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COMMENT

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

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Abstract

Over the past decades, thousands of different per- and polyfluoroalkyl substances (PFASs) have been produced and applied in various industrial processes and consumer products. Their structural diversity has reached a level that cannot be covered by classical target screening methods for individual compounds. Large-scale contaminations of soil, however, require the need to adapt new analytical methods that can describe a contamination more comprehensively. While sum parameters such as the total oxidisable precursor (TOP) assay have been developed in the past years, they are not yet applied in the regulatory context of PFASs.

In this commentary, we provide an overview on different approaches of the TOP assay as well as its benefits and disadvantages to other sum parameters for PFASs in soil samples. Furthermore, we elaborate its opportunities and its challenges that need to be tackled to implement the TOP assay as a regulatory tool. With several different approaches of the TOP assay being available, a sound and standardised method needs to be agreed upon and more research is necessary to better describe the method. Although the complexity of PFAS contaminations in soil cannot be fully covered by any analytical method alone, the TOP assay can provide valuable data to detect and characterise soil contamination as an inventory for subsequent remediation measures. Therefore, the TOP assay should be implemented as a useful tool both in research and in the regulatory context of PFASs.

Keywords: PFAS, Precursors, TOP assay, Regulation, Sum parameters, Soil contamination

Background

The group of per- and polyfluoroalkyl substances (PFASs) comprises a diverse group of anthropogenic chemicals that have been produced since the 1940s. Due to their unique properties, such as their high stability and water-, oil-, and grease-repellency, they are used in various industrial processes and consumer products [1]. These properties and their widespread use, however, also lead to adverse effects on human health and the environment that have been described for several substances from the group of PFASs [2, 3]. Some of the best-studied PFASs are perfluoroalkyl acids (PFAAs). Perfluorocarboxylic acids (PFCAs) and perfluorosulfonic acids (PFSAs) are the most prominent PFAAs in the environment. They are known to be

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environmentally persistent and are, therefore, referred to as 'forever chemicals' [4, 5]. Many other PFASs, however, are known to degrade to the group of PFAAs in the environment as well as in vivo and are termed precursors [6–8]. In 2018, the Organisation for Economic Co-operation and Development (OECD) identified 4730 different CAS entries belonging to the group of PFASs [9]. In recent years, industry shifted from the perfluoroalkylsulfonyl- and carbonylfluoride-based chemistry to an increased use of fluorotelomer-based substances and poly- and perfluorinated ethers [10–12]. Technical products, such as those applied in aqueous film-forming foams (AFFFs) or products for paper and textiles coating often contain PFAS mixtures. In addition, polymers with fluorinated side-chains are among the active ingredients, e.g., in products for grease-proofing of paper and board [1]. In contrast, most regulatory efforts and restrictions on national and international levels, such as the Stockholm Convention focus on a subset of a few PFASs, namely, PFAAs and in some cases their precursors [13–17]. The revised EU Drinking Water Directive entering into force in 2023, however, includes a 'PFAS Total' sum parametric value besides the sum of 20 individual PFAAs. A full ban of PFASs as a group has so far only been released in Denmark for the specific application in food packaging materials [18], but the European Commission intends to phase out the use of PFASs as a group in the European Union unless a specific use is proven essential for society [19].

Worldwide findings of individual PFASs in various environmental systems [20–23] and humans [24–27] already demonstrate the ubiquitous distribution of these substances. With the high number of different chemical structures within the group of PFASs comes the need to analyse them in a comprehensive manner. Yet, common targeted analytical methods may often underestimate the total PFAS burden, because they only include a few individual to a few dozen compounds [28, 29]. By only analysing the legacy PFASs, only a small part of the contamination may be visible and the outreach of the contamination may be underestimated to a great extent. For example, soil contaminations with unknown precursors would only be detected indirectly and with a temporal delay after they have formed PFAAs during their degradation processes. The underestimation may further lead to the inaccurate decision that no measures have to be applied which in turn may lead to a long-term contamination of underlying groundwater. Thus, to classify PFAS contaminated sites, an approximation for the extent of the PFAS burden is essential to decide on the measures to be taken. A good example is the PFAS contamination of an area in southwest Germany. Only the analysis of specific precursor substances could describe the extent of the contamination more comprehensively and the source of contamination could be narrowed down [30–33].

Given the relevance of precursors, sum parameters such as the total oxidisable precursor (TOP) assay [34] are of increasing importance to uncover the total PFAS burden. In the TOP assay, PFAA precursors are chemically oxidised to yield PFCAs of related chain lengths. Under certain conditions, it has also been described that sulfonamide-based precursors may also be transformed into PFSAs to some extent [35]. Thus, the degradation—which may occur in the course of time—can be simulated in the laboratory. The difference between the amount of PFCAs before (i.e., via target analysis) and after the TOP assay then provides information on the amount of precursors in a sample. Notably, the oxidative processes in the TOP assay do not fully correspond to those in the environmental degradation of precursors and the TOP assay does not cover all types of PFASs. Nevertheless, the method can be considered an important tool to illustrate precursors in a sample and to get a general idea of the total PFAS burden, e.g., when assessing a contamination in soil and groundwater [34, 36].

This commentary focusses on contaminations in soil as they are often of large scale and, therefore, of high relevance for the surrounding environment and humans. In addition, most soil contaminations represent initial contaminations, often characterised by a wide range of precursor substances rather than PFAAs, and, therefore, can be described by target methods only to a very limited extent.

In the subsequent remediation process, the TOP assay may also be used to assess the success of remediation measures [37]. For policy makers, the TOP assay may also allow monitoring of trends of unknown or unidentified PFASs, and thus observing shifts in production, e.g., from legacy to emerging PFASs. In addition, restrictions of precursors can be more extensively controlled by means of the TOP assay. Furthermore, the TOP assay can provide clues to the sources of a contamination, e.g., by analysing the patterns of formed oxidations products [38–40].

This commentary gives an overview of the opportunities, current varieties, challenges, and limitations of the TOP assay as well as its future needs and perspectives for the assessment of PFAS contamination in soils. In addition, a comparison with other sum parameters and other emerging analytical methods to overcome the complexity of PFASs is presented. Finally, this commentary aims to provide consultation on how the method can be applied into a regulatory context of PFASs.

Comparison of TOP assay methods to other sum parameters for organofluorine compounds in soils
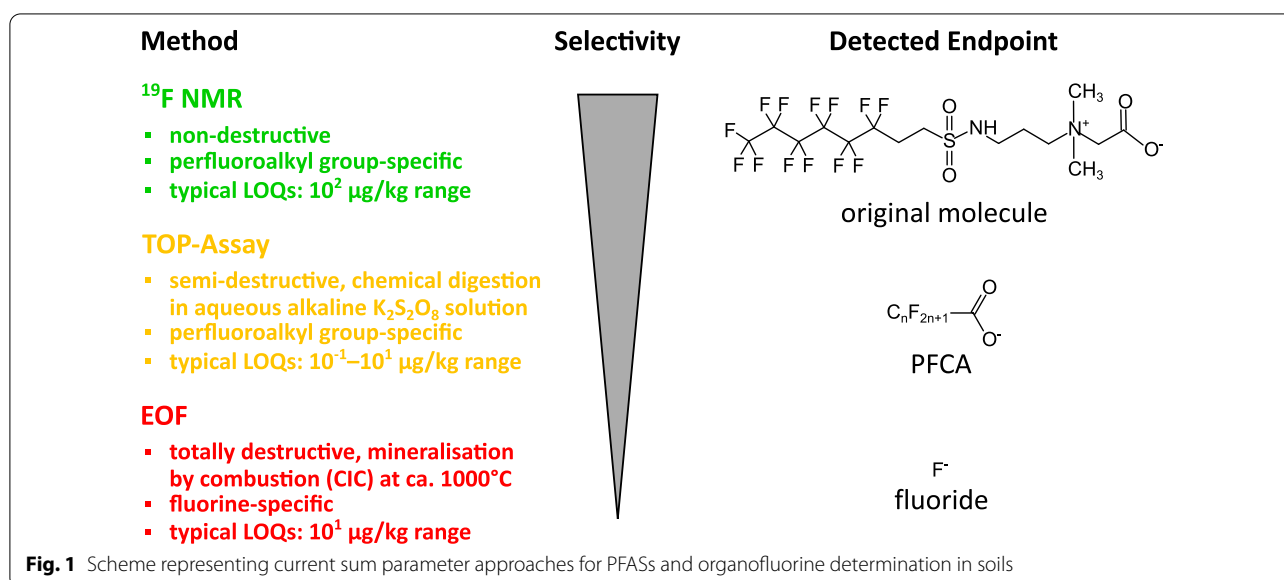
Several sum parameters for PFASs or organofluorine compounds have been described in literature. They can be divided into non-destructive, semi-destructive, and totally destructive approaches (Fig. 1). Aside from adsorbable organic fluorine (AOF) [41–45] for soil leachates, only extractable organic fluorine (EOF) [46, 47], TOP assay [42, 48, 49], and ^{19}F nuclear magnetic resonance (NMR) (BWPLUS project FluorTECH, unpublished results, 2021) have been applied quantitatively for soil samples or sediments and thus are considered here. The highest selectivity is linked to the non-destructive ^{19}F NMR analysis giving spectral information on the chemical structure of the organofluorine chemicals, including non-ionic substances. Specific signals of CF$_2$- and CF$_3$-groups can be used for identification of PFASs and for the quantitative PFAS-related organofluorine determination by standard addition. No extraction or other discriminating sample preparation steps are needed. Therefore, ^{19}F NMR can be considered a total PFAS method. However, its sensitivity for direct analysis is poor, and thus, only highly contaminated environmental samples (ppm levels) can be analysed by this technique.

EOF measurements by solvent extraction from soils followed by combustion ion chromatography (CIC) are more sensitive (ppb levels) than ^{19}F NMR, but show the poorest selectivity among the three described methods. Due to extract combustion the complete structural information is lost and organofluorine originating from other compounds than PFASs (e.g., even active ingredients of fluorinated pesticides or pharmaceuticals and their metabolites without CF$_2$ or CF$_3$ groups, e.g.,

fluoroquinolone antibiotics) are also contributing to this parameter [42]. This drawback of EOF is simultaneously its strength, because it covers all extractable PFASs and also those that are not amenable to TOP assay methods (see below). EOF measurements afford a CIC instrument, which is less often part of the standard equipment in environmental laboratories, while, e.g., LC–MS/MS is usually available in all laboratories analysing PFASs. Regarding the sensitivity, EOF methods typically show limits of quantification (LOQs) in the one- or two-digit ppb range [30, 50], while TOP assay methods show LOQs in the low ppb range or even lower [29, 48, 49], thus representing the most sensitive approach among sum parameters for organofluorine compounds in soil.

The TOP assay approaches possess an interim selectivity between ^{19}F NMR and EOF, because some structural information is conserved in form of PFCAs as products of a partial destruction by oxidative conversion in aqueous solution. However, most target analyses according to existing TOP assay protocols discriminate per- and polyfluoroalkylethers, because they are either not oxidised (perfluoroalkylethers) or oxidised to usually not measured products [51]. Since most TOP assay protocols for soils [48, 49] use oxidation of a methanolic soil extract, only extractable precursors are assessed. The use of other solvents may increase the extent of precursors accessible by the TOP assay. A direct oxidation by means of the so-called direct TOP assay (dTOP) [29, 52] avoids a discrimination by direct oxidation of a small aliquot of a soil sample with a high excess of a highly concentrated oxidation solution.

With a given TOP protocol, molar conversion factors for known precursors can be evaluated [34, 53, 54]. Based

Method

^{19}F NMR
- **non-destructive**
- **perfluoroalkyl group-specific**
- **typical LOQs: 10^2 µg/kg range**

TOP-Assay
- **semi-destructive, chemical digestion in aqueous alkaline K$_2$S$_2$O$_8$ solution**
- **perfluoroalkyl group-specific**
- **typical LOQs: 10^{-1}–10^1 µg/kg range**

EOF
- **totally destructive, mineralisation by combustion (CIC) at ca. 1000°C**
- **fluorine-specific**
- **typical LOQs: 10^1 µg/kg range**

Selectivity

Detected Endpoint

original molecule

C_nF_{2n+1}

PFCA

F$^-$
fluoride

Fig. 1 Scheme representing current sum parameter approaches for PFASs and organofluorine determination in soils

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on these factors the contribution of known precursors can be calculated and compared to the measured PFCA concentration to give the contribution of unknown precursors. The magnitude of unknown precursors can be estimated by comparing the PFCA amounts before and after the TOP assay while taking those PFCAs into account that have been formed from the known precursors in the sample. Other approaches such as by Robel et al. [54] estimate the amount of unknown precursors based on the amount of fluorine before and after the TOP assay. By means of the known PFCA homologue patterns after oxidation, some hints on the nature of abundant precursor classes in a sample can be deduced, e.g., perfluoroalkylsulfonamide-based vs. fluorotelomer-based precursors. Depending on the individual conditions of different TOP assay methods and especially for dTOP, the chain length reduction might shift the products to ultrashort-chain PFCAs or even to mineralization, and thus outside the analytical window of PFCA detection. However, the chain length pattern of PFCAs obtained in the TOP assay remains a valuable information that is not provided by ^{19}F NMR and EOF analyses.

Data interpretation

Drawing conclusions from the TOP assay requires a suitable evaluation and interpretation of the data in both quantitative and qualitative terms. In most cases, the TOP assay data has to be accompanied by a target analysis of the same sample, often referred to as data "before TOP assay". The difference between the levels of each analyte before and after the TOP assay gives a first indication on the presence of precursor substances in a sample. However, due to the partially destructive character of the TOP assay and its modifications, the mass balance before and after the TOP assay needs to be performed on a molar basis, especially if the balance is close to 100%. In this way, the loss of non- or polyfluorinated moieties and the partial loss of fluorine due to chain-length shortening of PFCAs are taken into account. In case the target analysis shows the presence of known precursors, their oxidative pathways must be either known (i.e., by conversion experiments) or estimated (i.e., with data from similar substances) to assess the balance of known and unknown precursors. To render the quantitative balancing as comprehensive as possible, the analyte spectrum should be as wide as possible to also assess precursors of ultrashort-chain PFCAs [49]. In addition, it needs to be considered that some groups of PFASs cannot be quantitatively covered by current TOP assay approaches as they are not oxidised at all or they form oxidation products that are not covered by typical targeted methods for PFAS analysis. This is the case, e.g., for volatile substances dissipating during the heating process or during evaporation

steps, precursors that form non-detectable or unknown oxidation products and (depending on the applied type of TOP assay) substances that cannot be extracted from samples with the chosen solvent or extraction method.

The TOP assay itself does not yield direct information on the identity of precursors present in a sample. If the formation of oxidation products cannot be explained by formation from known precursors covered in the target analysis, the chain length pattern in the oxidised samples can nevertheless yield valuable information on a PFAS burden. With a certain variation, many precursors are typically transformed to PFCAs of related chain lengths depending, e.g., on the fluorination status of the alkyl chain. Thus, the chain lengths of oxidation products in the TOP assay give a hint on the bioaccumulation and immobilization potential of unknown precursors in a sample. Short-chain oxidation products can be a hint for the increased mobility of precursors and thus their potential to contaminate ground and drinking water. This qualitative interpretation, however, strongly depends on the type of precursors in a sample and must, therefore, be treated with caution: while in the TOP assay, perfluoroalkylsulfonamide-based precursors typically yield PFCAs of the same perfluoroalkyl chain lengths as the precursors [34], they are transformed to PFSAs under environmental conditions [55]. At high concentrations of oxidation agents, an alkaline hydrolysis of sulfonamides leading to the partial formation of PFSAs in the TOP assay has also been described [35]. On the other hand, the polyfluorinated fluorotelomer precursors form a wider set of PFCAs with chain-lengths $n+1$, n, $n-1$, $n-2$, etc. (n being the number of perfluorinated carbons in the alkyl chain), both in the TOP assay [34] as well as in the environment [56, 57], thus biasing the interpretation of the chain length pattern. In addition, data from the TOP assay does not necessarily represent the total exposure potential as not all precursors in the TOP assay may be transformed to PFAAs in a timely manner. Therefore, the TOP assay can be considered a worst-case scenario for risk assessment.

Diversity of the TOP assay

Since the TOP assay has been first described in literature in 2012 by Houtz and Sedlak [34], several research groups and commercial labs have implemented and modified this method. The modifications include various methodological parameters aiming to improve the informative value of the TOP assay as well as to address matrix specific challenges.

One of the crucial decisions during the TOP assay analysis is the timing of adding isotopically labelled internal standards. While in some TOP assay approaches, internal standards are added to the sample after oxidation [48, 49]

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other laboratories add internal standards before oxidation [52]. Pre-oxidation addition is only possible for stable PFAA internal standards and compensates for analyte losses due to surface sorption, distribution to the air–water interface or an erroneous degradation of analytes, e.g., due to too low pH values (pH < 12 [34]) during the oxidation. In case of soil samples, the addition of internal standards should take place even before the extraction step. To reduce the risks of extraction losses or incomplete oxidation, modified oxidation approaches include a pre-oxidation with hydrogen peroxide (so far only applied to biosolids) [58] or the dTOP assay. In the dTOP process, only a small amount of soil sample is directly oxidised with high amounts of oxidation reagent, since high amounts of organic matrix components consume significant amounts of oxidation reagent. A disadvantage of the dTOP assay towards other TOP assay approaches is the reduced sensitivity (due to the small amount of sample used) as well as a more destructive character with reduced molar yields [29, 52].

While Houtz et al. [34] only included the analysis of PFCAs with chain lengths of C4 to C14, the spectrum of oxidation products has been expanded in other laboratories. For example, Janda et al. [49] included ultrashort-chain PFCAs (C2 and C3) and PFSAs into the TOP assay analysis of soil samples and demonstrated the quantitative importance of ultrashort-chain PFCAs to the overall PFAS burden. Zhang et al. [51] investigated the fate of per- and polyfluoroalkyl ether acids (PFEAs) in the TOP assay and found that some of these emerging substances are degraded to shorter-chain PFEAs, such as

perfluoromethoxypropionic acid (PFMOPrA) as an oxidation product of 4,8-dioxa-3H-perfluorononanoate (DONA). With the complexity within the group of PFASs and potential new PFASs to be launched in the future, the spectrum of oxidation products to be analysed also plays an important role for the comprehensiveness of the TOP assay.

Standardisation and quality control

The performance of the TOP assay strongly depends on operational conditions and the matrices to be analysed. A standard protocol is urgently needed on international (ISO) or European level (CEN) to ensure comparability of results of different laboratories. To our knowledge, besides a standardization project of the German Institute for Standardisation (DIN) regarding aqueous soil leachates, so far there are no activities on this topic concerning soils. Round robin tests, interlaboratory comparison studies and systematic robustness studies as basis for a standard are equally scarce and often focus on aqueous matrices [59]. Figure 2 shows an example of a comparison between the results from five laboratories (L1–L5) for a contaminated soil sample before and after TOP assay [BWPLUS project FluorTECH, unpublished results, 2021].

Before TOP assay, the mean concentrations ($n = 3$) of the twelve PFAAs measured by the different laboratories are well comparable, suggesting sufficient similarity among the applied analysis methods. Only after the TOP assay, significant differences in mean concentrations and variances occur due to inconsistent procedures
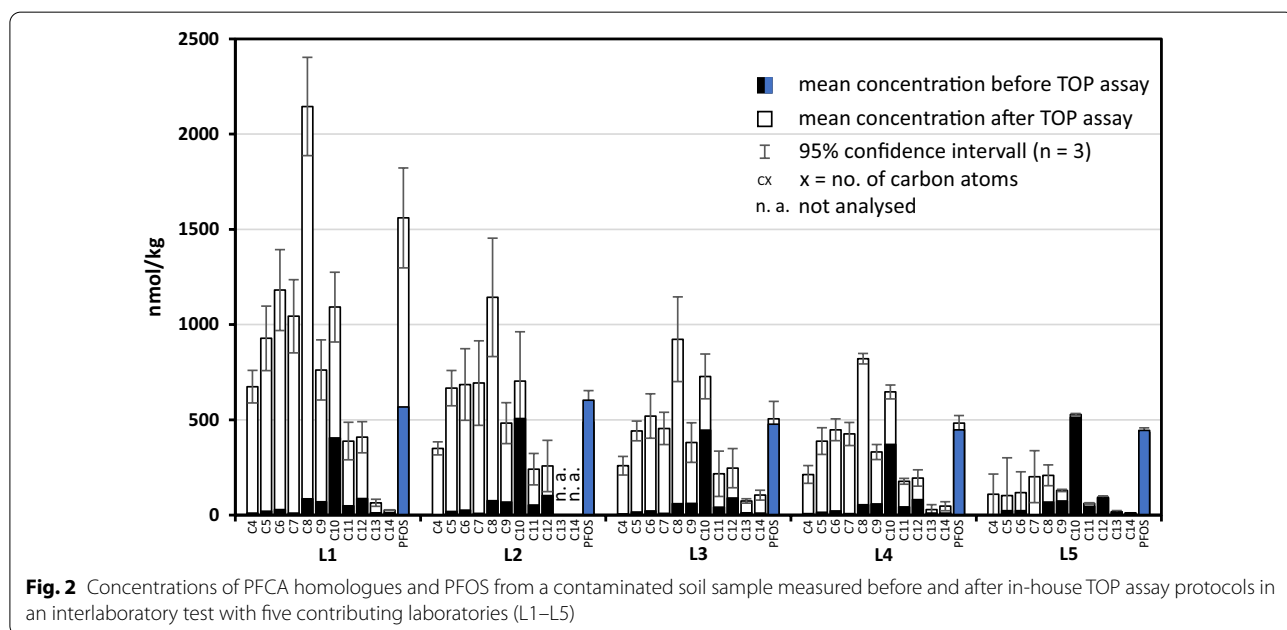
Fig. 2 Concentrations of PFCA homologues and PFOS from a contaminated soil sample measured before and after in-house TOP assay protocols in an interlaboratory test with five contributing laboratories (L1–L5)

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among the laboratories (L). For example, although L2–L4 produced similar values, confidence intervals from the triplicates were notably smaller with L4 compared to L2 and L3. Moreover, L5 shows generally lower concentrations than all the other laboratories and L1 generally higher ones. The most pronounced procedural difference between L1 and the other laboratories is that L1 applied the dTOP assay, which also resulted in a significant increase of the PFOS concentration. The results of this interlaboratory comparison emphasise the need for further standardisation of different TOP assay approaches.

The standard procedure for soil should define the pretreatment of a sample (drying, sieving, extraction versus direct oxidation), the parameters of oxidation as discussed above, such as pH, reaction time, and number of oxidation cycles, the analysis of individual compounds, blank values, LOQ, recovery and use of isotopically labelled compounds. Using isotopically labelled standards during the oxidation step [60], comparing original vs. diluted samples [61], and checking the absence of precursors after oxidation [62] are multiple ways of defining criteria for the oxidation performance. It is known that high levels of organic matter in biosolid samples interfere with the oxidation process and lead to an incomplete transformation of precursors [58] thus highlighting the need for quality control of the oxidation process. Isotopically labelled standards or selected precursors used for quality control of the oxidation process have to be carefully selected and urgently need to be standardised. Larsson [60], e.g., used $^{13}C_8$-FOSA, which is transformed to $^{13}C_8$-PFOA and does not interfere with $^{13}C_4$-PFOA, as internal standard for quantification. Before using oxidation standards, the degradation products in the TOP assay should be identified and potential overlaps should be excluded. The very limited availability of such internal precursor standards, however, currently poses a major challenge for this approach and should be addressed by producers of analytical PFAS standards. In this context, it also needs to be considered, that organic solvent used for spiking the internal standard might also interfere with the oxidation process, if it is not removed before. Plausibility checks should include that the sum of PFASs after oxidation is higher than or equal to the sum before. For interpretation and use of results, it is necessary to define which measurement uncertainty is acceptable and fit for purpose, e.g., for evaluating the human risk, toxicologically derived limit values are compared to measured values.

Regulatory context

In Germany, there are no binding limit values for PFASs in soil so far. However, in 2023, the amended Federal Soil Protection Ordinance will come into force regulating

seven PFASs in soil. These are the substances for which sufficient data were available for derivation of human health risks. It is obvious that those seven substances do not represent the whole spectrum of PFASs in soil, as PFAS contamination in soil originates from various sources containing a variety of PFASs, e.g., fire-fighting trainings, soil amendment with sewage sludge, or airborne deposition. If soil has been contaminated with PFASs, authorities have to decide whether measures such as excavation of soil or remediation have to be undertaken. Moreover, in case of agricultural areas, it is important to decide whether soils are still usable for food production. Thus, it is important to be aware of the dimension of the contamination. Due of the variety of PFASs, this is difficult and challenging, because as described earlier only a limited number of PFASs can be covered with standard-analytics. The TOP assay is a versatile tool to get a more extensive view on an PFAS contamination, since its ability to show the presence of many precursors that are not yet covered by targeted analysis.

Thus, the top assay can indicate the PFAS reservoir in the soil and serve as a rough estimate of the time course with regard to the leaching behaviour. The presence of large precursor quantities can lead to a long-lasting supply of PFAAs through degradation processes, which can contaminate the groundwater in the long term. The TOP assay can also be used as an indicator for the success of regulations, because the sum of PFAS pollution in the environment should decrease when PFAS use decreases. Due to the low transformation rates and the low mobility of some PFAS, this must be considered as a long-term aspect.

Another aspect is, that precursors can reach soil via air deposition. This has led to detectable background contamination [63, 64] and can be of concern in the vicinity of fluorochemical plants [65, 66]. The TOP assay can be a powerful tool to visualise the sum of airborne PFAS-emissions to soil, e.g., originating from industrial facilities.

In addition, the TOP assay indicates how well the target analysis describes the contamination. If the TOP assay indicates a high precursor load, a search for contamination sources should be carried out. This can provide valuable information on the actual substance spectrum, because different PFASs are used, e.g., in fire-fighting agents than, for example, in paper finishing [67]. Suspect screening or nontarget methods could then lead to further information on the precursors present in the soil and facilitate the handling of the contaminated soil. The search for the polluter could also be facilitated with this information. Thus, the extent of precursor substances may be helpful as a first screening and may be further used for environmental forensics to identify the origin

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of a contamination. However, the results received by the TOP assay have to be treated with caution. Because of the harsh oxidising procedure in the laboratory, different degradation processes may be possible in contrast to the conditions in the environment. Thus, a direct mapping of parent compounds (precursors) is not possible and a direct context to toxicological derivations is highly uncertain. Vice versa, the TOP assay cannot predict future groundwater contamination in a substance-specific manner.

The degree of interpretation of TOP assay data is highly dependent on whether the data are used for research or regulatory purposes. In research, data from the TOP assay can yield valuable information about the contamination in a sample and suggest further analyses to better describe the contamination. For regulatory purposes, however, interpretation of the data must be considered with more caution as its implications might have significant impacts, e.g., on the usability of the analysed soil or the subsequent actions by authorities. If the TOP assay is implemented into regulatory processes, it is, therefore, highly important to scientifically agree upon the possible interpretation of TOP assay data and the regulatory measures that can and should be derived thereof.

Despite the limits of the TOP assay for the analysis of soil samples, it is a helpful tool to get an overview of potential risks of the PFAS reservoir in soils groundwater pollution and pollution of food and feed with PFASs as a first step.

Conclusions

In the past years, a number of studies demonstrated that sum parameters are important tools to analytically keep up with the increasing complexity within the group of PFASs. In this commentary, we elaborated the benefits and drawbacks of the TOP assay for soil samples in comparison to other sum parameters, described the challenges and future needs as well as the opportunities of the TOP assay in a regulatory context. Although currently there is a set of different approaches compared to the original protocol, referred to as the TOP assay, there is no consistent method agreed upon yet. With the advantages and opportunities of the methods described above, there is a need (a) to internationally agree upon various parameters to yield a standardised and sound method, (b) to apply this standardised TOP assay in a regulatory context and (c) to conduct further research to better understand and optimise the TOP assay.

There is no currently available analytical method that can selectively cover the entirety of PFASs alone at a sufficiently sensitive level and admittedly the true 'PFAS Total' burden can never be completely assessed. However, with a set of powerful analytical tools, including target analysis, chemical and spectrometric sum parameters, and non-target screening approaches, the complexity of PFASs can be described in an increasingly comprehensive mosaic manner by taking advantage of each applied tool. The TOP assay can and should play an important role in this endeavour.

Abbreviations

AOF: Adsorbable organic fluorine; CEN: European Committee for Standardisation; CIC: Combustion ion chromatography; DIN: German Institute for Standardisation (Deutsches Institut für Normung); DONA: 4,8-Dioxa-3*H*-perfluorononanoate; dTOP: Direct total oxidisable precursor assay; EOF: Extractable organic fluorine; EU: European Union; ISO: International Organisation for Standardisation; LC–MS/MS: Liquid chromatography coupled to tandem mass spectrometry; LOQ: Limit of quantification; NMR: Nuclear magnetic resonance; OECD: Organisation for Economic Co-operation and Development; PFAAs: Perfluoroalkyl acids; PFASs: Per- and polyfluoroalkyl substances; PFCAs: Perfluorocarboxylic acids; PFEAs: Per- and polyfluoroalkyl ether acids; PFMOPrA: Perfluoromethoxypropionic acid; PFOA: Perfluorooctanoic acid; PFOS: Perfluorooctane sulfonic acid; PFSAs: Perfluorosulfonic acids; TOP: Total oxidisable precursor assay.

Availability of data and materials
Data sharing is not applicable to this article as no data sets were generated or analysed during the current study. The detailed data from the interlaboratory comparison study presented here will be made publically available in the future.

Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
Not applicable.

Competing interests
The authors declare that they have no competing interests.

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