effects on the human digestive tract, Caco2 cells (cells from human colorectal adenocarcinoma harboring enterocyte phenotype), were exposed for 24 hours to PFOS, PFHxA and PFPeA, both individually and in a ternary mixture. Chosen concentrations were based on those of European populations' serum. The relative expression of several target genes was determined by RT-qPCR. The studied genes were for inflammation: IL1 $\beta$ , TNF $\alpha$ , IL6, TGF $\beta$ ; for oxidative stress: SOD2, CAT, GPX1; for detoxication: CYP1A, AhR; for cellular junction characterization: CLDN1 and CLDN3. Preliminary results will be presented. The project also aims to evaluate the effects of PFAS in aquatic environments by exposing an aquatic invertebrate model (*Hydra sp.*) to mixtures of 9 PFAS. A multiscale analysis ranging from a molecular to a population level will be used to assess their biological activity.

#### Acknowledgements

This work was financially supported by the A\*midex Foundation and by the ERG foundation. We are grateful to all the volunteers of the VOCE (Volunteers for the Citizen Observation of the Environment) network implied in the drinking water field campaign.

## Release of Per- and Polyfluoroalkyl Substances (PFAS) from Soil to Groundwater: Importance of PFAS Family and Substance

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Keywords: Contaminated sites and soils, groundwater, persistent organic pollutants, PFAS, release, transfer

**Innovative aspect of the work:** In-depth analysis of the release of 30 PFAS, including fluorotelomers, from polluted soil highlighting the influence of multiple parameters.

**Objective:** understanding the release mechanisms of PFAS based on the different PFAS families, soil composition, cation type, and variations in ionic strength.

#### Abstract:

Per- and polyfluoroalkyl substances (PFAS) are a large group of synthetic chemical compounds characterized by the partial or complete substitution of hydrogen atoms in the carbon backbone with fluorine atoms. With over 12,000 known substances, PFAS are widely used in everyday products such as cookware, water-repellent clothing, food packaging, and firefighting foams due to their anti-adhesive, hydrophobic, and heat-resistant properties [1]. However, their persistence, mobility in the environment, and toxicity (linked to liver disorders, endocrine disruption, developmental abnormalities, and an increased risk of cancer [2]) make them a major concern. PFAS are detected in soils, water, food, and even human blood. In 2024, an interministerial action plan was implemented to enhance the understanding and management of these pollutants across various environmental matrices, including soil [3].

Understanding the behavior of PFAS in soils, particularly their release and transfer mechanisms, is crucial for assessing human exposure and developing effective management and remediation strategies for contaminated sites. These compounds feature a hydrophobic carbon chain and a hydrophilic functional group, which influence their mobility through hydrophobic and electrostatic interactions [4], [5]. Their retention in soils depends on multiple factors, including organic carbon content (TOC), clay composition, pH, and the ionic strength of groundwater. However, the mechanisms governing their retention, as well as the relative influence of different soil components, remain poorly understood. Further research is needed to better anticipate their environmental and health impacts and to optimize their management [6].

This study evaluated the release of PFAS from soil sampled at a former firefighter training site where firefighting foams had been used. The soil, predominantly sandy, exhibited a slightly basic pH (8.4), a low total organic carbon (TOC) content (10 g/kg), and a total PFAS concentration of 390  $\mu$ g/kg (based on the analysis of 58 PFAS). The results revealed a predominance of 8:2 FTS (32%), 6:2 FTS (29%), and PFOS (25%), consistent with the historical use of firefighting foams. The predominant detection of fluorotelomers also highlights the need to expand the list of 20 PFAS regulated under the European Drinking Water Directive to account for the major sources of PFAS contamination at such sites and to improve human health risk assessment.

Batch experiments were conducted at the laboratory scale to assess PFAS release kinetics under different experimental conditions, including variations in ionic strength (by adjusting salt concentrations in solution), the presence of two different cations ( $Ca^{2+}$  or  $Mg^{2+}$ ), and the absence or presence of sodium azide ( $NaN_3$ ) as a bacterial inhibitor. Release kinetics were monitored by analyzing samples collected at different agitation times (1, 6, 16, 24 hours, 3, 5, and 7 days). A total of 30 PFAS were analyzed using high-performance liquid chromatography coupled with tandem mass spectrometry.

The PFAS quantified in the solutions after agitation included PFCA (perfluorinated carboxylic acids with carbon chains ranging from C4 to C11), PFSA (perfluorinated sulfonic acids with carbon chains of C6, C8, and C9), fluorotelomers (6:2, 8:2, 10:2 FTS), and FOSA. The analysis revealed contrasting trends in release percentages depending on carbon chain length and experimental conditions.

For short-chain PFCA (particularly PFPeA, PFHxA, and PFHpA), an increase in release rates over time was observed under all tested conditions. Additionally, within the short-chain PFCA group, a longer carbon chain corresponded to higher release rates.

In the absence of a bacterial inhibitor, release rates were significantly higher than with presence for PFPeA and PFHxA after 7 days of agitation and for PFHpA after 5 days, without CaCl<sub>2</sub>. When CaCl<sub>2</sub> was added, release rates for these three compounds were also higher than without the bacterial inhibitor than with it after 5 days of agitation. Under these conditions, the release percentages of PFPeA, PFHxA, and PFHpA exceeded 100%. These unexpected high values could be attributed to the biotransformation of precursors, such as 6:2 FTS, into short-chain PFCA.

Long-chain PFCA (C $\geq$ 9) exhibited the opposite trend: their release rates decreased over time and with increasing fluorinated carbon chain length, regardless of experimental conditions. This behavior can be attributed to their higher hydrophobicity, which enhances their affinity for the solid matrix, thereby reducing their mobility. In the absence of a bacterial inhibitor, PFDA showed a decline in release rates over time. This decrease could suggest the transformation of long-chain PFCA into short-chain PFCA, potentially explaining the apparent increase in the release percentages of the latter in the absence of the bacterial inhibitor.

PFSA followed a similar trend to long-chain PFCA, with decreasing release percentages as carbon chain length increased under conditions without the bacterial inhibitor.

Additionally, increasing the ionic strength of the solution reduced the release rates of long-chain PFAA (perfluoroalkyl acids). This is likely due to competition in solution between dissolved ions and the more hydrophobic long-chain PFAA, which mainly favors adsorption onto the solid matrix. However, no significant difference was observed for short-chain PFAA when ionic strength was increased.



Figure 1 illustrates these results, showing the release kinetics of PFBA, PFHxA, and PFDA in the absence of CaCl<sub>2</sub> and NaN<sub>3</sub>.

Figure 1: Release kinetics of PFBA (C4), PFHpA (C7), PFDA (C10), and PFUnDA (C11) in the absence of CaCl<sub>2</sub> and NaN<sub>3</sub>

Fluorotelomers (6:2, 8:2, and 10:2 FTS) exhibited significantly lower release rates compared to PFCA and PFSA, despite their higher initial concentrations in the soil. For instance, the release of 6:2 FTS was approximately half of the release of PFAA, while 8:2 FTS had a release rate four times lower. Unlike PFAA, the release rates of fluorotelomers remained constant over time.

Although a decrease in fluorotelomer concentrations might be expected to explain the increasing release percentages of short-chain PFCA in the absence of a bacterial inhibitor, this trend was not observed. However, the degradation of fluorotelomers into short-chain PFCA could be counterbalanced by their release from the solid phase when the liquid phase becomes depleted, allowing equilibrium to be restored and masking any apparent variation in solution concentration due to their excess presence in the soil compared to other PFAS. This process may mask any apparent variation in fluorotelomer concentrations in solution, given their excess presence in the soil compared to other PFAS. To further investigate these mechanisms, additional degradation experiments are in progress to better understand fluorotelomer interactions and transformations in soil.

In conclusion, these novel findings, focusing on PFAS compounds whose behavior has been little or not studied in the literature (particularly FTS and PFAA beyond PFOS, PFOA, and PFHxA) highlight that both the PFAS family and carbon chain length are key factors in predicting their fate in soils. Moreover, considering their predominance in soils at sites where firefighting foams have been used, as well as their potential release and transfer from soil to groundwater, fluorotelomers should be monitored in both compartments. These results are also being further analyzed to strengthen the understanding of the physico-chemical mechanisms governing PFAS behavior in soils, including studies on different soil types. The upcoming interpretations of these findings will also be presented.

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## Relargage des substances per et polyfluoroalkylées (PFAS) des sols vers les eaux souterraines : importance de la famille de PFAS et de la substance

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Mots clés : Sites et sols pollués, eaux souterraines polluants organiques persistants, PFAS, relargage, transfert

**Caractère innovant du travail :** analyse approfondie du relargage de 30 PFAS, y compris les fluorotélomères, à partir de sols pollués, mettant en évidence l'influence de plusieurs paramètres.

**Objectif :** comprendre les mécanismes de relargage des PFAS en fonction des différentes familles de PFAS, de la composition du sol, du type de cations et de l'évolution de la force ionique.

#### Résumé :

Les substances per et polyfluoroalkylées (PFAS) constituent une classe de composés chimiques synthétiques caractérisés par la substitution partielle ou totale des atomes d'hydrogène du squelette carboné par des atomes de fluor. Comptant plus de 12 000 substances, elles sont largement employées dans des produits de la vie quotidienne tels que les ustensiles de cuisine, les vêtements déperlants, les emballages alimentaires et les mousses anti-incendie, en raison de leurs propriétés physico-chimiques antiadhésives, imperméables et de leur résistance à a chaleur [1]. Cependant, leur persistance et leur mobilité dans l'environnement ainsi que leur toxicité, (en particulier troubles hépatiques, perturbations endocriniennes, anomalies du développement et risque accru de cancers [2]) en font des substances préoccupantes. Les PFAS sont détectés mondialement dans l'eau, les sols, les aliments et même dans le sang humain, ce qui a conduit à une intensification des réglementations. En 2024, un plan d'action interministériel a été instauré pour améliorer la compréhension et la gestion de ces polluants dans différentes matrices y compris le sol [3].

La compréhension du comportement des PFAS dans les sols, notamment leurs mécanismes de relargage et de transfert, est essentielle pour évaluer l'exposition humaine et développer des stratégies de gestion et de remédiation efficaces des sites pollués. Ces composés possèdent une chaîne carbonée hydrophobe et un groupe fonctionnel hydrophile, influençant leur mobilité par des interactions hydrophobes et électrostatiques [4, 5]. Leur rétention dans les sols dépend de multiples facteurs, tels que la teneur en carbone organique (COT), la composition en argile, le pH et la force ionique des eaux souterraines. Toutefois, les mécanismes ainsi que la hiérarchisation des composants du sol régissant leur rétention restent mal compris, nécessitant des recherches approfondies pour mieux anticiper leur impact environnemental et sanitaire et optimiser leur gestion [6].

Cette étude a évalué le relargage des PFAS à partir d'un sol prélevé sur un ancien site d'entraînement au feu des pompiers, sur lequel des mousses anti-incendie ont été utilisées. Ce sol, majoritairement sableux, présentait un pH légèrement basique (8,4), une faible teneur en carbone organique total (10 g/kg) et une concentration totale en PFAS de 390 µg/kg (analyse d'une liste de 58 PFAS). L'analyse a mis en évidence une prédominance du 8:2 FTS (32 %), du 6:2 FTS (29 %) et du PFOS (25 %), ce qui est conforme avec une historique d'utilisation de mousses anti-incendie. La détection majoritaire de fluorotélomères souligne également la nécessité d'élargir la liste des 20 PFAS de la directive européenne relative à la qualité des eaux destinées à la consommation humaine, afin de prendre en compte la part majoritaire de la pollution en PFAS sur de tels sites et d'améliorer l'évaluation du risque sanitaire.

Sur ce sol, des cinétiques de relargage ont été menées en batch, au laboratoire, sous différentes conditions expérimentales comprenant : des variations de la force ionique (par une variation des concentrations en sel en solution), la présence de deux types de cations différents en solution (Ca<sup>2+</sup> ou Mg<sup>2+</sup>) et l'absence ou la présence d'azoture de sodium (NaN<sub>3</sub>) utilisé comme inhibiteur bactérien. La cinétique de relargage a été suivie par l'analyse d'échantillons prélevés à différents temps d'agitation (1, 6, 16 et 24 heures, puis 3, 5 et 7 jours). Un total de 30 PFAS a été analysé par chromatographie liquide à haute performance couplée à la spectrométrie de masse en tandem.

Les PFAS quantifiés dans les solutions après agitation incluaient les PFCA (acides carboxyliques perfluorés avec des chaînes carbonées allant de C4 à C11), les PFSA (acides sulfoniques perfluorés possédant des chaines carbonées de C6, C8, et C9), les fluorotélomères (6:2, 8:2, 10:2 FTS) et le FOSA. L'analyse révèle des tendances contrastées dans les pourcentages de relargage en fonction de la longueur de la chaîne carbonée et des conditions expérimentales.

En effet, pour les PFCA à chaîne courte (en particulier PFPeA, PFHxA et PFHpA) une augmentation des taux de relargage au fil du temps est observée dans toutes les conditions étudiées. De plus, parmi les PFCA à chaîne courte, une augmentation de la longueur de la chaîne carbonée mène à une augmentation des taux de relargage.

Sans inhibiteur bactérien, les taux de relargage sont significativement plus élevés qu'en présence d'inhibiteur bactérien pour le PFPeA et le PFHxA après un temps d'agitation de 7 jours et pour le PFHpA après un temps d'agitation de 5 jours, en l'absence de CaCl<sub>2</sub>. Sans inhibiteur bactérien, l'ajout de CaCl<sub>2</sub> mène à des taux de relargage plus élevés qu'en présence d'inhibiteur bactérien pour ces 3 molécules après 5 jours d'agitation. Dans ces conditions, les pourcentages de relargage des PFPeA, PFHxA et PFHpA dépassent les 100 %. Ces valeurs très élevées pourraient être attribuées à la biotransformation de précurseurs tels que le 6:2 FTS en PFCA à chaîne courte.

Les PFCA à chaîne longue (C≥9) montrent une tendance inverse : leurs taux de relargage diminuent avec le temps et à mesure que le nombre de carbones fluorés augmente, indépendamment des conditions expérimentales. Cette observation peut s'expliquer par leur hydrophobicité accrue, entraînant une affinité plus grande pour la matrice solide et réduisant ainsi leur mobilité. Sans inhibiteur bactérien, le PFDA présente une diminution du taux de relargage au fil du temps. Cette baisse pourrait indiquer une transformation des PFCA à chaîne longue en PFCA à chaîne courte, ce qui pourrait expliquer l'augmentation apparente des pourcentages de relargage de ces derniers en absence d'inhibiteur bactérien.

Les PFSA suivent une tendance similaire à celle des PFCA à chaîne longue, avec des pourcentages de relargage décroissants lorsque la longueur de la chaîne carbonée augmente dans les conditions où l'inhibiteur bactérien est absent.

Par ailleurs, l'augmentation de la force ionique de la solution réduit le taux de relargage des PFAA (acides perfluoroalkylés) à longue chaîne. Ceci est probablement dû à une compétition en solution entre les ions et les PFAA à longue chaîne, plus hydrophobes par nature et donc plus propices à être fixés sur la matrice solide. Aucune différence significative pour les PFAA à chaîne courte n'est observée lors de l'augmentation de la force ionique.





Figure 1:Cinétiques de relargage du PFBA (C4), du PFHpA (C7), du PFDA (C10) et du PFUnDA (C11) en l'absence de CaCl<sub>2</sub> et de NaN<sub>3</sub>

de CaCl<sub>2</sub> et de NaN<sub>3</sub>.

Les fluorotélomères (6:2, 8:2 et 10:2 FTS) présentent des taux de relargage significativement inférieurs à ceux des PFCA et PFSA, bien que leurs concentrations initiales dans le sol soient plus élevées. Par exemple, le relargage du 6:2 FTS est environ deux fois inférieur à celui des PFAA, tandis que celui du 8:2 FTS est quatre fois inférieur. Contrairement aux PFAA, les taux de relargage des fluorotélomères restent constants au fil du temps. Bien que l'on s'attende à une diminution des concentrations des fluorotélomères pour expliquer les pourcentages de relargage des PFCA à chaîne courte qui augmentent en l'absence d'inhibiteur bactérien, cette tendance n'est pas visible.

Cependant, la dégradation des fluorotélomères en PFCA à chaîne courte pourrait être compensée par un relargage à partir de la phase solide lorsque la phase liquide s'appauvrit en ces composés afin d'atteindre à nouveau l'équilibre, masquant ainsi toute variation apparente de concentration en solution, au regard de leur présence en excès dans le sol par rapport aux autres PFAS. Afin d'approfondir l'analyse de ces mécanismes, des expériences supplémentaires de dégradation ont été mises en œuvre. Ces travaux visent à mieux comprendre les interactions et les transformations des fluorotélomères dans le sol.

En conclusion, ces résultats originaux s'intéressant à des PFAS dont le comportement n'est peu voire pas étudié dans la littérature (notamment les FTS et les PFAA autres que le PFOS, le PFOA et le PFHxA) montrent que la famille de PFAS et la longueur de la chaîne carbonée sont des éléments majeurs à prendre en compte lors de la prédiction de leur devenir dans les sols. Par ailleurs, au regard de leur présence majoritaire dans les sols de sites sur lesquels des mousses d'extinction d'incendie ont été utilisées et de leur possible relargage et transfert des sols vers les eaux souterraines, les fluorotélomères doivent être suivis dans ces deux milieux. Ces résultats sont également en cours d'exploitation en vue d'aller plus loin sur la compréhension des mécanismes et paramètres physico-chimiques responsables du comportement des PFAS dans les sols, avec notamment d'autres types de sols testés. Ces interprétations seront également présentées.

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# PFAS Degradation During Hazardous Waste Incineration: A Pilot-Scale Study with Comprehensive Analytical Approach

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#### **Context and objectives**

PFAS, per- and poly-fluoroalkyl substances, are chemical compounds widely used in industry and everyday consumer products due to their non-stick, waterproof heat-resistant properties. However, these compounds are also resilient and persistent, posing major environmental challenges. Our study aims to evaluate the fate of PFAS during the thermal treatment of real PFAS-contaminated waste (fire-fighting foam AFFF, PFAS saturated adsorbent media,..).

#### **Material and Method**

This study was carried out on a R&D pilot unit, specifically designed to reproduce the incineration processes of hazardous waste under controlled laboratory conditions. Combustion temperatures (850-1100°C), post-combustion temperatures (1100-1200°C), residence times and other parameters such as flue gas composition (residual  $O_2$ % and water content) were set to mimic typical conditions of industrial-scale hazardous waste incineration.

An exhaustive and innovative analytical strategy was deployed to monitor PFAS during thermal treatment. The contaminated waste, combustion residues, and fumes were analyzed in LC/MS/MS to determine their content of targeted PFAS (49 substances). The OTM-45 method was particularly used to sample and analyze the gases at emission and thus calculate the destruction and removal efficiency (DRE) of PFAS. Analyses in combustion ionic chromatography (C-IC) were also performed to follow the fluorine (TF, TOF & HF). Finally, additional analyses were conducted in order to identify the presence of products of incomplete combustion (PICs) in the combustion gases. A non-target analysis (NTA) method was developed in liquid chromatography coupled with high-resolution mass spectrometry (LC-HRMS) and applied to the analytical fractions of the OTM-45 to identify semi-volatile PICs. Analyses according to the OTM-50 method, by canister sampling and GC/MS analyses, were also implemented to complete these research on very volatile species (like CF4, C2F6,..).

#### **Results and discussion**

R&D pilot tests allowed us to simulate waste incineration processes. In those conditions results showed good degradation of PFAS with DRE reaching up to 99.999% for POP compounds (PFOS, PFOA, PFHxS). The initial findings from applying non-targeted screening demonstrate its value by identifying substances that are typically not monitored by targeted analyses. It enables a more comprehensive understanding of the compounds that constitute the TOF, and the combination of these various analytical methods results in a more accurate characterization of PFAS fate during thermal treatment. This study provides crucial data on the effectiveness of thermal treatments for PFAS-containing waste and contributes to developing more efficient management strategies for these persistent pollutants.

Keywords: PFAS analysis, incineration, hazardous waste, destruction efficiency

# SARPI 🕢 VEOLIA

Retours d'Expérience sur le remplacement des émulseurs fluorés dans différents systèmes anti incendies : de la caractérisation aux traitements des déchets

**R**ETURNS OF **E**XPERIENCE FOR THE REPLACEMENT OF FLUORINATED AFFF IN DIFFERENT FIREFIGHTING SYSTEMS : FROM CHARACTERIZATION TO WASTE TREATMENT

**M**ATTHIEU **HIRRIEN** / **S**PECIAL PROJECTS AND **E**XPERTISE DEPARTMENT MANAGER - HEAD OF INNOVATION

Key words : pfas / afff / analysis / cleaning / rinse water / destruction / short chain / targets

Dans la multitude des problématiques rencontrés autour des PFAS, les émulseurs anti incendies fluorés sont, en terme de masse de PFAS, sans commune mesure avec les autres sources. Un fût de 200 L par exemple, peut contenir une dizaine de kg de PFAS alors que les seuils de potabilités sont en ng/l. Compte tenu de la mobilité de ces molécules, chaque utilisation est une potentielle catastrophe à venir et le remplacement des émulseurs fluorés est en cours à l'échelle européenne mais aussi dans d'autres pays. Les premières opérations ont lieu en France depuis mi 2024.

La présentation décrira des cas concrets réalisés en France ainsi qu'un autre en Australie pour apporter une mise en perspective. Dans la mesure du possible des données économiques seront utilisées pour mettre en contexte, de même que les réglementations concernées et les approches HSE utilisées lors des opérations.

Dans un premier temps seront présentées les premières étapes que sont la caractérisation des émulseurs (analyses complémentaires), des systèmes anti incendies (design, matières, ...) et la définition d'un protocole de vidange / nettoyage adapté. Puis, seront abordés, les moyens pour la vidange et le nettoyage, le traitement des eaux de rinçage sur place : leur performances et leurs limites. Enfin, la destruction des déchets résiduels sera également exposée : émulseurs vidangés, concentrats de traitement, EPI souillés, éléments du système non réutilisables.

# SARPI ThinkTech

# Two case studies demonstrating *in situ* groundwater PFAS remediation within high concentration source areas Deux études de cas illustrant la dépollution in situ de concentrations très élevées de PFAS dans la nappe en zones sources

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**Background:** Surface discharge of PFAS containing compounds, such as Aqueous Film Forming Foam, can result in significant levels of contamination at the capillary fringe and within the groundwater underlying these locations. Leaching of PFAS from the soils at the capillary fringe and from infiltration through the vadose zone may result in further discharge into the groundwater, with migration of these compounds creating environmental and human health risks to downgradient receptors. Stabilisation and retardation of PFAS in the capillary fringe and saturated zone using colloidal activated carbon (CAC) is becoming an important remedial option for risk mitigation through enhanced attenuation or in combination with source area soil removal or stabilization and plume treatment using a in situ barrier.

Two case studies are discussed that demonstrate different application approaches to this treatment, resulting from the requirements and conditions of each site. In the first site, PFAS use at a textile manufacturing facility in Belgium resulted in a diffuse PFAS plume comprising fluorotelomers 6:2, 8:2, 10:2 FTS and perfluorooctanoic acid (PFOA), within heterogeneous geology comprising sand and loamy clay. Nested monitoring wells revealed dissolved-phase PFAS concentrations exceeding 10,000 ng/L in the clay (capillary zone)—over 20 times higher than in the underlying saturated sand layer. Therefore, a solution was needed to eliminate the long-term PFAS flux from the source zone and prevent further plume development. In the second site, a fire training area at a Norwegian airport, excavation of PFAS impacted soils was scheduled. A residual groundwater plume existed in the gravel overlying crystalline bedrock immediately downgradient of the impacted soils. In order to target this plume and to avoid exacerbating the downgradient impact during the works due to disturbance and desorption, a barrier solution was desired to prevent further PFAS migration from the source area.

**Approach:** At the Belgian manufacturing site a pilot test was completed using low-pressure injection of liquid CAC using a rotating auger system to evenly distribute and coat both the saturated sand and confining clay layers. Nested monitoring wells were installed to validate the results. At the Norwegian airport, the CAC was mixed into clean sands and applied into an excavation perpendicular to plume propagation. This created a permeable reactive barrier to adsorb the contaminant influx, with the site entrance road being constructed over the top of the treatment zone.

**Results/Lessons Learned:** At the Belgian grid injection, the total PFAS concentrations in groundwater decreased by an average of 89% within the first month and reached reductions greater than 99% for all PFAS over five months. At the Norwegian site, downgradient Sum 35 compound PFAS concentrations reduced by 94%, with PFOS and PFOA reducing by 96% and 98% respectively, demonstrating the efficacy of the barrier.

Monitoring is ongoing at both sites, with the Belgian pilot now moving towards full-scale treatment. These promising results demonstrate the effectiveness of this *in situ* remediation approach as a solution for mitigating PFAS leaching and plume development, with broad-based applicability at similar sites to reduce environmental and public health risks.

# Effect of soil characteristics and mobilizing chemicals on perfluoroalkyl acid (PFAA) desorption during soil flushing

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In Europe alone, thousands of sites have been contaminated with per- and polyfluoroalkyl substances (PFASs). At the same time, current soil remediation techniques such as excavation and disposal of contaminated soil or in-situ immobilization are not sustainable. Soil flushing is another common remediation technique for soil contaminants, but only limited research has gone into finding an efficient soil flushing setup for perfluoroalkyl acids (PFAAs). This study screened an array of mobilizing chemicals (i.e. salts and surfactants) on their ability to enhance the desorption of a range of PFAA components in a soil flushing setup. Four salts and five surfactants were first tested in batch setup on four PFAS-contaminated soils from Flanders (Belgium) with contrasting physiochemical properties that are expected to influence mobility of PFAAs. The addition of mobilizing chemicals at 5 mg L<sup>-1</sup> generally facilitated desorption for all PFAA components in all soils, with the exception of the high PFAS-loaded, low organic carbon soil. Salts worked better than surfactants for shorter-chained PFAAs (PFBA, PFHxA & PFHxS), while surfactants outperformed salts for longer-chained PFAAs (PFOA, PFOS, PFNA & PFDA). Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> was the best salt as it reduces and hence dissolves iron, subsequently releasing PFAAs. The best surfactant was dodecylbenzene sulfonic acid (DBSA), of which the long apolar tail and sulfonate functional group are a strong combination to compete with adsorbed PFAAs. To practicalize batch results into a soil flushing setup, columns were filled with the four contaminated soils and 5 mg  $L^{-1}$  solutions were used to wash them. Preliminary experiments showed no effect of the flow rate on the desorption of PFAAs from the soil column, hence the fastest flow rate (i.e. 6 pore volumes per hour) was selected for further research. The most substantial results were observed when flushing with DBSA, as PFOS desorption increased from 13 to 44% of the total PFAS load in a highly organic soil and from 6 to 40% in a low pH soil. The use of mobilizing chemicals for flushing soils with low organic carbon and/or a high iron content was generally not beneficial and in some cases even exacerbating.





## Biopolymer based remediation of PFAS contaminated soils In Situ Treatment of an AFFF Contaminated Airport Site

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PFAS contamination from firefighting foams at airport sites has emerged as a significant environmental challenge in recent years. Unlike other contaminants, PFAS are particularly difficult to remediate due to their high mobility, persistence, toxicological uncertainties, and the technical limitations of current remediation technologies. The complexity of developing effective remediation strategies is further increased by site-specific factors such as the composition of PFAS compounds, soil properties and land use. Across the globe, numerous airport sites have been identified as PFAS hotspots, where decades of aqueous film-forming foam (AFFF) use have led to extensive soil and groundwater contamination.

#### The PFAS remediation task at hand

To address this challenge, Sensatec has conducted an in-situ remediation at one of these airport sites in late 2024 to January 2025. To maximize the success of the remediation, a feasibility study has been carried out beforehand in the company's soil and groundwater laboratory in Kiel. This preliminary investigation assessed the site-specific conditions, evaluated the effectiveness of different biopolymer treatments and optimized the remediation strategy accordingly. In this laboratory study, a PFAS removal rate of 95% in the soil has been achieved, demonstrating the high potential of the biopolymer solution for effectively mobilizing and removing PFAS from contaminated soil.

#### The remediation approach

The remediation process utilizes specially developed surface-active biopolymer compounds to effectively separate PFAS from the soil. The biopolymer solution infiltrated in the soil creates a more attractive water-soluble binding mechanism, enhancing the mobilization of PFAS from contaminated soil particles. As a result, the elution process facilitates the desorption of PFAS from soil particles, allowing their transfer into the pore water and subsequent transport toward a centrally located extraction well. With this hydraulic control measures, the PFAS-biopolymer complex is pumped toward the on-site water treatment system while preventing uncontrolled migration. The cleaned water can then by recycled.

The in-situ remediation has been conducted on an area of approximately 150 m<sup>2</sup>. Within the treatment zone, a centrally located monitoring well and four additional monitoring wells were installed to track PFAS mobilization and groundwater flow. To further assess the effect of the remediation process, two additional monitoring wells were installed—one upstream of the treatment zone to observe inflowing groundwater conditions and one downstream to evaluate potential PFAS load. The biopolymer solution was applied to the treatment area using irrigation hoses, ensuring even distribution across the surface. The remediation took place in a period of three months. To monitor the success of the remediation during the treatment, groundwater samples were collected from all monitoring wells at regular intervals, approximately every two weeks. Additionally, soil samples were taken to assess the progress of the remediation and to quantify the reduction of PFAS concentration in the treated soil. These sampling efforts provided key data for assessing the effectiveness of the applied biopolymer solution and allow for a comparative evaluation of field and lab scale.







Fig. 1: Impression of the treatment zone

#### **Remediation results in-situ**

To determine PFAS concentration in both soil and groundwater, target analysis was used. In the groundwater samples, a total of 27 individual PFAS parameters were analysed, with a detection limit of 0.05  $\mu$ g/L. For the soil samples, a 2:1 eluate as well as the soil itself was analysed for the same 27 individual substances. Additionally, to assess the potential release of precursor substances and their transformation into more mobile PFAS compounds, a TOP-assay was performed on the soil samples.

The initial loads of PFAS and their precursors in the solids and eluate in the 3 m thick unsaturated zone and in the uppermost meter of the saturated zone was recorded in five sub-areas of the test field. The flushing was carried out in several cycles over a total period of seven months by means of surface application of site water or the biopolymer condensate solution. The flushing water supplied to the aquifer was extracted via an abstraction well and, after conventional treatment using a gravel filter and water activated carbon, discharged into the rainwater drainage system.

After the first rinsing cycle with water over approx. 4 weeks, soil samples from the unsaturated zone in the solids already showed a removal rate of approx. 95 % (PFNA) and 35 % (PFOS). For the eluates, the removal was 97 % (PFNA) and 80 % (PFOS) respectively. Even with this first elution cycle, 400 g PFNA and 300 g PFOS were flushed out of the unsaturated soil zone. This corresponds approximately to the pollutant loads accumulating in the downstream contaminant plume within 10 years. After the subsequent two-week rinse with the biopolymer condensate solution and a four-week rinse with water, total contaminants of 98 % (PFNA) and 50 % (PFOS) was eliminated from the soil matrix. After completion of a further 6-week rinse with water, the remaining or still elutable pollutant concentrations in the soil were further monitored. In the course of the measurements, PFOS shows a significantly reduced decay behaviour compared to PFHxS and PFNA. Due to their surfactant-like character, the perfluorinated anions also sorb in the soil water/soil air interface. The different discharge behaviour of PFNA and PFOS in the unsaturated zone is attributed to the differing air/water interface activities of the two individual substances.

#### Service life of the sorption barrier / achievability of remediation targets

The knowledge gained from the balancing of the in-situ elution is used to calculate the service life of a sorption barrier to be erected in the plume to minimize the further input of pollutants into the uncontaminated aquifer. The service life of a sorption barrier will in any case increase with the decreasing substance concentrations in the inflow of the barrier. The reduction in the service life of the pollutant source will also result in a significant reduction in the required service life of the barrier.





The reduction of the current pollutant loads in the groundwater by approx. 80 % - can possibly be achieved by flushing the unsaturated zone alone. Achieving this goal is only questionable in the case of the less easily desorbable individual substance PFOS. Here, the results of the ongoing groundwater investigations will show whether the PFOS concentrations in the test field downstream will also be sufficiently reduced.

## Treatment of PFAS in industrial wastewater by advanced reduction process and electro-oxidation

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#### Keywords: PFAS, TFA, Advanced Reduction, Electro-oxidation, Industrial Effluent, Pilot

#### I. CONTEXT

Per- and polyfluoroalkyl substances (PFAS) are persistent pollutants found in both urban and industrial wastewater. Their high chemical stability, due to strong carbon-fluorine bonds, makes them difficult to remove using conventional treatment processes. Concentrations ranging from 15 to 1500 ng/L have been detected in 70% of the wastewater treatment plants studied (Lenka et al., 2021). In response to these concerns, European and French regulations have progressively restricted the use of certain PFAS and mandated their monitoring in water discharges.

Current mature technologies, such as activated carbon and membrane processes, can extract PFAS from water but do not destroy them, resulting in residual waste that must be managed. As an alternative, destructive treatment processes are emerging, including advanced electro-oxidation (EO) and UV/sulfite reduction, which have shown near-complete removal efficiencies under laboratory conditions (Fang et al., 2017; Liu et al., 2021). However, scaling these technologies to real effluents presents several challenges, notably the presence of cocontaminants, the formation of toxic by-products (e.g., trifluoroacetic acid (TFA)), and energy consumption.

#### **II. OBJECTIVES**

This study aims to develop and evaluate destructive treatment strategies for PFAS in complex industrial effluents. Two processes were investigated:

- Advanced Reduction Process (ARP) based on hydrated electron generation (e<sup>-</sup><sub>aq</sub>) via UV irradiation and chemical reducers (sulfites).
- > Electrochemical Advanced Oxidation Process (EAOP) to mineralize PFAS and potential by-products.

#### **III. METHODOLOGY**

The experiments focused on the treatment of groundwater contaminated with PFAS and industrial water contaminated with TFA using two destructive processes: Advanced Reduction Process (ARP) via UV/sulfites and Electrochemical Advanced Oxidation Process (EAOP) using the ElectRotate reactor.

The groundwater, containing 7 out of 20 PFAS (87 to 536 ng/L), was treated with ARP under UV irradiation in the presence of 20 mmol/L sulfites for 3 hours. EAOP was performed in 2L batch mode for 1 hour with a stainless steel cathode and boron-doped diamond anode. A current density of  $12.5 \text{ mA/cm}^2$ , a distance inter-electrode of 1 mm, a rotation speed of 15 rpm and a Na<sub>2</sub>SO<sub>4</sub> electrolyte (560 mg/L) was applied. Analyses focused on 20 PFAS degradation (HPLC-MS/MS).

For the industrial water, two types of samples were treated: raw effluent and concentrate obtained from a reverse osmosis pilot unit currently used to decontaminate the site. Each sample of 5 L was treated with ARP under UV irradiation in the presence of 20 mmol/L sulfites for 12 hours. EAOP was performed in batch mode for 12 hour with a stainless steel cathode and boron-doped diamond anode. A current intensity of 5 A, a distance interelectrode of 1 mm and 4 blades with a rotation speed of 15 rpm was applied. Analyses focused on Trifluoroacetic acid (TFA) contaminants degradation (HPLC-MS/MS).

These experiments allowed for the evaluation of the process efficiency under real conditions and helped identify challenges related to complex matrices.

#### **IV. RESULTS**

The experiments demonstrated effective removal of perfluorocarboxylic acids (PFCAs) in groundwater, with reduction rates ranging from 56% to 81% using ARP. In contrast, ARP showed limited efficiency for perfluorosulfonic acids (PFSAs), with removal rates below 40%. EAOP enabled complete degradation of the quantified PFAS, although the formation of chlorinated by-products (chlorates and perchlorates) was observed, highlighting the need to optimize operating conditions to minimize these undesirable compounds. EAOP showed moderate energy consumption of 9 kWh/m<sup>3</sup> after 1 hour of treatment, which is three times more efficient than a commercial EAOP system (30 kWh/m<sup>3</sup>). The ARP process showed an energy consumption of 6 kWh/m<sup>3</sup>, confirming the energetic and economic relevance of these technologies for treating PFAS-contaminated groundwater.



Figure 1: Summary of the concentrations of the 20 PFAS analyzed in the groundwater (left) and summary of the removal performance of the 7 quantified PFAS (right) by ARP (orange) and EAOP (yellow).

The results presented in Figure 2 illustrate the removal kinetics of one short-chain PFAS compounds trifluoroacetic acid (TFA) during EAOP treatment of both raw industrial effluent (green) and reverse osmosis concentrate (blue). After 6 hours of treatment, removal rates exceeded 99% for TFA in both matrices. These results confirm the effectiveness of EAOP in treating highly persistent and oxidized PFAS such as TFA, which is typically resistant to most other processes. We also identified the formation of perchlorates during the treatment, reaching concentrations on the order of g/L.

These findings highlight the robustness of EAOP for treating PFAS-contaminated effluents, including concentrated streams, and the importance of monitoring intermediate compounds to ensure complete mineralization and minimize secondary pollution. We are currently testing a sequential combination of advanced oxidation followed by ARP (UV/sulfites), which has been shown to increase PFAS defluorination (Liu et al., 2021) while limiting the formation of chlorinated by-products.



Figure 2: Evolution of TFA concentrations during EAOP treatment of the raw effluent (green) and reverse osmosis concentrate (blue).

#### **V. CONCLUSIONS**

Advanced reduction is effective on PFCAs under controlled conditions, but limited by matrix effects in real effluents. EAOP is a promising option for destructive treatment of concentrates, and offers a viable path to hybrid approaches combining separation (e.g., reverse osmosis) and destruction (EAOP). A 1 m<sup>3</sup>/h pilot unit is planned for deployment to treat real industrial effluents contaminated with TFA and other PFAS. Future work includes monitoring fluoride release, the limitation and/or destruction of perchlorates generated during treatment, and optimization of combined ARP/EAOP treatments for full-scale application.

# Comparative analysis of treatment processes to develop a solution adapted to the characteristics of PFAS pollution in water resources

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Keywords: Activated Carbon, Drinking Water, Nanofiltration, PFAS, Resin

**Innovation:** Conducting pilot tests, both in the laboratory and in situ, to choose a PFAS treatment solution while positioning the chosen solution in terms of its effectiveness on pesticide metabolites.

#### **Context and objectives**

Grand Annecy, a Public Establishment for Intercommunal Cooperation (EPCI), has had the "drinking water" competence since January 1, 2017, which it exercises through the action of the Water Directorate, formed by pooling the resources of the various structures responsible for water supply in the serviced area.

In this perimeter, the main source of drinking water supply is provided by 5 pumping stations in Lake Annecy.

The second strategic resource of the Grand Annecy community is the 'Iles' aquifer. Following the PFAS issue encountered in the neighboring municipality of Rumilly, Grand Annecy initiated the monitoring of these pollutants in drinking water at the end of 2022. These analyses then showed the presence of PFAS in these waters. The increase in PFAS concentrations led Grand Annecy to completely stop pumping from this aquifer in February 2023.

However, the 2021 master plan for drinking water supply highlights the importance of this resource for securing the overall drinking water production system of Grand Annecy.

Therefore, Grand Annecy wishes to study the feasibility of treating PFAS contained in these waters to have an alternative or complementary resource to Lake Annecy again.

#### Method

The study is based on a multi-criteria comparison of different PFAS treatment processes in water resources: performance, investment and operating costs, productivity and availability, footprint, environmental impact (waste, energy, greenhouse gas emissions (GHG), etc.). These criteria will be prioritized according to the project's constraints and issues, in consultation with the project owner.

To anticipate potential regulatory changes regarding pesticide metabolites, it is also requested to position PFAS treatments in terms of their effectiveness on these molecules.

The analysis of water samples focuses on 28 PFAS molecules. Additionally, it seemed relevant to also monitor the AOF parameter (adsorbable organic fluorine), which is often used as an indicator of the total PFAS concentration in the water.

The study covers the individual treatment of each PFAS initially contained in groundwater, as well as the reduction of the sum of 20 PFAS and the sum of 8 PFAS.

For the issue of pesticide metabolites, the considered molecules are the relevant and non-relevant substances established by ANSES (updated April 29, 2024).

This study consists of two phases.

Phase 1 involves laboratory trials on activated carbons, resins, and membrane treatments.

It was decided to test four qualities of activated carbon (AC) at different dosages (1 granular, 2 microgranular, 1 powder; 3 suppliers) at the laboratory scale to compare their PFAS capture efficiency. Granular activated carbon (GAC) or micro-granular activated carbon ( $\mu$ GAC) has the advantage of potentially being reactivated after use, while powdered activated carbon (PAC) must be disposed of.

Depending on the choice of membrane, membrane filtration can retain and concentrate various pollutants initially contained in the water. Given the variability in PFAS size, a nanofiltration (NF) membrane and a reverse osmosis (RO) membrane were tested comparatively.

For each test, the evolution of TOC and each PFAS concentration has been monitored.

Phase 2 is a minimum 6-month in-situ pilot testing phase, with the long-term goal of testing and comparing the effectiveness of 2 supports selected at the end of phase 1 (1 powdered activated carbon and 1 granular activated carbon, which require different implementation conditions) and thus more precisely estimating the performance and loading rates of the supports (saturation).

#### Results

The quality of the Iles aquifer water seems to vary little, with a total concentration of 28 PFAS ranging from 270 ng/L to 380 ng/L; on average, PFAS concentrations are as follows:



In fact, 7 PFAS are measured above the quantification limits; these are mainly short-chain PFAS (PFBA, PFPeA, PFHxA, PFHpA) in number (4 molecules) and concentration (expressed in ng/L); the other 3 are long-chain PFAS (PFOA, PFHxS, and PFOS) and are in the minority.

Regardless of the support (activated carbon or resin), we observe a decrease in PFAS concentrations in treated waters for the studied dosage range, and in most cases proportionally to the support dosage.

Powdered activated carbon seems particularly effective even at the lowest dosages.

For resins, tests conducted at pH 6.5 do not yield better results than at pH 8.

Effectiveness is similar between granular and micro-granular activated carbons and resins at equivalent dosages.

After NF and RO membrane filtration (recovery = 80%), no PFAS were quantified in the permeates (< LQ). The second RO treatment of the first permeate is limited (total recovery = 91%), and traces of PFHxA were measured in the permeate.

Given these results (laboratory tests) and the advantages and disadvantages of the considered processes, we decided to test at the in-situ pilot scale:

- ✓ 1 GAC column filter: classic process, easily exploitable, whose performance on PFAS needs to be verified;
- ✓ And 1 PAC pilot: higher technicality, but potentially better performance.

The 2 pilot units have been implemented on-site to monitor these 2 lines in parallel. The presented results are those obtained at mid-term (3 months).

The equivalent of 7000 BV was treated on GAC without filter breakthrough; the treated water is free of PFAS; even the shortest PFAS are still retained on GAC.

On the PAC pilot, the obtained results do not meet expectations; the first results obtained with short contact times (30 and 60 min) and low dosages (10 to 100 mg/L) show PFAS concentrations still too high in treated waters. The tests will be continued, particularly by increasing the activated carbon dosage.



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#### Thermal Conductive Heating with Reburn Technology for PFAS Remediation

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The remediation of per- and polyfluoroalkyl substances (PFAS) presents a major environmental challenge due to their persistence, chemical stability, and resistance to conventional treatment methods. Haemers Technologies has developed an innovative approach integrating Thermal Conductive Heating (TCH) with Reburn Technology to remove and destroy PFAS in situ, eliminating the need for secondary waste management.

TCH operates by applying uniform, controlled heating to contaminated soil, raising its temperature to 350-450°C. At these temperatures, PFAS compounds volatilize and migrate into the vapor phase. Unlike traditional thermal desorption, which captures and condenses PFAS vapors for external treatment, Haemers Technologies' system directly injects the extracted vapors into high-temperature Smart Burners operating at 1,400°C. This ensures complete oxidation of PFAS into harmless byproducts such as  $CO_2$ ,  $H_2O$ ,  $H_F$ , and  $SO_2$ , effectively eliminating the contamination without generating additional waste streams.

The pilot project at Korsør Firefighter School, Denmark, demonstrates the scalability and efficiency of this solution. Housed in a fully insulated 20-foot container, the system treats 15.5 m<sup>3</sup> of contaminated soil per batch across two treatment cycles. Advanced real-time monitoring, including hydrogen fluoride (HF) gas detection and non-target chemical analysis in collaboration with the University of Copenhagen, ensures process safety and efficiency. By eliminating reliance on secondary waste treatment, this integrated approach significantly reduces operational costs, energy consumption, and logistical constraints.

With its modular design and direct destruction capability, this technology offers a cost-effective, sustainable alternative to traditional landfill disposal or incineration, redefining PFAS remediation for large-scale applications.

The project is scheduled to take place in Q3 2024 and Q1-Q2 2025. During the conference, we will have the opportunity to present the latest results and insights gained from these field applications.

# A generic defluorination method for multiple classes of PFAS by means of solvothermal destruction

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#### Abstract

Per- and polyfluorinated substances (PFAS) are manmade chemicals known for their unique properties such as oil, dirt and water repellency, as well as thermal and chemical resistance. However, the highly inert nature of these compounds leads to extreme persistence in the environment and living organisms, resulting in long-term exposure and serious health effects. Despite significant regulations, highly scrutinized PFAS (like PFOA and PFOS) are often replaced by other (shorter) PFAS, thereby continuing to pose serious risks for both mankind and the environment. Up till now, PFAS waste is either disposed in dedicated salt cell landfills, or incinerated at elevated temperatures in rotary kilns (> 1000 °C); neither can be denoted as a sustainable solution. Alternative and more sustainable state-of-the-art PFAS mineralization techniques are promising, but still face challenges that hamper their applicability such as the incomplete degradation of long-chain PFAS into more stable and recalcitrant short-chain PFAS as well as the energy consumption of certain technologies.

Another destruction method involves a solvothermal mineralization of PFAS under alkaline conditions and mild temperatures. This process is carried out in a closed batch system, allowing for a controlled environment where the distribution of the starting components and (gas-phase) intermediates across the different phases can be closely monitored. Here, we report that this approach is highly effective for a wide range of different PFAS, and leads to (nearly) complete mineralization of different perfluorocarboxylic acids (PFCAs) and perfluorosulfonic acids (PFSAs) (Figure 1). Whereas ultra-short PFAS, such as trifluoroacetic acid and trifluoromethanesulfonate, are completely mineralized (100% F<sup>-</sup> ) in an aqueous alkaline solution at elevated temperature, the mineralization of longer chain PFAS proceeds with more difficulty due to a limited solubility of these compounds in the aqueous solution. Through the addition of a well-chosen co-solvent, their solubility could be increased, thereby effectively increasing the mineralization yield of the longer chain PFAS to a minimum of 90% for all evaluated substrates. As such, a generic and relatively mild procedure is provided that is able to destroy both (ultra-)short and long chain PFAS, which is currently non-existent in literature. In addition, unprecedented mechanistic insights are provided that bring understanding to the degradation pathways of different PFAS molecules under alkaline conditions. For PFCAs, decarboxylation is the first step, producing a fluorinated alkane that must be reabsorbed into the liquid phase for further defluorination. For PFSAs, desulfonation does not seem to occur, suggesting an alternative degradation pathway in which defluorination proceeds directly.



200 - 250 °C, 1 - 24 h 4 - 8 M KOH, (co-solvent)

KF + defluorinated organic compounds

Figure 1: Overall scheme of the mineralization of PFCAs and PFSAs.

#### Keywords

Sustainable PFAS remediation, solvothermal alkaline defluorination




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