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CRITICAL REVIEW

The quest for the perfect "total PFAS" method: how can the total oxidisable precursor (TOP) assay be made reliable?

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Abstract
Per- and polyfluoroalkyl substances (PFAS) make up a large and complex class of manmade chemicals. They have been widely used in numerous industrial branches and are incorporated into many consumer products. Today, there is a consensus on the fact that PFAS are present in all environmental compartments and that populations all over the world are subjected to them via internal exposure. It has been estimated that thousands of individual PFAS have been manufactured and marketed since the 1950s, to which impurities present in commercial products and intermediate environmental transformation products should be added. Since it is unrealistic to be able to individually identify, detect and quantify all the PFAS present in a sample, several analytical approaches have been developed to assess the presence of "hidden/unseen" PFAS. One of these, known as the total oxidisable precursor (TOP) assay, was first described in 2012. Basically, it converts some PFAS, hereafter referred to as precursors, into stable terminal products readily measurable by routine target methods. This review is based on more than 100 studies in which the original TOP assay was simply applied or optimised. The review found that the TOP assay was selective, sensitive, applicable to many matrices, useful within a forensic context, inexpensive, and easy to implement and has been assessed in the literature on a wide range of precursors. However, this method comprises many subtleties and has some flaws that operators should be made aware of so that they may be addressed as far as possible. Finally, this review tries to lay the foundations for better practices and quality assurance/quality control measures, in order to improve accuracy and reliability of TOP assay results.

Keywords PFAS · TOP assay · Surrogate method · Total PFAS · DTOP · PhotoTOP · Oxidisable precursor · Quality assurance/quality control (QA/QC)

Introduction

Per- and polyfluoroalkyl substances (PFAS) make up a large and complex class of manmade chemicals recently defined as "fluorinated substances that contain at least one fully fluorinated methyl or methylene carbon atom (without any H/Cl/Br/I atom attached to it), i.e. with a few noted exceptions, any chemical with at least a perfluorinated methyl group (-CF_3) or a perfluorinated methylene group (-CF_2-) is a PFAS" [1]. The first forms of PFAS were discovered in the 1930 s and their industrial production started at the beginning of the 1950 s [2, 3]. They have been widely used in numerous industrial branches (for instance as processing aids for fluoropolymer manufacture) and commercial applications (for instance coating products to provide water and oil repellency, stain resistance and soil release) [4]. Consequently, they are found in many consumer products (e.g. lubricants and greases, floor polish, carpets, leather, paper and packaging, cosmetics, pesticides, sports articles, textiles and upholstery, cook- and bakeware) [4]. The first evidence of their occurrence in the environment, wildlife and humans was reported in the late 1990 s [5–7]. This is obviously a consequence of their large-scale use combined with the unique chemical and thermal stability of the C-F bond. Today, there is a consensus on the fact that they are found in all environmental compartments and that populations the world over are subjected to them via internal exposure [8]. Furthermore, two of the most well-studied PFAS (PFOA (perfluorooctanoic acid) and PFOS (perfluorooctane sulphonic acid)) have been associated with a variety of health

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problems such as cancer, thyroid disease, pregnancy-induced hypertension and impaired immune function [9]. However, it has been estimated that thousands of individual PFAS have been manufactured and marketed since the 1950 s [10], to which impurities present in commercial products (residual raw material, oxidation or unintentional synthetic by-products) and intermediate environmental transformation products should be added.

So far, only a limited number of PFAS can be quantitatively detected by target analysis [11–13]. This lack of quantification is due to the limited number of analytical standards available (fewer than 100), difficulties associated with the chromatographic detection of certain PFAS (especially for ultra-short-chain PFAS or zwitterionic PFAS) and trade secrets, which prevent open access to knowledge on the exact chemical structure of many PFAS. Furthermore, to anticipate regulatory requirements, companies regularly market novel PFAS as fluorinated alternatives to legacy PFAS [14, 15], thus complicating the task of understanding the full extent of PFAS contamination.

Faced with such challenges, it is unrealistic to be able to individually detect and quantify all the PFAS present in a sample and a pragmatic approach is needed to assess the PFAS burden. One possible solution resides in the implementation of methods able to provide quantitative information on PFAS that are difficult if not to say impossible to measure for the reasons previously mentioned. For this purpose, different analytical approaches have been developed, such as extractable organic fluorine (EOF) [16], absorbable organic fluorine (AOF) [17], and the total oxidisable precursor (TOP) assay [18].

Based on more than 100 studies (Table S1), the present paper aims to provide a broad overview of the total oxidisable precursor (TOP) assay, a promising approach to highlight the presence of "hidden/unseen" PFAS, i.e. undetected by conventional target analysis. This paper addresses (a) the principle behind the method, (b) its advantages over other surrogate methods/non-targeted techniques (NTS) for organofluorine determination, (c) criticisms, (d) its limitations and how to circumvent them, (e) recommendations in order to provide accurate, reliable results. Unlike previous reviews [19, 20], this review focuses on the weaknesses of the TOP assay then provide recommendations on good practices as well as QA/QC measures.

Principle behind the method

The total oxidisable precursor (TOP) assay, first described in 2012 by Houtz and Sedlak [18], was originally applied to urban runoff water samples. By oxidative digestion at an elevated temperature (around 85 °C) for several hours (usually 6 h), the TOP assay can convert some PFAS, herein after referred to as precursors, into stable terminal products (mainly perfluoroalkyl carboxylic acids (PFCAs)), which are readily measurable by liquid chromatography-tandem mass spectrometry. In this article, the term "precursors" refers to PFAS with the potential to be oxidised into other PFAS under laboratory-controlled conditions. Consequently, this definition does not cover all types of PFAS, since some can remain intact after the TOP assay [14, 21].

In brief, precursors present in the sample are exposed to hydroxyl radicals (•OH) produced from persulphate ($S_2O_8^{2-}$) thermolysis under alkaline conditions (pH > 12) in an aqueous medium. Hydroxyl radicals react via hydrogen atom abstraction along with a less prominent electron transfer process [22].

$$S_2O_8^{2-} + \text{heat} \rightarrow 2 \cdot SO_4^{-} \qquad \text{(persulphate thermolysis)}$$

$$\cdot SO_4^{-} + OH- \rightarrow SO_4^{2-} + \cdot OH \quad \text{(sulphate radical conversion to hydroxyl radical)}$$

Basically, two types of precursors can be considered: perfluoroalkyl sulphonyl derivatives (C_nF_{2n+1}-SO_2-R) and fluorotelomers (C_nF_{2n+1}-C_pH_{2p}-R'), where R and R' are various functional groups. This distinction is important since the pattern of terminal products generated after the TOP assay is different. Hence, the excess of hydroxyl radicals generated by persulphate thermolysis oxidises a perfluoroalkyl sulphonyl derivative to yield a single PFCA. For instance, perfluorooctane sulphonamide (FOSA; C_8F_{17}-SO_2-NH_2) is quantitatively converted into PFOA (C_7F_{15}-CO_2H) [18]. Fluorotelomers react to form a suite of terminal products of varying chain lengths. For instance, 6:2 fluorotelomer sulphonate (6:2 FTSA; C_6F_{13}-C_2H_4-SO_3H) is converted into a suite of PFCA, including perfluoropentanoic acid (PFPeA; C_4F_9-CO_2H), perfluorohexanoic acid (PFHxA; C_5F_{11}-CO_2H), perfluorobutanoic acid (PFBA; C_3F_7-CO_2H) and perfluoroheptanoic acid (PFHpA; C_6F_{13}-CO_2H) [18]. The oxidation of fluorotelomers in the TOP assay appears to unzip the chains, resulting in a suite of PFCAs with shorter chains than the parent substances. This process does not occur to such an extent for perfluoroalkyl sulphonyl derivatives, as only one PFCA is generally formed.

It is important to maintain a high pH during the oxidation step to promote •SO_4^- conversion to •OH and limit degradation of the PFCAs being formed [11, 21]. Indeed, several studies have shown that sulphate radicals (•SO_4^-), produced by thermolysis of persulphate, can transform PFCAs into PFCAs with a shorter chain length, especially under acidic conditions (pH ≤ 3) [23–27]. In contrast, PFCAs do not undergo further oxidation by •OH [18, 21, 28–30]. Interestingly, Liu et al. [31] compared acid and alkaline persulphate digestion with several PFAS, including perfluorinated and polyfluorinated ether sulphonates (PFESA) and legacy fluorotelomers. They concluded that both approaches could provide more confidence in assigning chain length of primary precursors and detecting emerging PFAS, such as Nafion™-related fluorinated ether sulphonates. However, their uncommon digestion conditions (heating in a pressure cooker to 120 °C) limit any comparison with other studies.

After the TOP assay, the measured PFCA concentration is the sum of PFCAs initially present in the sample (before the assay) and PFCAs formed from the oxidised precursors. It is worth noting that the TOP assay converts all precursors to PFCAs whereas, in the environment, some of them (for instance perfluorooctane sulphonamide (FOSA)) may be transformed into perfluoroalkyl sulphonic acids (PFSAs) as end-stage by-products, e.g. perfluorooctane sulphonic acid, PFOS [18, 21]. In addition, PFSAs initially present in the sample are stable since they did not react with sulphate or hydroxyl radicals [21]. However, some studies have mentioned a net increase in some PFSAs upon oxidation [30, 32–36]. In some cases, it was suggested that this observation could be related to elevated alkaline conditions, which can initiate hydrolysis of a number of environmental PFSA precursors, partially converting them into PFSAs rather than the expected PFCAs [21, 36]. It is also likely that the presence of more easily oxidisable chemicals in a sample (organic matter, organic solvents) may play a role too. Hence, hydroxyl radicals react first with these radical scavengers, leaving some PFAS to undergo hydrolysis, which partially converts them into PFSAs [21].

To counteract some drawbacks of the TOP assay (detailed further below), some adjustments have been suggested by different researchers. For instance, some authors activated persulphate by ultraviolet (UV) light instead of heat, still under alkaline conditions. This alternative method is known as the UV-activated TOP assay [37–39]. Other approaches have investigated replacing potassium persulphate by oxidising agents such as ozone [40], UV/H_2O_2 [41] or UV/TiO_2 [42]. The latter is known as the PhotoTOP assay. Some of these new approaches (UV/H_2O_2, UV-activated or ozone TOP assay) have shown several promising advantages, including enhanced preservation of the perfluoroalkyl chain length of the precursors, an improved ability to analyse samples with high concentrations of organic matter and a shorter reaction time. However, they warrant further evaluation, development and optimisation before being applied as a routine method. They will not therefore be addressed in the following sections.

Initially, the TOP assay was only performed on liquid samples. For solid matrices such as soil, sediment or textiles, an extraction step was usually carried out beforehand. To account for non-extractable precursors in these samples, a modified TOP assay was suggested where, instead of oxidising extracts, small amounts of samples may be directly exposed to the oxidising agent. This method is known as the direct TOP assay (dTOP) [33, 43]. To clearly distinguish between the TOP assay protocol with and without preliminary extraction, the acronyms suggested by Lange et al. [20] (eTOP and dTOP) should be adopted in future articles.

Advantages of the TOP assay

Compared with other surrogate analytical methods for organofluorine determination, TOP assay approaches have several advantages, including having been assessed with multiple precursors by many different research teams.

Specificity and sensitivity

Currently, the TOP assay is considered the most selective technique for quantifying total PFAS [44]. In contrast to other surrogate analytical methods available for organofluorine determination (e.g. EOF, AOF or TROF (total reducible organofluorine assay) [45]), the TOP assay is specifically for PFAS. Fluorinated compounds (such as certain pharmaceuticals, pesticides or inorganic fluorinated ions) that are not considered PFAS according to the recent terminology [1], are not detected. Consequently, the risk of false positives is greatly reduced. However, some PFAS are stable in the TOP assay or oxidised to yield usually unmonitored end products (see below). Therefore, this approach and its alternative methods (e.g. dTOP and PhotoTOP) are expected to underestimate the proportion of precursors, though the scale of this underestimation cannot be easily assessed.

Unlike the other surrogate analytical methods, the TOP assay is extremely sensitive (Table S2). The limits of quantification (LOQ) achieved are generally similar to targeted PFAS analysis (in the low part per trillion range).

Applicable to many different matrices

The TOP assay was first developed as a research tool and applied to quantify precursors in urban runoff [18]. Since then, it has been applied to characterise various aqueous and nonaqueous matrices, including groundwater [46–49], drinking water [50, 51], wastewater [52–55], landfill leachate [56, 57], precipitation [58], sediment [59–61], soil [46, 62, 63], dust [64, 65], compost [66, 67], food packaging [34, 68], aqueous film-forming foam (AFFF) concentrates [69–72], insecticides [73], consumer products [74, 75], textiles [32, 76, 77], animal tissue [12, 78, 79], vegetables [80] and human serum [30, 81].

The dTOP assay, specially developed for solid samples, has directly been used on matrices such as soil [82–84], sediment [85], suspended particulate matter [33, 86], eggs from laying hens [43], and textiles [87, 88]. The PhotoTOP assay has been applied to textiles [87], PFAS-coated paper and technical PFAS products [42].

Assessed with many different precursors

The degradation profiles of more than 40 PFAS have been studied under TOP assay conditions as described in the literature. For many of them, comparable data are available (Table S3), but for a few, only partial or non-comparable information was provided (Table S4). For many of them, model precursors disappeared completely, yet total molar yields were not always close to 100%. This may be due to an inability to quantify some oxidation end products (due to too high LOQs, untargeted end products or loss of TOP endpoints during analytical steps, for example). According to Cioni et al. [30], n:2 FTSA were less reactive than n:2 FTCA with the same number of fluorinated carbons, and reactivity decreased for longer fluorotelomers.

A useful tool within a forensic context

Some authors used the TOP assay data to go beyond the simple observation of the analytical changes in PFCA concentrations before and after oxidation and applied it as a semi-quantitative method. Houtz et al. [46] used the ratio of linear to branched isomers of perfluorinated homologues to infer the manufacturing origins of PFAS in an AFFF-contaminated area (electrochemical fluorination (ECF) versus fluorotelomer manufacturing process). Later, this approach was refined using a statistical technique (Bayesian inference) to take into account analytical uncertainties, incomplete recoveries and variability in product yields following oxidation [69, 89]. In this case, the authors applied their method to reconstruct the original perfluorinated chain lengths, manufacturing origin and concentrations of precursor compounds in AFFF-impacted environments. Combined with spatial relationships between sampling locations and major PFAS sources, this approach can help distinguish the fingerprints of different PFAS sources [11, 89]. Rodowa et al. [90] used the TOP assay data as a proxy for the breakthrough of oxidisable PFAS on GAC systems because it occurs before PFOSs and PFOAs. Göckener et al. [91] suggested that the TOP assay may also be used to monitor trends of unknown or unscreened PFAS, thus observing shifts in production (e.g. from legacy to emerging PFAS) and the enforcement of PFAS restrictions.

Inexpensive and easy to implement

The TOP assay uses a simple chemical reaction and does not require specialised equipment other than a high-pressure liquid chromatograph coupled to a tandem mass spectrometer, which is an instrument now found commonly in both research and commercial laboratories. Therefore, this method offers practical advantages over other approaches (e.g. AOF, EOF, NTS) which require specialised equipment and expert knowledge. Although duplicate samples need to be analysed (before and after oxidation), the TOP assay is inexpensive compared with the investment needed to perform other approaches.

Criticisms

Many papers have highlighted limitations of the TOP assay, sometimes arising from a misunderstanding of the scope of this method. For instance, the aggressive oxidation conditions of the TOP assay have often been criticised for not replicating some of the transformation processes that occur in abiotic or biotic environments [92]. More specifically, the TOP assay and the PhotoTOP assay convert some precursors, such as perfluoroalkyl sulphonamides, into PFCAs, whereas they would naturally be transformed into PFSAs [18, 87, 93]. Moreover, the TOP assay is often considered as a worst-case scenario, since its strongly oxidative conditions may greatly exceed those of natural processes occurring under typical environmental conditions [13, 94, 95]. Thus, the results are not representative of the precursor mass that is susceptible to oxidation in the environment, and the concentration of the targeted oxidation endpoints is likely to be higher than those that could be released in the field [92, 94]. Finally, some authors have pointed out that the TOP assay does not form all of the final and intermediate transformation products that could be formed in the environment [13, 43, 92]. However, although this assay does not replicate the transformation pathways that occur in the environment, it is above all an important tool for assessing the presence of unknown precursors and providing valuable information on potential natural degradation processes [43]. It was not developed as a tool to mimic natural transformation and metabolism processes and it is definitely not a predictor of the environmental endpoint breakdown of precursors.

Since the chemical structure of the oxidised precursors is not fully preserved under TOP assay conditions, it is not possible to identify them unless they have been included in the target PFAS list and detected beforehand in the samples. However, the TOP assay provides insights into the chemical structure of unknown precursors [62, 95]. The pattern of PFCA products upon oxidation, the presence of branched or specific oxidation endpoints (e.g. PFMOPrA from ADONA) allow for some inferences as to precursor chain length or method of synthesis [43, 59]. For instance, the presence of PFSAs after oxidation is not observed for precursors that do not contain sulphonic groups [30, 81]. Under TOP assay conditions, some precursors produced by ECF yield almost exclusively a single PFCA with the same number of carbon atoms as the precursor (with molar yields over 90%), as well as a mixture of branched and linear isomers [78, 96, 97]. Conversely, precursors produced by fluorotelomerisation yield a suite of linear PFCAs of different chain lengths [18, 64]. In most cases (Table S3), n:2 fluorotelomer oxidation produces a suite of PFCAs with no more than n perfluorinated carbon atoms, the PFCAs usually formed with the greatest molar yield having n-1 perfluorinated carbon atoms. Interestingly, the PhotoTOP assay offers more structural information than the dTOP assay because it conserves the chain lengths of precursors better (less chain shortening) [42, 87]. Other approaches, such as AOF or EOF, do not provide such information, as they do not preserve the perfluoroalkyl moieties at all.

TOP assay limitations and how to circumvent them

The TOP assay and its alternative methods undoubtedly have some weaknesses that should be known and addressed insofar as possible. The following section focuses on these key issues.

Loss of precursors following a prior extraction step

The TOP assay was originally applied directly to water samples [18]. To implement the TOP assay on solid matrices (e.g. soil, sludge, sediment, textiles, fish tissue), some authors have used an organic solvent extraction [32, 46, 62, 76] or ion-pairing extraction [78] prior to oxidation. This requires the comprehensive extraction of precursors with various polarities, having stronger or weaker interactions with the matrix. This additional step inevitably leads to bias, as the extraction's effectiveness strongly depends on the extraction conditions used [91]. Some authors have honestly recognised the inability of their extraction protocol to effectively extract the full suite of precursors present in the samples they analysed [64, 76, 78]. After the extraction step, the organic solvent has to be evaporated since it may lower the oxidation yield as a radical scavenger. This additional step may lead to a loss of some PFAS by volatilisation, again increasing the underestimation of the amount of precursors present [21, 32]. Furthermore, Macorps et al. [61] found that PFCA patterns obtained after oxidation of model precursors were different when tests were performed on spiked solvent or spiked water. They recommended performing conversion experiments under conditions similar to those used for samples.

To circumvent this discrimination of certain PFAS due to solubility or volatility characteristics, Göckener et al. [13] developed the direct TOP assay (dTOP), a variant of the TOP assay that works with solid samples without prior extraction. However, dTOP also has some flaws. Due to the general aqueous experimental setup of the dTOP assay, an increasing loss of long-chain PFAS was observed after oxidation, most likely due to sorption to soil particles [83]. These authors suggested carrying out an additional extraction step on the remaining soil particles after the dTOP assay with an appropriate organic solvent in the original vessel. The probable inclusion of previously non-extractable precursors in the dTOP method could limit comparability with other methods needing an extraction step (e.g. NTS, target analysis and EOF) [86]. Finally, dTOP suffers from poor sensitivity, as only small samples can be used to enable complete oxidation [59]. The PhotoTOP assay has also been applied to paper and fabric samples by direct oxidation [42]. Direct fabric oxidation generated many different PFCAs, but no PFAS were found in either the oxidised or unoxidised fabric extracts.

Loss of volatile or semi-volatile PFAS

An evaporation step is needed prior to oxidation when a previous extraction step is carried out with solvents, since they consume most or all the hydroxyl radicals. In addition, the TOP assay is usually performed at temperatures above 80 °C, which are likely to trigger the volatilisation of some PFAS. Both steps can lead to the loss of volatile or semi-volatile PFAS such as fluorotelomer alcohols (n:2 FTOH), sulphonamides and sulphonamido ethanols [59, 98]. According to Zhu and Kannan [76], this explains the variable and poor recoveries reported in the literature for some volatile PFAS.

To prevent this loss, a few specific precautions have been implemented by various authors. Mumtaz et al. [99] tightened the bottle lids and properly sealed them with Parafilm™ before the oxidation step, making sure they kept a headspace volume of less than 1 mL. Zhu and Kannan [76] added 1 mL of water to their combined extract before concentration under a nitrogen stream.

Some PFAS are not affected by the oxidation process

Research on the fate of different PFAS during the TOP assay has shown that some of them are totally recalcitrant under these oxidation conditions. A tentative list based on the literature has been provided in Table S5. As a general rule, PFAS without at least one C-H bond for •OH attack are stable under TOP assay conditions [31].

Some authors have considered these recalcitrant PFAS as "new terminal products" which should be included in the TOP assay analyte spectrum [14, 100]. It is definitely a good laboratory practice to screen for as many PFAS as possible, including non-oxidisable ones (at least prior to oxidation) in order to obtain the most accurate insight on the PFAS burden of a sample.

Interference due to high salt content after TOP oxidation

The TOP assay gives a solution containing large amounts of SO_4^{2-} and other ions resulting from the addition of the basic persulphate solution then the pH adjustment after oxidation. This procedure hampers the subsequent quantitative determination of oxidation endpoints by reducing the effectiveness of MS ionisation [101]. To avoid injecting large quantities of salt into the LC instrument, either solid-phase extraction (SPE) or appropriate dilution prior to injection have been suggested [14, 47]. However, several authors have noticed that the high amount of salt reduced the extraction recovery of ultra- and short-chain PFAS in anion exchange-based methods [62]. Furthermore, Joerss et al. [100] observed lower absolute recoveries of internal standards in an oxidised sample aliquot than in unoxidised aliquots. To assess the interference to SPE recoveries due to high salt content, we conducted our own experiment in which we spiked an ultrapure water sample and an oxidised blank with 18 precursors (unpublished). We performed SPE and compared recoveries (Fig. 1). The recoveries were adversely affected for at least eight precursors. Additionally, as the literature has pointed out, an evaporation step after SPE risks losing more PFAS by volatilisation.

To remove excessive SO_4^{2-} and Cl^- in the solution after a TOP assay and analyse ultra-short-chain PFAS, Wang et al. [102] used a sequential combination of ion chromatography (IC) and anion exchange SPE cartridges. To avoid using SPE, other analytical treatments have also been carried out (Table S1), such as liquid-liquid extraction [30, 32], dilution with methanol and cleaning with ENVI-Carb^{TM} [66], addition of organic solvents and chilling overnight at −10 °C [101] and the precipitation of salts with organic solvents [12, 47, 79, 103]. PhotoTOP may be considered an interesting alternative, since it does not result in a solution containing large amounts of salts, thus reducing sample preparation efforts prior to injection [42, 87].

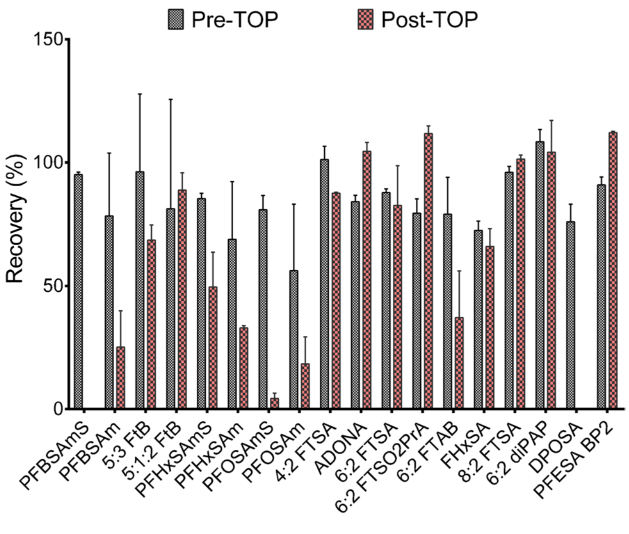

Fig. 1 Effect of high salt content on precursor recoveries following SPE (Pre-TOP, ultrapure water spiked with precursors; post-TOP, oxidised and neutralised blank spiked with precursors)

Matrix complexity can preclude complete conversion of precursors

The presence of competing organic molecules or minerals (e.g. reduced metals) within a sample may consume most or all the hydroxyl radicals (•OH) and could interfere with the complete oxidation of precursors [92, 96, 104]. The substantial concentration of precursors, which is the case of AFFF, can also lead to exhaustion of the oxidant [36]. The sample matrix can also affect the pH, which may modify the optimal oxidation conditions ($pH > 12$) [105]. Consequently, some precursors can remain partially or totally intact after the TOP assay, or can form oxidation intermediates instead of the expected endpoints. Incomplete oxidation can thus distort and underestimate the TOP assay results.

To prevent the scavenging of oxidant within the sample, several different approaches have been suggested in the literature. One option is to use harsher conditions than those initially set by Houtz and Sedlak [18] (i.e. more oxidant, a longer heating time and extra oxidation cycles). In general, the effect of increasing the amount of oxidant used was larger than the improvement observed by increasing the heating time [30]. However, it is very difficult to set up the optimal conditions as they depend on the sample's background and composition, and thus need to be optimised for each type of sample. Moreover, multiple applications of the TOP assay or increasing the amount of oxidant used has the drawback of adding salt to the samples [41]. Based on a few experiments, Zweigle et al. [42] assumed that, when ensuring a long enough oxidation time, matrix-independent oxidation can be achieved with PhotoTOP. A second approach proposed in the literature was to lower the concentration of competing molecules by diluting the sample prior to oxidation [11, 36, 70]. Such a dilution approach could help obtain consistent oxidation performances across samples, but requires the prior knowledge of the PFAS level and possible presence of radical scavengers [47]. A third approach proposed to reduce the presence of dissolved organic carbon by performing an ENVI-Carb clean-up step prior to the TOP assay [106]. Such an approach was recommended in the case of samples containing a high amount of organic matter, such as biosolids, composted materials and paper waste. Another proposal intended to reduce the presence of dissolved organic carbon was to perform a pre-treatment step with a mild oxidant such as hydrogen peroxide prior to extraction and oxidation [104]. To ensure the perfect optimisation of oxidative conditions and the full conversion of precursors after oxidation, different strategies have been implemented. In some experiments, a labelled model precursor (usually ^{13}C8-FOSA) was added to all samples prior to oxidation as an indicator of effectiveness [21, 65, 96]. Complete oxidation was then confirmed by measuring recovery of the known end products of the effectiveness indicator used (^{13}C8-PFOA in the case of ^{13}C8-FOSA). This assumes that the labelled model precursor has a higher concentration than other precursors present in the samples and that it is not much more easily oxidisable than them [105, 107]. The use of other commercially available ^{13}Cx-labelled fluorotelomers and sulphonamides is less straightforward. This is because they may oxidise to unlabelled end products (e.g. PFCAs) — thereby positively interfering with target PFAS — or oxidise to labelled PFCAs, thereby interfering with either the labelled internal standards used to quantify target PFCAs or the labelled surrogates used to monitor extraction effectiveness [36, 108]. Several authors included a quality control (QC) by spiking samples with unlabelled precursors [52, 56, 59, 79, 109]. The acceptance criterion in this case was the absence of the spiked precursor material after the TOP assay [12, 110]. However, a spiked QC (usually ultrapure water) assumes that the sample matrix does not preclude the complete conversion of precursors, and spiking samples with unlabelled precursors requires additional analysis (spiked and unspiked samples) because their terminal products are added to those from native precursors. Another suggested approach was to perform the TOP assay under various conditions (changing the dilution rate, reaction time and/or oxidant dosage, for example) [30, 87, 105]. If the levels of target end products measured following the different experiments are the same or do not significantly increase, then the oxidation process is presumed to be completed. This assumes that the number and conditions of experiments are sufficiently broad to reach the precursors' complete oxidation. This practice is meaningful when analysing raw materials such as firefighting foam concentrates or analysing heavily contaminated samples with unknown precursors. Finally, the absence of target precursors after the TOP assay can also be considered a criterion of complete oxidation that is easy to implement. In the PFAS National Environmental Management Plan (NEMP) [111], this criterion was translated into a post-oxidation ratio test for aqueous or soil samples ($\sum$ [PFAA precursors]/$\sum$ [Total PFAS]). However, this assumes that the analytical method used targets a large number and a wide variety of precursors, and that their recoveries and analysis in the post-TOP matrix are complete and reproducible.

The TOP assay's accuracy depends on the range of target end products

The vast majority of studies focused their post-TOP screening on PFCAs with at least four carbon atoms ($C \geq 4$) (Table S1). However, many authors reported that other stable end products besides PFCA ($C \geq 4$) should be considered [18, 30]. Firstly, the TOP assay as commonly performed does not capture ultra-short-chain PFCAs ($C \leq 3$) (less than 20% of the studies referenced in Table S1), despite the fact they can make a marked contribution to the molar mass balance of some precursors after oxidation [62, 96]. This limitation stems from the difficulty in analysing ultra-short-chain PFAS in the reaction mixture, high in ion content, and the resulting need to use a dedicated method with ion chromatography/mass spectrometry [62, 112]. Göckener et al. [91] pointed out that reduction in chain length depends on oxidative conditions (especially for dTOP). In some cases, these conditions could increase the formation of ultra-short-chain PFCAs, a blind spot in many studies. Moreover, the screening for ultra-short-chain PFCAs after oxidation could reveal the presence of CF_3- or CF_3-CF_2-containing precursors, such as some pharmaceuticals and agrochemicals [59]. However, the yields of ultra-short-chain PFCAs from all these chemicals in the TOP assay are not fully known [62, 81, 113].

It should be noted that not all the end products are PFCAs. Based on the literature, a tentative list of the PFAS to target after oxidation has been provided in Table S6. Several studies highlighted the importance of revising the current approach, which considers only PFCAs ($C \geq 4$) as terminal products of the TOP assay [14, 62, 114, 115]. It is recommended to include far more terminal products in the TOP assay analysis spectrum to obtain a more comprehensive picture of the unknown PFAS fraction in a sample.

Presentation and interpretation of TOP assay results is inconsistent across studies

The simplest way to present the results is to calculate the net generation of precursors as a difference and a ratio between corresponding pairs of endpoint concentrations after oxidation and before oxidation [74, 81, 116]. To take into account analytical uncertainties, they applied a cutoff of 20% change in endpoint concentrations. Specifically, if the ratio was ≥ 1.2, the difference was calculated as the endpoint concentration after oxidation minus the endpoint concentration before oxidation. If the ratio was <1.2, the difference was set to zero [81] or changes in concentration were considered not observable [107].

In some studies, the molar concentration of each endpoint after oxidation was corrected by subtracting the molar concentration before oxidation, as well as the molar contribution of all the targeted precursors quantified in the unoxidised sample [58, 61, 117–120]. The latter can be calculated from the expected molar conversion rates to endpoints of each targeted precursor. It assumes that their individual degradation profiles are known under the experimental conditions used. A molar concentration of non-targeted/unknown precursors was inferred as follows:

$$[\text{non} - \text{targeted/unknown precursors}] = \sum[\text{endpoints}]_{\text{after}-\text{TOP}} - \left[\text{endpoints from quantified precursors}\right] - [\text{endpoints}]_{\text{before}-\text{TOP}}$$

It assumes that one mole of each non-targeted/unknown precursor produced one mole of endpoints with a similar chain length, as their conversion yields were unknown [61, 119]. The total molar concentration of PFAS can be approximated by adding together the endpoint molar concentrations before the TOP assay ($[\text{endpoints}]_{\text{before-TOP}}$), the molar concentration of targeted precursors before the assay and molar concentration of non-targeted/unknown precursors ([non-targeted/unknown precursors]) [52].

A more realistic assessment of the presence of non-targeted/unknown precursors is obtained by taking into account the endpoints generated by oxidation of detected precursors. Otherwise, there is a risk of overestimating the molar concentration of non-targeted/unknown precursors. Similarly, it is speculative to assume that the lack of quantification of target precursors after oxidation means a 100% conversion to known endpoints without knowing their individual degradation profiles under the experimental conditions used.

There is no standardised TOP assay protocol or regulatory guidance

Since TOP assay effectiveness depends on factors such as sample matrix and PFAS concentration, various modified versions of the original method [18] have been suggested. The published methods differ in aspects such as amount and concentration of oxidation agent, oxidation time or the kind of sample treatment prior to oxidation (e.g. extraction) and the list of targeted end products. Consequently, the precision and reliability of TOP assay results can be questioned and they may not be comparable from one laboratory to the next [92, 95].

Since oxidation conditions are specific to sample type, it appears impossible to recommend a single, standardised protocol. However, it is essential that the analytical conditions are precisely described (e.g. dilution, pre-treatment steps, oxidation conditions) and that a suite of quality controls are implemented, as suggested below. In any case, the TOP assay should be considered a semi-quantitative analytical tool for establishing the presence of precursor compounds in a sample. As a proof of its value, it was included as a monitoring technique in the Australian government's national environmental management plan [111].

Recommendations on best practices and quality assurance/quality control measures

The aim of this paper was not to provide a consensus method, which appears utopic in light of the diversity of situations that could be encountered when analysing environmental samples or consumer goods. It rather provides advice on how to improve the reliability and accuracy of TOP assay results. It also reinforces and completes recommendations given by Ateia et al. [19].

Preliminary conditions required for the TOP assay and suggested acceptance criteria (Table 1)

1. The list of targeted terminal products should include ultra-short-chain PFCA and as many other PFAS identified as terminal products as possible (Table S5). Known unoxidisable PFAS such as GenX™ (HFPO-DA) should also be screened for before and after oxidation. The list of targeted precursors should be as exhaustive as possible. Based on the referenced studies (Table S1), at least 40 PFAS could be reasonably measured. It is recommended to use isotopically mass-labelled analogues whenever possible, and ideally the unoxidisable ones should be spiked at the beginning of the procedure and the oxidisable ones just after oxidation.

2. If a pre-treatment step is required before performing the TOP assay (e.g. solvent extraction, SPE, filtration, purification), the recovery of all the targeted PFAS (precursors and terminal products) should first be investigated in at least five different matrices similar to those of the samples analysed (i.e. not in ultrapure water). Three replicates per matrix should be analysed and the results mentioned in the study report. "Replicate" in this case

Table 1 Recommended quality control and acceptance criteria for the TOP assay, based on a literature review (bold figures in brackets refer to the subsection number of the "Recommendations on best practices and quality assurance/quality control measures" section in the article)

Pre-required analytical validation[a]		Steps of the analytical procedure	Routine quality control[a]	
Performance criteria	Acceptance criteria		Quality control criteria	Acceptance criteria
• A greater number of PFAS is targeted, including US-PFAS (1)	• ≥ 40	Analytical method	• Addition of stable internal standards	
• No significant loss of target PFAS (endpoints, precursors and unoxidisable PFAS) (2) • No contamination of blanks (6)	• Recoveries = 100 ± 20%, n ≥ 5 matrices • [PFAS] < ½ LOQ	Pre-treatment[b]	• Unoxidised blanks (**8**) • Addition of probe precursor (e.g. ^{13}C8-FOSA) (**9**)	• [PFAS] < ½ LOQ
		Oxidation	• pH measurement (**9**)	• pH ≥ 12
• No significant loss of terminal products including unoxidisable PFAS (3)	• Recoveries ≥ 60%, n ≥ 5 matrices	Post-oxidation treatment	• pH measurement (**9**) • Addition of oxidisable internal standards	• pH ≥ 12
• Precursors are detectable in post-TOP matrix (4) • No loss of precursors (heated controls) (5)	• Recoveries ≥ 60%			
• No contamination of blanks (6)	• [PFAS] < ½ LOQ			
		Analysis	• Oxidised blanks (**8**) • Probe precursor (e.g. ^{13}C8-FOSA) (**9**) • Quantification of precursors (**9**) • ≥ 3 replicates (**10**)	• [PFAS] < ½ LOQ • Molar yield > 85% • < LOQ or < 5% of initial concentration • RSD ≤ 20%
• The individual degradation profile of each targeted precursor is known (7)		Reporting of results		

[a] The prerequisite analytical validation and routine quality control provided in this table are focused on the TOP assay. General quality control measures and analytical performance criteria required for analytical methods (sensitivity check, calibration, field blanks, sample collection, etc.) have not been included here since they can be found in different standard/guidelines dedicated to target PFAS determination

[b] Extraction, filtration, clean-up, etc.

means pre-treating at least three different aliquots of a sample and not three injections of one pre-treated aliquot. It must be kept in mind that spiking PFAS in a sample, especially solid matrices (e.g. soil, sludge, sediment), does not perfectly mimic the behaviour of PFAS, which have already been sorbed to the matrix for many years [83]. The issue of the best ways to spike a sample is outside the scope of this paper. Based on technical guidelines [121, 122], the minimum requirement for individual recoveries should be in the range of 80–120%.

3. The recoveries of terminal products after oxidation should also be investigated. In the absence of precursors, it means that the concentration of the targeted terminal products should not be significantly different before and after oxidation. In practice, targeted stable endpoints (such as PFCAs and PFSAs) and their isotopically-labelled internal standard could be added to different samples and quantified before and after oxidation [81, 98, 123]. These samples should be fairly representative of the analysed matrices and ideally free of precursors and targeted endpoints. For this purpose, Simonnet-Laprade et al. [119] used mineral water and Fontainebleau sand. Based on published results, the mean recoveries of terminal products and known unoxidisable precursors after oxidation should be above 60% [81, 98]. This acceptance criterion is probably more realistic than that given in the HEPA [111] guidelines: "The total PFAS concentration post-TOP Assay should be greater or equal to the total PFAS concentration pre-TOP Assay, which signifies no material losses observed in preparation steps, noting a decrease of up to 10% might be expected due to normal analytical variability".
4. Moreover, the ability of the analytical method to quantify the potential presence of precursors in the post-TOP matrix must also be checked. Indeed, ionisation interference due to high salt content or loss during analytical

treatments used to reduce the salt content could impair precursor detection, leading to false and optimistic assertions on oxidation performance. This is a common pitfall of many studies, which restrict their target list after oxidation to terminal products. It is of utmost importance to know whether precursors are still present after oxidation, since this provides information on the completeness of the oxidation. In practice, precursors and their isotopically-labelled homologues could be spiked at known concentrations in oxidised samples, having previously checked their absence. This addition should be performed just after oxidation and before any of the analytical treatments implemented prior to injection in the LC/MS. These experiments should be carried out in triplicate, preferably in at least five different sample types.

5. Some authors suggested using heated controls of different PFAS without adding oxidant or NaOH to assess potential loss upon heating [14, 18]. If the recoveries of terminal products after oxidation have already been checked and were within the satisfactory range, these controls are only recommended for targeted precursors. Without real feedback from experience, mean recoveries above 60% could be suggested for precursors in the post-TOP matrix and heated controls.
6. Unoxidised and oxidised procedural blanks should be performed to monitor background contamination in laboratory materials (e.g. solvents, tubing, reagents and sampling containers). In some studies, TOP assay results were subtracted from the background concentrations found in procedural blanks [21, 60, 78, 123]. In our opinion, this practice is not recommended. In the case of background contamination, the report should provide information on the contamination level and its recurrence (e.g. relative standard deviation across the analytical series) and the LOQ calculation should take into account this contamination. PFAS levels should systematically be below half of the LOQ.
7. To accurately assess the presence of non-targeted/unknown precursors, it is necessary that the individual degradation profile of each targeted precursor was previously determined under the oxidation conditions used and in matrices similar to those analysed. These results should be used to correct the molar concentration of each endpoint after oxidation.

Routine quality control and acceptance criteria (Table 1)

8. Unoxidised and oxidised procedural blanks should be added to each sample series. They must be below half of the LOQ determined during the method's validation. The study should report information on blank results and the LOQ's calculation should take into account any recurrent contamination above half of the LOQ.
9. To ensure that all precursors are efficiently converted to end products, several control measures should be implemented. None is fully satisfactory, but together they minimise the risk of incomplete oxidation.

    - First, an oxidation effectiveness indicator such as ^{13}C8-FOSA could be added to all samples [21, 96, 107, 124]. As stated by Larsson [21] himself and Pettersson et al. [107], this does not mean that all potential PFAS precursors have been fully converted. Other unknown compound groups with different chemical moieties than -SO_2NH_2 may require an additional oxidation agent or increased reaction time. Based on published results, molar yields of ^{13}C8-FOSA to ^{13}C8-PFOA should be above 85% in every sample.
    - The absence of all targeted precursors should be checked after oxidation [78, 104, 111, 125]. In the absence of native targeted precursors before oxidation, samples should be spiked to evaluate the potential for incomplete conversion of known precursors. Any oxidised samples with quantifiable targeted precursors should be excluded [11, 107, 115]. The HEPA [111] guidelines give the following criteria: "for aqueous samples, sum of [PFAA precursors] divided by sum of [Total PFAS] <5%" and "for soil samples, sum of [PFAA precursors] divided by sum of [Total PFAS] <10%". All these criteria are relevant only if a great number of precursors are analysed and only if it has been previously demonstrated that their recovery and analysis in the post-TOP matrix were complete and reliable, respectively.
    - It is important to maintain highly alkaline conditions throughout the oxidation process [36, 117, 126]. Indeed, an insufficient amount of NaOH can affect oxidation and lead to PFCA degradation. Consequently, pH should be monitored after the addition of NaOH to the samples [97, 127]. If necessary, more NaOH should be added [115]. The pH should also be checked after oxidation [71]. Any samples with a post-TOP pH < 12 should be discarded [36, 71, 115, 117, 126].

10. To confirm the repeatability of the whole procedure, the TOP assay should be performed at least in triplicate on each sample. Avoid replicate injections of a single oxidation because this practice overestimates repeatability. Several studies performed triplicates, which could be considered a minimum requirement [18, 30, 35, 63, 80, 100, 128–130]. Relative standard deviation across replicates should be provided with the TOP

assay results. Based on authors' observations, relative standard deviation (RSD) across sample replicates should be ≤ 20% [14, 30, 48, 69, 100].

Conclusion

Conventional target analysis can only be used to analyse a small proportion of the PFAS present in the environment or consumer goods. To improve our understanding of potential PFAS exposure, fate and transport in the environment, there is a clear need for methods capable of capturing PFAS other than those captured in routine target analysis. To reduce the unidentified organic fluorine fraction, several approaches have been developed.

Among them, the TOP assay has established itself as a useful tool. This surrogate method has proved to be selective, sensitive, applicable to many matrices, assessed in the literature on many different precursors, useful within a forensic context, inexpensive and easy to implement. However, this method comprises many subtleties and has some flaws that should be known and addressed insofar as possible. By following the recommendations provided in this review, both the accuracy and reliability of TOP assay results could be improved. Proficiency tests can also help to improve robustness and standardise the TOP assay procedure within laboratories, but very few have been organised up to date. This is a major and important challenge, since the European Commission recently adopted a Total PFAS limit (0.5 µg/L) for drinking water, but did not specify a dedicated method. To earn the confidence of regulators, relevant quality assurance/quality control measures should be systematically implemented when performing the TOP assay.

The TOP assay does not quantify or identify the structures of individual precursors. It is an indirect approach that only provides rough estimates of oxidisable precursors. It does not account for any PFAS that are not oxidisable or that oxidise to substances that are not included in the targeted analyte list. Consequently, it may underestimate PFAS abundance. It is a screening method for evaluating the presence of unknown precursors in a sample. To identify these unknown precursors, further analytical investigations such as NTS are needed. It is important to be aware of these limitations when interpreting data from a TOP assay. Currently, there is no true "total PFAS" method and to obtain a comprehensive overview of the PFAS burden of a sample, several complementary analytical approaches are still necessary. The TOP assay should definitely be included in this panoply of approaches.

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Declarations

Conflict of interest The author declares no competing interests.

References

1. OECD. Reconciling Terminology of the Universe of Per- and Polyfluoroalkyl Substances: Recommendations and Practical Guidance. Organisation for Economic Co-operation and Development; 2021 9 July 2021. Report No.: ENV/CBC/MONO(2021)25.
2. Gaines LGT. Historical and current usage of per- and polyfluoroalkyl substances (PFAS): A literature review. Am J Ind Med. 2023;66(5):353–78.
3. Okazoe T. Overview on the history of organofluorine chemistry from the viewpoint of material industry. Proc Japan Acad Series B. 2009;85(8):276–89.
4. Glüge J, Scheringer M, Cousins IT, Dewitt JC, Goldenman G, Herzke D, et al. An overview of the uses of per- And polyfluoroalkyl substances (PFAS). Environ Sci Process Impacts. 2020;22(12):2345–73.
5. Giesy JP, Kannan K. Peer Reviewed: perfluorochemical surfactants in the environment. Environ Sci Technol. 2002;36(7):146A-52A.
6. Kannan K, Koistinen J, Beckmen K, Evans T, Gorzelany JF, Hansen KJ, et al. Accumulation of perfluorooctane sulfonate in marine mammals. Environ Sci Technol. 2001;35(8):1593–8.
7. Kannan K, Corsolini S, Falandysz J, Fillmann G, Kumar KS, Loganathan BG, et al. Perfluorooctanesulfonate and related fluorochemicals in human blood from several countries. Environ Sci Technol. 2004;38(17):4489–95.
8. Kuo K-Y, Chen Y, Chuang Y, Lin P, Lin Y-J. Worldwide serum concentration-based probabilistic mixture risk assessment of perfluoroalkyl substances among pregnant women, infants, and children. Ecotoxicol Environ Saf. 2023;268:115712.
9. Obsekov V, Kahn LG, Trasande L. Leveraging systematic reviews to explore disease burden and costs of per- and polyfluoroalkyl substance exposures in the United States. Exposure and Health. 2023;15(2):373–94.
10. OECD. Toward A New Comprehensive Global Database of Per- And Polyfluoroalkyl Susbtsances (PFASs). Organisation for Economic Co-operation and Development; 2018 4 May 2018. Contract No.: ENV/JM/MONO(2018)7.
11. Antell EH, Yi S, Olivares CI, Ruyle BJ, Kim JT, Tsou K, et al. The Total Oxidizable Precursor (TOP) Assay as a Forensic Tool for Per- and Polyfluoroalkyl Substances (PFAS) source apportionment. ACS ES&T Water. 2023;4(3):948–57.
12. Rupp J, Guckert M, Berger U, Drost W, Mader A, Nodler K, et al. Comprehensive target analysis and TOP assay of per- and polyfluoroalkyl substances (PFAS) in wild boar livers indicate contamination hot-spots in the environment. Sci Total Environ. 2023;871: 162028.

13. Göckener B, Fliedner A, Rüdel H, Fettig I, Koschorreck J. Exploring unknown per- and polyfluoroalkyl substances in the German environment – The total oxidizable precursor assay as helpful tool in research and regulation. Sci Total Environ. 2021;782: 146825.
14. Zhang C, Hopkins ZR, McCord J, Strynar MJ, Knappe DRU. Fate of per- and polyfluoroalkyl ether acids in the total oxidizable precursor assay and implications for the analysis of impacted Water. Environ Sci Technol Lett. 2019;6(11):662–8.
15. Wang Z, Cousins IT, Scheringer M, Hungerbühler K. Fluorinated alternatives to long-chain perfluoroalkyl carboxylic acids (PFCAs), perfluoroalkane sulfonic acids (PFSAs) and their potential precursors. Environ Int. 2013;60:242–8.
16. Miyake Y, Yamashita N, Rostkowski P, So MK, Taniyasu S, Lam PKS, et al. Determination of trace levels of total fluorine in water using combustion ion chromatography for fluorine: A mass balance approach to determine individual perfluorinated chemicals in water. J Chromatogr A. 2007;1143(1–2):98–104.
17. Wagner A, Raue B, Brauch HJ, Worch E, Lange FT. Determination of adsorbable organic fluorine from aqueous environmental samples by adsorption to polystyrene-divinylbenzene based activated carbon and combustion ion chromatography. J Chromatogr A. 2013;1295:82–9.
18. Houtz EF, Sedlak DL. Oxidative conversion as a means of detecting precursors to perfluoroalkyl acids in urban runoff. Environ Sci Technol. 2012;46(17):9342–9.
19. Ateia M, Chiang D, Cashman M, Acheson C. Total Oxidizable Precursor (TOP) Assay─best practices, capabilities and limitations for pfas site investigation and remediation. Environ Sci Technol Lett. 2023;10(4):292–301.
20. Lange FT, Freeling F, Göckener B. Persulfate-based total oxidizable precursor (TOP) assay approaches for advanced PFAS assessment in the environment – A review. Trends Environ Analytic Chem. 2024;44:e00242.
21. Larsson P. Total Oxydizable Precursor Assay: Applications and Limitations. A Study on the Occurrence of Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) in Chinese Wastewater Treatment PLants: Örebro University; 2020.
22. Olmez-Hanci T, Arslan-Alaton I. Comparison of sulfate and hydroxyl radical based advanced oxidation of phenol. Chem Eng J (Lausanne). 2013;224(1):10–6.
23. Bruton TA, Sedlak DL. Treatment of aqueous film-forming foam by heat-activated persulfate under conditions representative of in situ chemical oxidation. Environ Sci Technol. 2017;51(23):13878–85.
24. Sun B, Ma J, Sedlak DL. Chemisorption of perfluorooctanoic acid on powdered activated carbon initiated by persulfate in aqueous solution. Environ Sci Technol. 2016;50(14):7618–24.
25. Yin P, Hu Z, Song X, Liu J, Lin N. Activated persulfate oxidation of perfluorooctanoic acid (PFOA) in groundwater under acidic conditions. Int J Environ Res Public Health. 2016;13(6):602.
26. Hori H, Yamamoto A, Hayakawa E, Taniyasu S, Yamashita N, Kutsuna S, et al. Efficient decomposition of environmentally persistent perfluorocarboxylic acids by use of persulfate as a photochemical oxidant. Environ Sci Technol. 2005;39(7):2383–8.
27. Marchione AA, Diaz EL, Boyle JE. Kinetics of Aqueous persulfate-induced oxidative degradation of heptafluorobutanoate, pentafluoropropionate, and trifluoroacetate. J Fluorine Chem. 2021;252:109902.
28. Gauthier SA, Mabury SA. Aqueous photolysis of 8:2 fluorotelomer alcohol. Environ Toxicol Chem. 2005;24(8):1837–46.
29. Plumlee MH, McNeill K, Reinhard M. Indirect photolysis of perfluorochemicals: hydroxyl radical-initiated oxidation of N-Ethyl Perfluorooctane Sulfonamido Acetate (N-EtFOSAA) and Other Perfluoroalkanesulfonamides. Environ Sci Technol. 2009;43(10):3662–8.
30. Cioni L, Nikiforov V, Coêlho ACMF, Sandanger TM, Herzke D. Total oxidizable precursors assay for PFAS in human serum. Environ Int. 2022;170:107656.
31. Liu Z, Jin B, Rao D, Bentel MJ, Liu T, Gao J, et al. Oxidative transformation of nafion-related fluorinated ether sulfonates: comparison with legacy PFAS structures and opportunities of acidic persulfate digestion for PFAS Precursor Analysis. Environ Sci Technol. 2024;58(14):6415–24.
32. Robel AE, Marshall K, Dickinson M, Lunderberg D, Butt C, Peaslee G, et al. Closing the mass balance on fluorine on papers and textiles. Environ Sci Technol. 2017;51(16):9022–32.
33. Göckener B, Fliedner A, Rüdel H, Badry A, Koschorreck J. Long-Term Trends of Per- and Polyfluoroalkyl Substances (PFAS) in suspended particular matter from german rivers using the Direct Total Oxidizable Precursor (dTOP) Assay. Environ Sci Technol. 2022;56(1):208–17.
34. Sapozhnikova Y, Taylor RB, Bedi M, Ng C. Assessing per- and polyfluoroalkyl substances in globally sourced food packaging. Chemosphere. 2023;337:139381.
35. Jia Y, Shan C, Fu W, Wei S, Pan B. Occurrences and fates of per- and polyfluoralkyl substances in textile dyeing wastewater along full-scale treatment processes. Water Res. 2023;242: 120289.
36. Nolan A, Grimison C, Lavetz R, Slee D, Lim C, Centner M, et al., editors. Improving measurement reliability of the PFAS TOP assay. Proceedings of the 8th International Contaminated Site Remediation Conference; 2019; Australia.
37. Fan X, Bao Y, Mumtaz M, Huang J, Yu G. Determination of total oxidizable precursors in foam surfactants and foam contaminated water based on UV-activated persulfate oxidation. Sci Total Environ. 2021;763:142943.
38. Patch D, O'Connor N, Vereecken T, Murphy D, Munoz G, Ross I, et al. Advancing PFAS characterization: Enhancing the total oxidizable precursor assay with improved sample processing and UV activation. Sci Total Environ. 2024;909:168145.
39. Glover CM, Pazoki F, Munoz G, Sauvé S, Liu J. Applying the modified UV-activated TOP assay to complex matrices impacted by aqueous film-forming foams. Sci Total Environ. 2024;924:171292.
40. Kaiser A-M, Saracevic E, Schaar HP, Weiss S, Hornek-Gausterer R. Ozone as oxidizing agent for the total oxidizable precursor (TOP) assay and as a preceding step for activated carbon treatments concerning per- and polyfluoroalkyl substance removal. J Environ Manag. 2021;300:113692.
41. Patch D, O'Connor N, Ahmed E, Houtz E, Bentel M, Ross I, et al. Advancing PFAS characterization: Development and optimization of a UV-H2O2-TOP assay for improved PFCA chain length preservation and organic matter tolerance. Sci Total Environ. 2024;946: 174079.
42. Zweigle J, Bugsel B, Capitain C, Zwiener C. PhotoTOP: PFAS Precursor Characterization by UV/TiO2 Photocatalysis. Environ Sci Technol. 2022;56(22):15728–36.
43. Gockener B, Eichhorn M, Lammer R, Kotthoff M, Kowalczyk J, Numata J, et al. 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. J Agric Food Chem. 2020;68(45):12527–38.
44. McDonough CA, Guelfo JL, Higgins CP. Measuring total PFASs in water: The tradeoff between selectivity and inclusivity. Curr Opinion Environ Sci Health. 2019;7:13–8.
45. Fan X, Jiang Y, Guan X, Bao Y, Gu M, Mumtaz M, et al. Determination of total reducible organofluorine in PFAS-impacted aqueous samples based on hydrated electron defluorination. Sci Total Environ. 2022;829: 154548.
46. Houtz EF, Higgins CP, Field JA, Sedlak DL. Persistence of Perfluoroalkyl Acid Precursors in AFFF-Impacted Groundwater and Soil. Environ Sci Technol. 2013;47(15):8187–95.

47. Martin D, Munoz G, Mejia-Avendaño S, Duy SV, Yao Y, Volchek K, et al. Zwitterionic, cationic, and anionic perfluoroalkyl and polyfluoroalkyl substances integrated into total oxidizable precursor assay of contaminated groundwater. Talanta. 2019;195:533–42.
48. Weber AK, Barber LB, LeBlanc DR, Sunderland EM, Vecitis CD. Geochemical and hydrologic factors controlling subsurface transport of poly- and perfluoroalkyl substances, cape cod, massachusetts. Environ Sci Technol. 2017;51(8):4269–79.
49. Dauchy X, Boiteux V, Colin A, Hémard J, Bach C, Rosin C, et al. Deep seepage of per- and polyfluoroalkyl substances through the soil of a firefighter training site and subsequent groundwater contamination. Chemosphere. 2019;214:729–37.
50. Pelch KE, McKnight T, Reade A. 70 analyte PFAS test method highlights need for expanded testing of PFAS in drinking water. Sci Total Environ. 2023;876:162978.
51. Boiteux V, Dauchy X, Bach C, Colin A, Hemard J, Sagres V, et al. Concentrations and patterns of perfluoroalkyl and polyfluoroalkyl substances in a river and three drinking water treatment plants near and far from a major production source. Sci Total Environ. 2017;583:393–400.
52. Houtz EF, Sutton R, Park J-S, Sedlak M. Poly- and perfluoroalkyl substances in wastewater: Significance of unknown precursors, manufacturing shifts, and likely AFFF impacts. Water Res. 2016;95:142–9.
53. Kim J, Xin X, Mamo BT, Hawkins GL, Li K, Chen Y, et al. Occurrence and Fate of Ultrashort-Chain and Other Per- and Polyfluoroalkyl Substances (PFAS) in wastewater treatment plants. ACS ES&T Water. 2022;2(8):1380–90.
54. Schaefer CE, Hooper JL, Strom LE, Abusallout I, Dickenson ERV, Thompson KA, et al. Occurrence of quantifiable and semi-quantifiable poly- and perfluoroalkyl substances in united states wastewater treatment plants. Water Res. 2023;233: 119724.
55. Dauchy X, Boiteux V, Bach C, Colin A, Hemard J, Rosin C, et al. Mass flows and fate of per- and polyfluoroalkyl substances (PFASs) in the wastewater treatment plant of a fluorochemical manufacturing facility. Sci Total Environ. 2017;576:549–58.
56. Chen Y, Zhang H, Liu Y, Bowden JA, Tolaymat TM, Townsend TG, et al. Concentrations of perfluoroalkyl and polyfluoroalkyl substances before and after full-scale landfill leachate treatment. Waste Management. 2022;153:110–20.
57. Capozzi SL, Leang AL, Rodenburg LA, Chandramouli B, Delistraty DA, Carter CH. PFAS in municipal landfill leachate: Occurrence, transformation, and sources. Chemosphere. 2023;334: 138924.
58. Chen H, Zhang L, Li M, Yao Y, Zhao Z, Munoz G, et al. Per- and polyfluoroalkyl substances (PFASs) in precipitation from mainland China: Contributions of unknown precursors and short-chain (C2–C3) perfluoroalkyl carboxylic acids. Water Res. 2019;153:169–77.
59. Guckert M, Scheurer M, Schaffer M, Reemtsma T, Nödler K. Combining target analysis with sum parameters—a comprehensive approach to determine sediment contamination with PFAS and further fluorinated substances. Environ Sci Pollut Res. 2022;29(57):85802–14.
60. Tokranov AK, LeBlanc DR, Pickard HM, Ruyle BJ, Barber LB, Hull RB, et al. Surface-water/groundwater boundaries affect seasonal PFAS concentrations and PFAA precursor transformations. Environ Sci: Process Impacts. 2021;23(12):1893–905.
61. Macorps N, Labadie P, Lestremau F, Assoumani A, Budzinski H. Per- and polyfluoroalkyl substances (PFAS) in surface sediments: Occurrence, patterns, spatial distribution and contribution of unattributed precursors in French aquatic environments. Sci Total Environ. 2023;874: 162493.
62. Janda J, Nödler K, Scheurer M, Happel O, Nürenberg G, Zwiener C, et al. Closing the gap - inclusion of ultrashort-chain perfluoroalkyl carboxylic acids in the total oxidizable precursor (TOP) assay protocol2019.
63. Zhou J, Zhao G, Li M, Li J, Liang X, Yang X, et al. Three-dimensional spatial distribution of legacy and novel poly/perfluoroalkyl substances in the Tibetan Plateau soil: Implications for transport and sources. Environ Int. 2022;158:107007.
64. Wang B, Yao Y, Wang Y, Chen H, Sun H. Per- and polyfluoroalkyl substances in outdoor and indoor dust from Mainland China: contributions of unknown precursors and implications for human exposure. Environ Sci Technol. 2022;56(10):6036–45.
65. Juhasz AL, Keith A, Jones R, Kastury F. Impact of precursors and bioaccessibility on childhood PFAS exposure from house dust. Sci Total Environ. 2023;889:164306.
66. Choi YJ, Kim Lazcano R, Yousefi P, Trim H, Lee LS. Perfluoroalkyl Acid Characterization in U.S. Municipal Organic Solid Waste Composts. Environ Sci Technol Lett. 2019;6(6):372–7.
67. Sivaram AK, Panneerselvan L, Surapaneni A, Lee E, Kannan K, Megharaj M. Per- and polyfluoroalkyl substances (PFAS) in commercial composts, garden soils, and potting mixes of Australia. Environ Adv. 2022;7: 100174.
68. Chinthakindi S, Zhu H, Kannan K. An exploratory analysis of poly- and per-fluoroalkyl substances in pet food packaging from the United States. Environ Technol Innov. 2021;21:101247.
69. Ruyle BJ, Thackray CP, McCord JP, Strynar MJ, Mauge-Lewis KA, Fenton SE, et al. Reconstructing the composition of per- and polyfluoroalkyl substances in contemporary aqueous film-forming foams. Environ Sci Technol Lett. 2021;8(1):59–65.
70. Mumtaz M, Bao Y, Liu L, Huang J, Cagnetta G, Yu G. Per- and polyfluoroalkyl substances in representative fluorocarbon surfactants used in chinese film-forming foams: levels, profile shift, and environmental implications. Environ Sci Technol Lett. 2019;6(5):259–64.
71. Shojaei M, Joyce AS, Ferguson PL, Guelfo JL. Novel per- and polyfluoroalkyl substances in an active-use C6-based aqueous film forming foam. J Hazard Mater Lett. 2022;3:100061.
72. Dauchy X, Boiteux V, Bach C, Rosin C, Munoz J-F. Per- and polyfluoroalkyl substances in firefighting foam concentrates and water samples collected near sites impacted by the use of these foams. Chemosphere. 2017;183:53–61.
73. Lasee S, McDermett K, Kumar N, Guelfo J, Payton P, Yang Z, et al. Targeted analysis and Total Oxidizable Precursor assay of several insecticides for PFAS. J Hazard Mater Lett. 2022;3:100067.
74. Rodgers KM, Swartz CH, Occhialini J, Bassignani P, McCurdy M, Schaider LA. How Well Do Product Labels Indicate the Presence of PFAS in Consumer Items Used by Children and Adolescents? Environ Sci Technol. 2022;56(10):6294–304.
75. Wu Y, Miller GZ, Gearhart J, Peaslee G, Venier M. Side-chain fluorotelomer-based polymers in children car seats. Environ Pollut. 2021;268: 115477.
76. Zhu H, Kannan K. Total oxidizable precursor assay in the determination of perfluoroalkyl acids in textiles collected from the United States. Environ Pollut. 2020;265: 114940.
77. Schellenberger S, Liagkouridis I, Awad R, Khan S, Plassmann M, Peters G, et al. An Outdoor Aging Study to Investigate the Release of Per- And Polyfluoroalkyl Substances (PFAS) from Functional Textiles. Environ Sci Technol. 2022;56(6):3471–9.
78. Pickard HM, Ruyle BJ, Thackray CP, Chovancova A, Dassuncao C, Becanova J, et al. PFAS and Precursor bioaccumulation in freshwater recreational fish: implications for fish advisories. Environ Sci Technol. 2022;56(22):15573–83.
79. Guckert M, Rupp J, Nürenberg G, Nödler K, Koschorreck J, Berger U, et al. Differences in the internal PFAS patterns of herbivores, omnivores and carnivores - lessons learned from target

screening and the total oxidizable precursor assay. Sci Total Environ. 2023;875: 162361.

80. Liu S, Liu Z, Tan W, Johnson AC, Sweetman AJ, Sun X, et al. Transport and transformation of perfluoroalkyl acids, isomer profiles, novel alternatives and unknown precursors from factories to dinner plates in China: New insights into crop bioaccumulation prediction and risk assessment. Environ Int. 2023;172:107795.
81. Cioni L, Plassmann M, Benskin JP, Coêlho ACMF, Nøst TH, Rylander C, et al. Fluorine Mass Balance, including Total Fluorine, Extractable Organic Fluorine, Oxidizable Precursors, and Target Per- and Polyfluoroalkyl Substances, in Pooled Human Serum from the Tromsø Population in 1986, 2007, and 2015. Environ Sci Technol. 2023;57(40):14849–60.
82. Lämmer R, Weidemann E, Göckener B, Stahl T, Breuer J, Kowalczyk J, et al. Evaluation of the transformation and leaching behavior of two polyfluoroalkyl phosphate diesters in a field lysimeter study. J Agric Food Chem. 2022;70(45):14329–38.
83. Zweigle J, Bugsel B, Röhler K, Haluska AA, Zwiener C. PFAS-contaminated soil site in germany: nontarget screening before and after direct TOP assay by Kendrick Mass Defect and FindPFΔS. Environ Sci Technol. 2023;57(16):6647–55.
84. Röhler K, Susset B, Grathwohl P. Production of perfluoroalkyl acids (PFAAs) from precursors in contaminated agricultural soils: Batch and leaching experiments. Sci Total Environ. 2023;902:166555.
85. Göckener B, Fliedner A, Weinfurtner K, Rüdel H, Badry A, Koschorreck J. Tracking down unknown PFAS pollution – The direct TOP assay in spatial monitoring of surface waters in Germany. Sci Total Environ. 2023;898:165425.
86. Simon F, Gehrenkemper L, Becher S, Dierkes G, Langhammer N, Cossmer A, et al. Quantification and characterization of PFASs in suspended particulate matter (SPM) of German rivers using EOF, dTOPA, (non-)target HRMS. Sci Total Environ. 2023;885: 163753.
87. Zweigle J, Capitain C, Simon F, Roesch P, Bugsel B, Zwiener C. Non-extractable PFAS in functional textiles – characterization by complementary methods: oxidation, hydrolysis, and fluorine sum parameters. Environ Sci Process Impacts. 2023;25(8):1298–310.
88. Liagkouridis I, Awad R, Schellenberger S, Plassmann MM, Cousins IT, Benskin JP. Combined use of total fluorine and oxidative fingerprinting for quantitative determination of side-chain fluorinated polymers in textiles. Environ Sci Technol Lett. 2022;9(1):30–6.
89. Ruyle BJ, Pickard HM, LeBlanc DR, Tokranov AK, Thackray CP, Hu XC, et al. Isolating the AFFF signature in coastal watersheds using oxidizable PFAS precursors and unexplained organofluorine. Environ Sci Technol. 2021;55(6):3686–95.
90. Rodowa AE, Knappe DRU, Chiang SYD, Pohlmann D, Varley C, Bodour A, et al. Pilot scale removal of per- and polyfluoroalkyl substances and precursors from AFFF-impacted groundwater by granular activated carbon. Environ Sci Water Res Technol. 2020;6(4):1083–94.
91. Göckener B, Lange FT, Lesmeister L, Gökçe E, Dahme HU, Bandow N, et al. Digging deep—implementation, standardisation and interpretation of a total oxidisable precursor (TOP) assay within the regulatory context of per- and polyfluoroalkyl substances (PFASs) in soil. Environ Sci Eur. 2022;34(1):52.
92. Casson R, Chiang S-Y. Integrating total oxidizable precursor assay data to evaluate fate and transport of PFASs. Remed J. 2018;28(2):71–87.
93. Mejia Avendaño S, Liu J. Production of PFOS from aerobic soil biotransformation of two perfluoroalkyl sulfonamide derivatives. Chemosphere. 2015;119:1084–90.
94. Bryant JD, Anderson R, Bolyard SC, Bradburne JT, Brusseau ML, Carey G, et al. PFAS Experts Symposium 2: Key advances in poly- and perfluoroalkyl characterization, fate, and transport. Remed J. 2022;32(1–2):19–28.
95. Chiang D. What Total Oxidizable Precursor Assay Can and Cannot Tell Us about Precursors of Perfluoroalkyl Acids. Boston, USA: CDM Smith; 2019.
96. Tsou K, Antell E, Duan Y, Olivares CI, Yi S, Alvarez-Cohen L, et al. Improved Total Oxidizable Precursor Assay for Quantifying Polyfluorinated Compounds Amenable to Oxidative Conversion to Perfluoroalkyl Carboxylic Acids. ACS ES&T Water. 2023.
97. Fredriksson F, Kärrman A, Eriksson U, Yeung LWY. Analysis and characterization of novel fluorinated compounds used in surface treatments products. Chemosphere. 2022;302: 134720.
98. Xia C, Diamond ML, Peaslee GF, Peng H, Blum A, Wang Z, et al. Per- and Polyfluoroalkyl Substances in North American School Uniforms. Environ Sci Technol. 2022;56(19):13845–57.
99. Mumtaz M, Bao Y, Li W, Kong L, Huang J, Yu G. Screening of textile finishing agents available on the Chinese market: An important source of per- and polyfluoroalkyl substances to the environment. Frontiers of Environmental Science and Engineering. 2019;13(5).
100. Joerss H, Schramm T-R, Sun L, Guo C, Tang J, Ebinghaus R. Per- and polyfluoroalkyl substances in Chinese and German river water – Point source- and country-specific fingerprints including unknown precursors. Environ Pollut. 2020;267: 115567.
101. Schaefer CE, Hooper J, Modiri-Gharehveran M, Drennan DM, Beecher N, Lee L. Release of poly- and perfluoroalkyl substances from finished biosolids in soil mesocosms. Water Res. 2022;217: 118405.
102. Wang B, Yao Y, Chen H, Chang S, Tian Y, Sun H. Per- and polyfluoroalkyl substances and the contribution of unknown precursors and short-chain (C2–C3) perfluoroalkyl carboxylic acids at solid waste disposal facilities. Sci Total Environ. 2020;705: 135832.
103. Liu M, Munoz G, Vo Duy S, Sauvé S, Liu J. Per- and polyfluoroalkyl substances in contaminated soil and groundwater at airports: a canadian case study. Environ Sci Technol. 2022;56(2):885–95.
104. Hutchinson S, Rieck T, Wu X. Advanced PFAS precursor digestion methods for biosolids. Environ Chem. 2020;17(8):558–67.
105. Koch A, Aro R, Wang T, Yeung LWY. Towards a comprehensive analytical workflow for the chemical characterisation of organofluorine in consumer products and environmental samples. TrAC Trends Anal Chem. 2020;123: 115423.
106. Kim Lazcano R, Choi YJ, Mashtare ML, Lee LS. Characterizing and Comparing Per- and Polyfluoroalkyl Substances in Commercially Available Biosolid and Organic Non-Biosolid-Based Products. Environ Sci Technol. 2020;54(14):8640–8.
107. Pettersson M, Ericson Jogsten I, Yeung LWY. Ways to improve understanding of PFAS contamination - A case study within a waste management facility. Environ Pollut. 2024;356:124352.
108. Collins A, Krause MJ, Bessler SM, Brougham A, McKnight T, Strock T, et al. City-scale impacts of PFAS from normal and elevated temperature landfill leachates on wastewater treatment plant influent. J Hazard Mater. 2024;480:136270.
109. Neuwald IJ, Zahn D, Knepper TP. Are (fluorinated) ionic liquids relevant environmental contaminants? High-resolution mass spectrometric screening for per- and polyfluoroalkyl substances in environmental water samples led to the detection of a fluorinated ionic liquid. Anal Bioanal Chem. 2020;412(20):4881–92.
110. Wellmitz J, Bandow N, Koschorreck J. Long-term trend data for PFAS in soils from German ecosystems, including TOP assay. Sci Total Environ. 2023;893: 164586.
111. HEPA. PFAS National Environmental Management Plan Version 2.0. 2020;134.

112. Neuwald IJ, Hübner D, Wiegand HL, Valkov V, Borchers U, Nödler K, et al. Ultra-Short-Chain PFASs in the Sources of German Drinking Water: Prevalent, Overlooked, Difficult to Remove, and Unregulated. Environ Sci Technol. 2022.
113. Baqar M, Zhao M, Saleem R, Cheng Z, Fang B, Dong X, et al. Identification of Emerging Per- and Polyfluoroalkyl Substances (PFAS) in E-waste Recycling Practices and New Precursors for Trifluoroacetic Acid. Environ Sci Technol. 2024;58(36):16153–63.
114. Zhao M, Yao Y, Dong X, Baqar M, Fang B, Chen H, et al. Nontarget Identification of Novel Per- and Polyfluoroalkyl Substances (PFAS) in soils from an oil refinery in Southwestern China: A Combined Approach with TOP Assay. Environ Sci Technol. 2023;57(48):20194–205.
115. Shojaei M, Kumar N, Guelfo JL. An Integrated approach for determination of total per- and polyfluoroalkyl substances (PFAS). Environ Sci Technol. 2022;56(20):14517–27.
116. Coêlho MFAC, Cioni L, Van Dreunen W, Berg V, Rylander C, Urbarova I, et al. Legacy perfluoroalkyl acids and their oxidizable precursors in plasma samples of Norwegian women. Enviro Int. 2023;178:108026.
117. Glover CM, Quiñones O, Dickenson ERV. Removal of perfluoroalkyl and polyfluoroalkyl substances in potable reuse systems. Water Res. 2018;144:454–61.
118. Gonzalez D, Thompson K, Quiñones O, Dickenson E, Bott C. Assessment of PFAS fate, transport, and treatment inhibition associated with a simulated AFFF release within a wastewater treatment plant. Chemosphere. 2021;262: 127900.
119. Simonnet-Laprade C, Budzinski H, Maciejewski K, Le Menach K, Santos R, Alliot F, et al. 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. Environ Sci Process Impacts. 2019;21(11):1864–74.
120. Qiao B, Chen H, Song D, Yu H, Baqar M, Li X, et al. Multimedia distribution and release characteristics of emerging PFAS in wastewater treatment plants in Tianjin, China. J Hazard Mater. 2024;475:134879.
121. EURL-POPs. Guidance document on analytical parameters for the determination of per- and polyfluoroalkyl substances (PFAS) in food and feed. EURL for halogenated POPs in Feed and Food 2024.
122. EU. Technical guidelines regarding methods of analysis for monitoring of per- and polyfluoroalkyl substances (PFAS) in water intended for human consumption. European Commission; 2024. Contract No.: C/2024/4910.
123. Zhu H, Kannan K. A pilot study of per- and polyfluoroalkyl substances in automotive lubricant oils from the United States. Environ Technol Innov. 2020;19:100943.
124. Jiao E, Larsson P, Wang Q, Zhu Z, Yin D, Kärrman A, et al. Further Insight into Extractable (Organo)fluorine Mass Balance Analysis of Tap Water from Shanghai, China. Environ Sci Technol. 2023;57(38):14330–9.
125. Nickerson A, Maizel AC, Kulkarni PR, Adamson DT, Kornuc JJ, Higgins CP. Enhanced Extraction of AFFF-Associated PFASs from Source Zone Soils. Environ Sci Technol. 2020;54(8):4952–62.
126. Behnisch PA, Besselink H, Weber R, Willand W, Huang J, Brouwer A. Developing potency factors for thyroid hormone disruption by PFASs using TTR-TRβ CALUX® bioassay and assessment of PFASs mixtures in technical products. Environ Int. 2021;157:106791.
127. Martínez J, Picardo M, Peñalver A, Fabregas J, Aguilar C, Borrull F. Occurrence and health risk assessment of PFAS and possible precursors: a case study in a drinking water treatment plant and bottled water (south Catalonia, Spain). Environ Sci Pollut Res. 2024;31(45):56536–49.
128. Liu M, Glover CM, Munoz G, Duy SV, Sauvé S, Liu J. Hunting the missing fluorine in aqueous film-forming foams containing per- and polyfluoroalkyl substances. J Hazard Mater. 2024;464:133006.
129. Müller V, Kindness A, Feldmann J. Fluorine mass balance analysis of PFAS in communal waters at a wastewater plant from Austria. Water Res. 2023;244:120501.
130. Katz DR, Sullivan JC, Rosa K, Gardiner CL, Robuck AR, Lohmann R, et al. Transport and fate of aqueous film forming foam in an urban estuary. Environ Pollut. 2022;300:118963.