2023 PFAS analytical exchange - TOP Assay Method Comparison

March 2023

Provided by Environment Agency (UK) In collaboration with University of the Basque Country (Spain), Vito NV (Belgium), Fraunhofer IME (Germany), Örebro University (Sweden), Wageningen University & Research (Netherlands), Luleå University of Technology (Sweden), German Federal Environment Agency (Germany), The French National Centre for Scientific Research (France)

For further information relating to this report please contact Alun James:
Alun.James@environment-agency.gov.uk

Steering Group and Authors

Alun James – Environment Agency (England, UK)
Belén González-Gaya– University of the Basque Country (Spain)
Bernd Göckener – Fraunhofer IME (Germany)
Ruben Kause – Wageningen University & Research (Netherlands)
Nicole Bandow – German Federal Environment Agency (Germany)
Leo Yeung – Örebro University (Sweden)
Pierre Labadie – The French National Centre for Scientific Research (France)
Kelsey Flanagan – Luleå University of Technology (Sweden)
Griet Jacobs – Vito NV (Belgium)

Glossary

EOF – Extractable organofluorine

FASA - Perfluoroalkane sulfonamides

HILIC - Hydrophilic interaction liquid chromatography

LC-MS/MS - Liquid Chromatography with tandem mass spectrometry

LOQ – Limit of Quantification

PFAAs - Perfluoroalkyl acids

PFAS - Per-and polyfluoroalkyl substances

PFCAs - Perfluoroalkyl carboxylic acids

PFSAs - Perfluoroalkane sulfonic acids

SPE – Solid phase extraction

TOF – Total extractable organofluorine

TOP – Total oxidisable precursors

WwTP – Wastewater treatment plants

Background

This work is coordinated through the NORMAN network, which is a platform that supports the exchange of information on emerging environmental substances and encourages the validation and harmonisation of common measurement methods and monitoring tools. This work follows on from previous work, NORMAN network PFAS Analytical Exchange[1] conducted in 2021. This showed a significant interest in total oxidisable precursors (TOP) assay analysis for perfluorinated substances, with 13 laboratories indicating the ability to conduct the TOP assay in water and 9 of these able to apply it to sediment or biota. The work also revealed that a further 8 laboratories are prioritising developing future TOP capabilities. In April 2022, the "PFAS analytical exchange - TOP Assay Method Comparison" activity was commissioned. The responses to this work are discussed here.

Introduction

There are over 4700 (Wang et al., 2021) known per-and polyfluoroalkyl substances (PFAS) and many of the structures and behaviours of PFAS which are poorly understood and subject to corporate secrecy. Different PFAS often have subtle differences in chemical make-up and this diversity makes their analysis notoriously difficult. Conventional targeted analysis can currently detect around 50 PFAS and many go undetected in environmental samples (Göckener et al., 2021; Karrman et al., 2019).

A number of methods are available that provide an indication of the PFAS burden of a sample. Some of the most commonly approaches includes measures of total fluorine, organofluorine, precursor and intermediates (Aro et al., 2022; Björnsdotter et al., 2021; Cousins et al., 2020; Göckener et al., 2020; Göckener et al., 2021; Guelfo et al., 2021; Taniyasu et al., 2022). The methods employed to quantify these are used in different applications to investigate and assess the risk from PFAS (Herzke et al., 2023).

A number of studies have demonstrated that in some environmental compartments, only a small amount of the total PFAS are identified by target analysis. For example Göckener et al. (2021) observed a significant increase in the sum of perfluoroalkyl carboxylic acids ($\sum$PFCA) in bream filets following TOP assay with an 18 – 91-fold increase. In contrast, the same study showed a closer agreement with targeted analyses in bream livers and herring gull eggs thus illustrating the complexity of PFAS in the environment. Furthermore a study based in the Nordic Environment found that 8% of the PFAS burden was detected in surface water, 9% in WwTP sludge, 26% in fresh water fish, 37% in marine mammals, and 42% in marine fish when targeted analysis was compared to EOF. Sources of the unidentified burden may include fluorinated pesticides, pharmaceuticals, and other low-fluorinated substances (Karrman et al., 2019).

Due to the limitation in targeted PFAS analysis there is increased interest in the application of total oxidisable precursors (TOP) assay analysis and its ability to overcome the shortfalls in targeted analytical methods. The methods rely on the high stability of Perfluoroalkyl acids (PFCAs) and Perfluoroalkane sulfonic acids (PFSAs) substances which are not readily degraded under environmental conditions. The majority of PFAS however contain C-atoms that are not fully fluorinated and have less stable bonds to other hydrogen atoms which are often undetectable with conventional targeted analysis. By breaking down the bonds of these polyfluorinated moieties (often termed precursors or intermediates), stable end products are formed that are more readily analysed by established methods.

TOP assay is a semi-destructive method used to measure all perfluoroalkyl moieties in a sample by the oxidative conversion of precursors (Houtz & Sedlak, 2012). The principle of the approach is to

---

[1] http://normandata.eu/sites/default/files/files/QA-QC Issues/2021 NORMAN network PFAS Analytical Exchange Final Report.pdf

create an oxidative environment of excess hydroxyl radicals (OH*) which converts PFAA precursors in the sample to PFCAs prior to analysis. In most approaches the OH* is produced by the thermolysis of persulfate under basic (pH >12) conditions.

TOP assay analysis is now commonly applied in PFAS research and is increasingly used by risk assessors and risk managers. Australia is one of the few countries that currently integrates TOP assay into PFAS management. For example the Queensland Department of Environment and Heritage Protection uses the TOP assay in regulating fluorine contents of aqueous film forming foams (Ross et al., 2021). With the increasing application of TOP assay, it is important to better understand the capability, limitations, precision, and accuracy of the technique. Recent evidence shows that efforts are being made to improve consistency in the methods. For example Ateia et al. (2023) made a number of recommendations for TOP assay analysis that included:

- Triple replicates of samples with and without TOP
- Targeted analysis should include ultrashort-chain PFAS
- Spike 20% of samples with precursors and intermediates
- Collect data on salinity, organic Carbon and pH of the sample, these measurements can provide indirect evidence to help explain data variability and confirm the complexity of the oxidation process.
- Calculate mole yield and degree of oxidation in matrix spike samples.

Furthermore a previous small scale inter-laboratory study by Nolan et al. (2019) showed significant variability between facilities (Nolan et al., 2019), potentially as a result of the lack of standardised methods highlighting the need to introduce consistency.

Objectives
- To better understand the TOP assay capabilities across the Norman Network including the type of media and status of the method.
- To ascertain method procedures commonly employed.
- To identify the current limitations of the analysis.
- To examine levels of quality control and detection limits of available methods.

Methodological Approach

In April 2022 the "PFAS analytical exchange - TOP Assay Method Comparison" activity was launched seeking volunteers to establish membership of a steering goup. Following a positive response from the NORMAN network membership, this steering group was formed and met for the first time on May the 4th 2022. In a series of subsequent virtual meetings, the group discussed the intended outcomes and developed a questionnaire aimed at better understanding TOP assay capabilities across the network. The questionnaire was circulated in September 2022. The responses were received by November 2022 and are summarised in the results section below.

It is important to note that the results from this work are semi-quantitative. Due to their subjectivity it was not possible to make fully quantifiable comparisons of the methods and performance of analytical procedures, this would require further work and interlaboratory studies.

Level of Response

A total of 11 laboratories responded to the questionnaire, of which 8 are research based, 2 governmental, and 1 commercial. The lack of participation from commercial laboratories was assumed to be a result of commercial secrecy and an absence from the NORMAN network. Three of

the laboratories were based in the Netherlands, 3 in France, 2 in Germany and one from Spain, Sweden and the UK. Eight of these were routinely performing TOP assay analysis and provided sufficient detail for this study. Two of the 8 laboratories had an official accredited PFAS method (targeted analysis). However, all laboratories have quality control measures and in-house validation. In general, the number of TOP assay samples analysed by each laboratory was low, i.e. 50-200 per annum. As some laboratories used different methods for different media, information on 15 methods were gathered. The relatively small cohort in this study needs to be considered when interpreting the results and conclusions.

Results

TOP assay capabilities

This section focusses on understanding the capabilities of the respondents to undertake TOP Assay analysis on different matrices. In total 43 different matrices were included in the questionnaire which fell into 8 categories (Table 1). The responses showed that the main capabilities exist in water, soil / sediment and biota; only 2 labs with the ability to analyse plant biota and human samples.

Table 1 : Categories and subcategories of the different matrices included in the questionnaire and the number of responses.

Category	No of Labs
Water - total	**4**
Water - Drinking Water	4
Water - Surface (fresh) waters	2
Water - Waste Water	3
Soil and Sediment - total	**4**
Soil and Sediment - Suspended Particulate Matter	2
Soil and Sediment - Sediment	3
Soil and Sediment - Soil	4
Biota (animals) - total	**4**
Biota (animals) - Fish (organ / tissue)	4
Biota (animals) - Invertebrates	3
Biota (animals)- Cetacean (organ / tissue)	1
Biota (animals)- Eggs	1
Biota (plants and algae)	**2**
Biota (plants and algae) - Seaweed	1
Biota (plants and algae) - Food (crops)	1
Biota (plants and algae) - Whole Plant	2
Biota (plants and algae) - Plant parts	2
Human	**1**
Human - Plasma	1
Air and gasses	0
Filter Paper / Cartridges / Passive discs	0
Products and Consumables	0

Methods employed by participants

The following section focuses on the methods used, the basis of the method, any modification, and the general performance of the method. Participants were asked to comment on any modification made, the reasoning behind this and their confidence in the method.

All methods for water and sediment were originally based on Houtz and Sedlak (2012). There were a number of published modified methods reported, including direct TOP assay (Göckener et al., 2021) and biota method (Simonnet-Laprade et al., 2019). Modifications reported by the laboratories included reagent amounts and concentration, longer oxidation times, heating method, additional cleaning for better blank results and the separation of inorganic particles. Although these were not applied consistently, in general reagent volumes and/or strength were increased when compared to the original methods.

Validation, quality assurance and performance

This section is designed to capture the key steps executed in the method which may influence the performance and provide information on successful or unsuccessful modifications. The validation questions will provide information on the confidence of the results, their repeatability, reproducibility, accuracy and precision.

- All of the responses undertook sample spiking
- 3 laboratories collected field blanks
- 6 laboratories included procedural blank
- 7 laboratories included instrument blanks
- 7 of the 8 subtracted the blank
- 7 of the 8 analysed for precursors following oxidation
- 3 laboratories have analysed for < C4 PFCA during validation
- All laboratories measured concentrations before and after analysis
- Only 1 laboratory has employed fluorine mass balance
- 6 of the 8 used mass labelled internal standards.

Typical Recovery Rates and analytical precision

This section is designed to capture semi-quantitative data on analytical capabilities. All of the laboratories analysed C4 – C14 PFCA and C4 – C10 PFSA, one laboratory routinely analysed for <C2. Fluorotelomer sulfonates were commonly used to monitor sample oxidation with 6:2 FTSA being most common, other substances used to monitor oxidation included HFPO-DA, PFMoPrA and PFECHS.

Each laboratory was asked to report on the typical recovery rates and analytical precision of their analysis and to select option from Table 2 below for each target analyte.

Table 2 : Shows the fixed response categories used in the questionnaire for the recovery rates and analytical precision.

Typical Recovery rates	Analytical Precision
<70%	>50%
70% - 90%	30% - 40%
90% - 110%	20% - 30%
110% - 130%	10% - 20%
>130%	<10%

The recovery rates were consistent between matrices with most laboratories reporting rates either in the 70-90% category or the 90% - 110%. In general, the higher recovery rates were reported for the C6 - C11 PFCA and C4 – C10 PFSA.

As with the recovery rates, analytical precision was relatively consistent between the different matrices. A high level of precision was reported by all laboratories with the majority falling in the 10-20%. Only 4 methods (3 by one laboratory) reported %RSD in the <10% and included a graphite clean-up and a polymetric solid phase extraction. The only PFAS where precision was in the 30-40% were C4, C5 and the long chain C16 and C18 PFCA whilst PFSA showed better analytical precision.

Limits of Quantification

This section is designed to capture semi-quantitively data on limits of quantification (LOQs), where the respondees were asked to report their limit of detection in the final measured extract into different rages from <0.5ng/L to >50ng/L.

The method designed for water matrices achieved the lowest LOQs with the majority reporting LOQs of <0.5ng/L and one laboratory reporting higher (5ng/L-10ng/L). Only one laboratory reported LOQs for C2 PFCA which was >50ng/L. While two laboratories achieved <0.5ng/L LOQs, generally higher LOQs were reported in sediments. For biota only one laboratory reported LOQ below 50ng/L at 10 – 50 ng/L.

Respondees were asked to provide information on analysis of PFAS outside the standard analytical suite with the ultra-short chain (C2 and C3 PFCA) and the long chain PFCA (C>14 PFCA). Only one laboratory routinely included analysis of C2 and C3 PFCAs (using SFC-MS/MS), and another laboratory was developing its capability with hydrophilic interaction liquid chromatography (HILIC). One laboratory provided data on C16 and C18 PFCA. These responses suggested research in the area is needed with poor recovery rates, low precision and high LOQ being reported for these compounds.

Conversion of data

TOP Assay data is reported in molar concentration and the concentrations of ∑PFCAs, ∑PFSAs and ∑pre-PFAAs are compared before and after oxidation to estimate the concentration of PFCAs resulting from the oxidation of unidentified precursors. Published examples of these methods are given in Janda et al. (2019) and Simonnet-Laprade et al. (2019). One way to determine mole yields is to use quality assurance/quality control samples spiked with known quantities of PFAS precursors and intermediates. By measuring these samples before and after TOP treatment, it is possible to calculate the mole yields of the spikes (Ateia et al., 2023).

Discussions.

Following the launch of the project the steering group formulated and circulated the questionnaire, where 11 laboratories responded. While the interest and application of TOP Assay is growing the level of response across the network suggests the community is still small. The lack of commercial laboratories suggests many are not an (active) member of the NORMAN network or were unwilling to disclose details of their method. Several commercial laboratories do offer the service and including these in any future work, in particular ring tries would significantly improve our understanding of the accuracy and precision of TOP assay methods.

Across the cohort the main capabilities exist in water, soil and sediment and animal biota analysis. While no laboratories reported on stormwater, biosolids, sludge and compost the nature of these matrices should be amenable to the methods currently being employed. Testing and validation of the methods is needed, especially for media with high inorganic matter and/or a high carbon content to ensure extraction and oxidation are complete. Similarly, following validation, the analysis of plant and human samples should be informed by other biota methods. The survey showed several gaps in

the ability to process more complex media such as waste, consumables (fabrics, carpets, cosmetics, plastic etc) air and gasses. Consideration towards the application and purpose of TOP Assay in other matrices is needed prior to development, alongside consideration for other, potentially more suitable methods for analysing total PFAS.

All laboratories analysed C4 – C14 PFCA, C4 – C10 PFSA and at least one PFCA precursor (commonly 4:2 FTSA or 6:2 FTSA) with only one laboratory analysing C3 – PFCA, and a different laboratory reported on analysis of C16, C18 PFCA and >C12 PFSA. All laboratories undertook accompanying targeted analysis, similarly, non-targeted methods are also used to provide valuable information on the potential PFAS in the sample (3 of the 8 laboratories reported undertaking this). Only one laboratory had used EOF in combination with TOP Assay.

Validation of these methods commonly included procedural blanks, instrumental blanks, sample spiking to monitored precursors and measurements of PFAS before and after TOP Assay, where appropriate, field blanks were also undertaken. It is strongly advised that the validation of the methods using these techniques is routinely employed.

Issues reported during the validation of methods by the cohort included blank contamination, sorption of PFAS to container walls, incomplete oxidation and poor recovery. To overcome these issues, modifications to the methods included differing reagent concentration, oxidation times, heating method, separation of the inorganic fraction and additional cleaning. All methods were originally based on the method by Houtz and Sedlak (2012).

Göckener et al. (2021) observed issues with the low extractability of known (spiked) precursors and incomplete oxidation (presumably due to high matrix load in the extracts). To overcome this, the ratio of solid sample to liquid reagent (S:L ratio) was reduced and the sample was neutralised and cleaned using SPE, by avoiding an extraction step the potential for losses is reduced (Göckener et al., 2021).

For biota the sample particle size is reduced followed by microwave assisted solvent extraction with methanol and hydroxide. The samples are then cleaned with a graphitized carbon and the solvents evaporated (pre-concentration) prior to oxidation, this is an adaptation of the Houtz and Sedlak method (Simonnet-Laprade et al., 2019).

There was little continuity in the modifications made by the laboratories, despite the reported issues being broadly similar. Differing reaction times, reagent strength, heating methods and additional cleaning were used in matrices other than water, laboratories generally increased reagent strengths and decreased S:L ratio. Increasing the amount / concentration of the reagents introduced a risk of over oxidising the sample that may attack the C-F skeleton and lead to the variations in the TOP assay products (Al Amin et al., 2021). This can lead to poor mass balance of the sample due to the difficulty in quantifying ultra-short chain PFAS. Consideration and measurement of the over oxidation of a sample should be given during method development.

Recovery rates across the cohort were high with the majority in the 90 -110% range, lower recovery rates were generally reported in the C4, C5, C12, C13 and C14 PFCA. With regards to the matrix, water and soil/sediment methods achieved the best recovery, with lower levels in animal biota.

Water matrices produced results with the lowest LOQs with the majority of labs achieving <0.5ng/L, these levels of quantification are suitable for current regulation with PFOS EQS at 65 ng/L in the UK and Norway. Recent advice from the USEPA however reduced the guidance for drinking water to 4 ng/L and if figures this low are to become established, analytical capabilities will need to improve to align with regulation. Responses for the soil / sediment suggested higher LOQs across the cohort, however low LOQs (<0.05 ng/L) were reported.

Issues raised by the cohort included the difficulty in the analysis of the C2 and C3 PFCA and the analysis of long chain PFAS and branched isomers. There were also issues reported with the calibration of FTSA compounds due to non-linear responses and data quality issues with FTCA, as FTCA may slowly transform into FTUCA. One significant point raised during the work was the lack of standards and there is a urgent need for a new "oxidation standard" which contains known, isotopically labelled precursor substances. This would allow greater confidence in analysis and facilitate the development and validation of methods.

A number of laboratories reported participation in proficiency testing with the majority focused on targeted PFAS analytical methods including methods including UNEP POPs, ISO21675 PFAS in water using SPE and LC-MS/MS and AQS Proficiency testing. Two laboratories indicated their participation in ring trials although it is not clear if this is TOP assay focused. All respondees were interested in interlaboratory studies with particular interest in water and sediment/soil.

Conclusions.

While there were small deviations in the methods employed by the cohort included in this study, there are examples of methods achieving excellent data quality in all matrices with high recovery rates, analytical precision and low limits of quantification. When applied to water matrices, data quality was high across all participants and suggests that the methods are reliable, and data can be interpreted with a high degree of confidence. For soil/sediment and biota analysis responses suggested an increased variability in performance, though, without the direct comparison of methods (interlaboratory studies) this study is unable to make recommendations on the suitability of method modifications.

The work showed the need for robust methodology and the application of procedural blanks, instrumental blanks, field blanks, and sample spiking to monitored precursors. Further recommendations include the need for additional cleaning to prevent blank contamination and carryover, and the associated impact from the sorption of PFAS onto container walls.

Further Work and outstanding questions

- Better understand ultra-short and long-chain PFAS to improve TOP Assay application and achieve better mass balances.
- Communicate the need for a new "oxidation standard" which contains known, isotopically labelled precursor substances.
- Provide continuity with S:L ratio, reagent concentration for soil/sediment and for biota.
- Establish if some washing buffers are more prone to losses of precursors.
- Understand the impact of evaporating the organic solvent.
- Refine methods and provide steer on methods which monitor over oxidation and the unwanted modification of the C-F skeleton.
- Comparison of direct TOP assay with TOP Assay following solid phase abstraction.
- The role of TOP Assay in influencing compliance and legislation

Many of these questions require further work. A recommendation for submission in 2023/2024 NORMAN work program is an expression of interest for participating in proficiency testing and ring trials in water, soils/sediments and biota matrices. Previous work in a small scale inter-laboratory study shows significant variability (Nolan et al., 2019), potentially as a result of the lack of standardised methods. Interested parties would need to agree upon materials and detailed method procedures to obtain meaningful data. This work showed an interest a range of matrices including groundwater, drinking water, surface water, soil, sediment, and biota. The potential for including a common pre-defined method that is employed be each laboratory, in combination with an inhouse method should be considered.

References

Al Amin, M., Luo, Y., Nolan, A., Robinson, F., Niu, J., Warner, S., Liu, Y., Dharmarajan, R., Mallavarapu, M., Naidu, R., & Fang, C. (2021). Total oxidisable precursor assay towards selective detection of PFAS in AFFF. *Journal of Cleaner Production*, *328*, 129568. https://doi.org/https://doi.org/10.1016/j.jclepro.2021.129568

Aro, R., Eriksson, U., Kärrman, A., Jakobsson, K., & Yeung, L. W. Y. (2022). Extractable organofluorine analysis: A way to screen for elevated per- and polyfluoroalkyl substance contamination in humans? *Environment International*, *159*, 107035. https://doi.org/https://doi.org/10.1016/j.envint.2021.107035

Ateia, M., Chiang, D., Cashman, M., & Acheson, C. (2023). Total Oxidizable Precursor (TOP) Assay—Best Practices, Capabilities and Limitations for PFAS Site Investigation and Remediation. *Environmental Science & Technology Letters*. https://doi.org/10.1021/acs.estlett.3c00061

Björnsdotter, M. K., Hartz, W. F., Kallenborn, R., Ericson Jogsten, I., Humby, J. D., Kärrman, A., & Yeung, L. W. Y. (2021). Levels and Seasonal Trends of C1–C4 Perfluoroalkyl Acids and the Discovery of Trifluoromethane Sulfonic Acid in Surface Snow in the Arctic. *Environmental Science & Technology*, *55*(23), 15853-15861. https://doi.org/10.1021/acs.est.1c04776

Cousins, I. T., DeWitt, J. C., Glüge, J., Goldenman, G., Herzke, D., Lohmann, R., Miller, M., Ng, C. A., Scheringer, M., Vierke, L., & Wang, Z. (2020). Strategies for grouping per- and polyfluoroalkyl substances (PFAS) to protect human and environmental health [10.1039/D0EM00147C]. *Environmental Science: Processes & Impacts*, *22*(7), 1444-1460. https://doi.org/10.1039/D0EM00147C

Göckener, B., Eichhorn, M., Lämmer, R., Kotthoff, M., Kowalczyk, J., Numata, J., Schafft, H., Lahrssen-Wiederholt, M., & Bücking, M. (2020). Transfer of Per- and Polyfluoroalkyl Substances (PFAS) from Feed into the Eggs of Laying Hens. Part 1: Analytical Results Including a Modified Total Oxidizable Precursor Assay. *Journal of Agricultural and Food Chemistry*, *68*(45), 12527-12538. https://doi.org/10.1021/acs.jafc.0c04456

Göckener, B., Fliedner, A., Rüdel, H., Fettig, I., & Koschorreck, J. (2021). Exploring unknown per- and polyfluoroalkyl substances in the German environment – The total oxidizable precursor assay as helpful tool in research and regulation. *Science of The Total Environment*, *782*, 146825. https://doi.org/https://doi.org/10.1016/j.scitotenv.2021.146825

Guelfo, J. L., Korzeniowski, S., Mills, M. A., Anderson, J., Anderson, R. H., Arblaster, J. A., Conder, J. M., Cousins, I. T., Dasu, K., Henry, B. J., Lee, L. S., Liu, J., McKenzie, E. R., & Willey, J. (2021). Environmental Sources, Chemistry, Fate, and Transport of Per- and Polyfluoroalkyl Substances: State of the Science, Key Knowledge Gaps, and Recommendations Presented at the August 2019 SETAC Focus Topic Meeting. *Environmental Toxicology and Chemistry*, *40*(12), 3234-3260. https://doi.org/https://doi.org/10.1002/etc.5182

Herzke, D., Nikiforov, V., Yeung, L. W. Y., Moe, B., Routti, H., Nygård, T., Gabrielsen, G. W., & Hanssen, L. (2023). Targeted PFAS analyses and extractable organofluorine – Enhancing our understanding of the presence of unknown PFAS in Norwegian wildlife. *Environment International*, *171*, 107640. https://doi.org/https://doi.org/10.1016/j.envint.2022.107640

Houtz, E. F., & Sedlak, D. L. (2012). Oxidative Conversion as a Means of Detecting Precursors to Perfluoroalkyl Acids in Urban Runoff. *Environmental Science & Technology*, *46*(17), 9342-9349. https://doi.org/10.1021/es302274g

Karrman, A., Wang, T., Kallenborn, R., Langseter, A., Ræder, E., Lyche, J., Yeung, L., Chen, F., Eriksson, U., Aro, R., Fredriksson, F., & Grønhovd, S. (2019). *PFASs in the Nordic environment*. https://doi.org/10.6027/TN2019-515

Nolan, A., Grimison, C., Lavetz, R., Slee, D., Lim, C., Centner, M., McGrath, S., Symons, B., & Bowles, K. (2019). Improving measurement reliability of the PFAS top assay. Proceedings of the 8th International Contaminated Site Remediation Conference, Australia.

Ross, I., Storch, P., Schaefer, T., & Ramsden, N. (2021). Fluorine Free Foams. In (pp. 23-50). https://doi.org/10.1201/9781003024521-3

Simonnet-Laprade, C., Budzinski, H., Maciejewski, K., Le Menach, K., Santos, R., Alliot, F., Goutte, A., & Labadie, P. (2019). Biomagnification of perfluoroalkyl acids (PFAAs) in the food web

of an urban river: assessment of the trophic transfer of targeted and unknown precursors and implications [10.1039/C9EM00322C]. *Environmental Science: Processes & Impacts*, *21*(11), 1864-1874. https://doi.org/10.1039/C9EM00322C

Taniyasu, S., Yeung, L. W. Y., Lin, H., Yamazaki, E., Eun, H., Lam, P. K. S., & Yamashita, N. (2022). Quality assurance and quality control of solid phase extraction for PFAS in water and novel analytical techniques for PFAS analysis. *Chemosphere*, *288*, 132440. https://doi.org/https://doi.org/10.1016/j.chemosphere.2021.132440

Wang, Z., Buser, A. M., Cousins, I. T., Demattio, S., Drost, W., Johansson, O., Ohno, K., Patlewicz, G., Richard, A. M., Walker, G. W., White, G. S., & Leinala, E. (2021). A New OECD Definition for Per- and Polyfluoroalkyl Substances. *Environmental Science & Technology*, *55*(23), 15575-15578. https://doi.org/10.1021/acs.est.1c06896