



From Sludge to Soil

Exploring the Presence, Stability and Mobility of
PFAS and PFAS Precursors in Swedish Sewage
Sludge

Stina Åström

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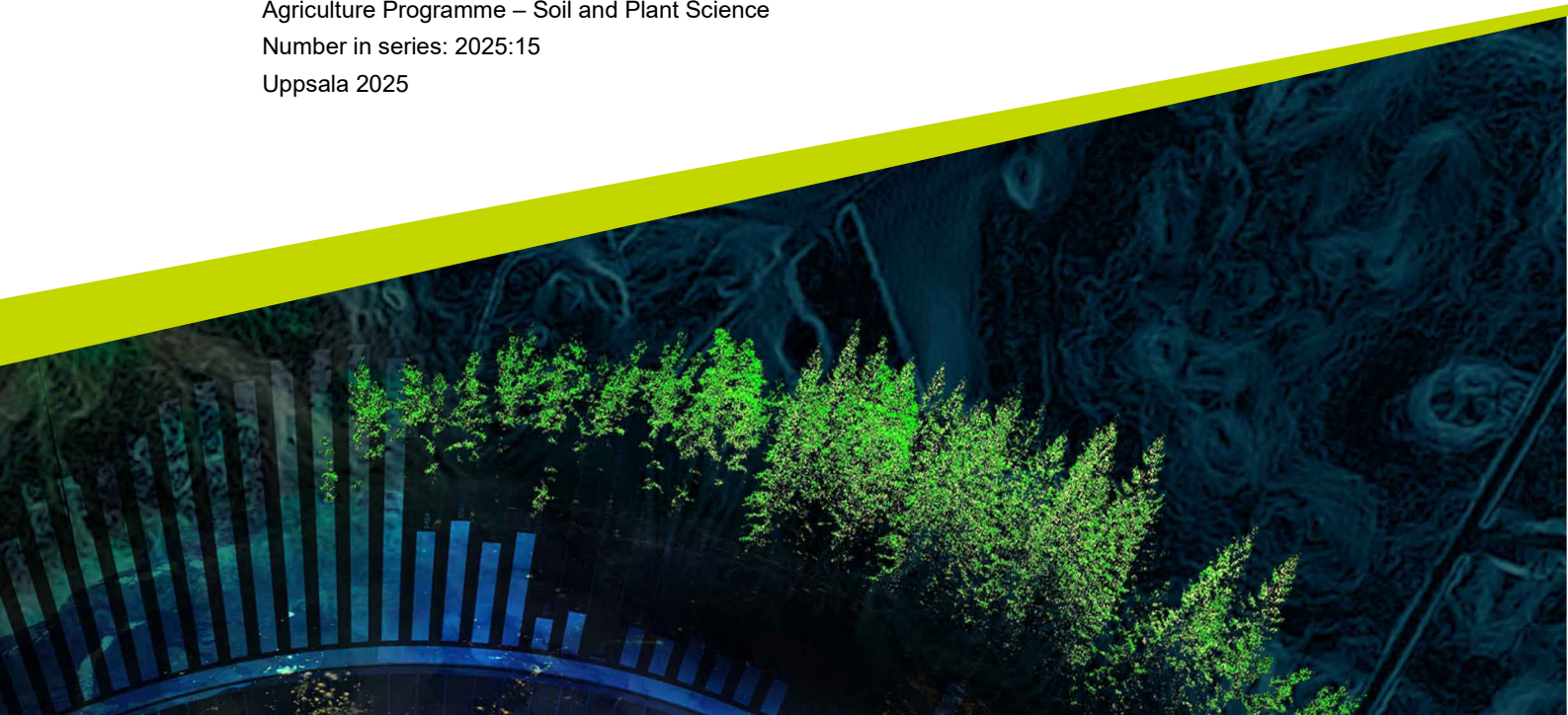
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From Sludge to Soil. Exploring the Presence, Stability and Mobility of PFAS and PFAS Precursors in Swedish Sewage Sludge

Från slam till jord. En undersökning av förekomst, stabilitet och mobilitet av PFAS och dess prekursorer i svenskt avloppsslam

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Abstract

Per- and polyfluoroalkyl substances (PFAS) are subjects of increased regulatory measures, however, current methods and limits focus mainly on a few legacy compounds, such as PFOS and PFOA. Novel PFAS substances that can transform into legacy PFAS, i.e. precursors, are largely excluded from both analysis and regulation, despite their potential risks. In Sweden, 85% of sewage sludge is applied to soil, mainly on farmland. New REVAQ guidelines set limits for 4 and 22 specific PFAS compounds respectively, but do not account for precursors despite several studies indicating that they represent a dominant portion of the PFAS content. This study aimed to assess the PFAS content in digested and undigested sewage sludge from five wastewater treatment plants using both target analysis (49 compounds) and the Total Oxidizable Precursor (TOP) assay. Leachability was evaluated using a leaching test, and short-term degradation of precursors in soil was monitored over four weeks. Results showed that the TOP assay revealed significantly higher PFAS concentrations than target analysis alone, particularly in undigested sludge, indicating a high content of precursors. The dominant PFASs included PFOS and PFHpA, using target analysis and TOP Assay, respectively. No degradation of precursors was observed over the incubation period. The findings suggest that precursors make up a substantial portion of total PFAS in sludge and that current target-based analyses underestimate true PFAS levels. The TOP assay is recommended for inclusion in standard PFAS monitoring protocols for sewage sludge.

Keywords: PFAS, sewage sludge, Swedish sewage sludge, PFAS precursors, target analysis, TOP Assay

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1. Introduction

Per- and polyfluoroalkyl substances, known as PFAS, are synthetic substances used in a wide range of products and processes. However, risks associated to health and environment have become more evident for several of these compounds and as a result, certain regulatory thresholds and guideline values have been introduced. Despite this, knowledge and regulation of this large and diverse class of substances is still sparse.

In Sweden, both PFAS regulations and analyses in various sectors are typically based on a limited number of PFAS compounds (European Union, 2023; Livsmedelsverket, 2022). As for 2025, PFAS guidelines are introduced to Swedish sewage sludge, produced by wastewater treatment plants (WWTPs) certified with REVAQ-certificates (Svenskt Vatten, 2025). The certification guidelines use two commercially common analytical packages: PFAS4 and PFAS22, that target only 4 and 22 specific substances respectively. Precursors of PFAS are PFAS compounds that can undergo partial degradation under certain environmental conditions, with the terminal product often being a highly persistent PFAS, such as PFOS. Despite this, it remains uncommon to include newer PFASs, such as PFAS precursors, in routine PFAS investigations (KEMI, 2024).

The limited analytical scope of Swedish sewage sludge is particularly relevant considering its predominant management method. Approximately 85% of Swedish sewage sludge production is repurposed as a soil additive, primarily for agricultural use and land restoration purposes (SCB, 2022). As such, PFASs not included in these investigations may be introduced to our environment through soil applications. Previous studies on sewage sludge have shown that PFAS precursors may account for as much as 75% of the total identified PFAS content (Kärrman et al., 2019). Knowledge about PFAS in sewage sludge, particularly regarding precursors, remains limited. Further research is needed to ensure safe handling and recycling of sewage sludge.

Aim and objectives

The aim of this study is to improve our understanding of PFAS and PFAS precursors in Swedish sewage sludge, and to highlight the limitations of the commonly used PFAS analysis method of today. In this project, a total of 49 PFAS compounds were analysed in digested and undigested sewage sludge from five wastewater treatment plants, primarily located in the Mälaren region in Sweden. The results address the occurrences, concentrations, and leachability of various PFAS compounds. The study also investigates the short-term degradation of PFAS precursors.

Additionally, the report discusses the following questions:

- What concentrations of PFASs and their precursors are found using target analysis and TOP assay in the investigated sewage sludge?
- How are the identified PFASs and their precursors distributed between the water phase and the soil phase in sewage sludge – and which may be more prone to leaching?
- Can the degradation of precursors be detected using the selected methods, after a short incubation period of four weeks?

2. Background

2.1 PFAS – Per- and Polyfluoroalkyl Substances

The bond between a carbon and fluorine atom (C-F) has a strong chemical resistance (Key et al., 1997). Natural organofluoride compounds are rare and contain only one C-F bond within the molecule (Jia et al. 2024). However, with synthetically induced reactions, more of these highly resistant bonds can be introduced to the molecule. Increasing the number of fluorines bound to a single carbon further strengthens the molecule, creating useful compounds that can resist strong chemical reactions.

2.1.1 Definition

PFAS, Per- and polyfluoroalkyl substances, are synthetically made fluorinated organic substances. However, there is yet no consensus or globally accepted definition of the exact molecular composition of PFAS. In 2021 the Organisation for Economic Co-operation and Development (OECD) suggested defining PFAS as molecules containing at least one fully fluorinated methyl ($-\text{CF}_3$) or methylene carbon atom ($-\text{CF}_2-$), not connected to H, Cl, Br or I atoms (OECD, 2021). This definition is accepted and used by the Swedish Chemicals Agency (KEMI). However, when reporting PFAS products for registration, the Swedish Chemicals Agency Regulations use another definition for practical reasons. Here, PFAS is defined as molecules containing at least one fully fluorinated alkyl group ($-\text{C}_n\text{F}_{2n+1}$) (KEMI, 2022b).

In summary, the term PFAS primarily refers to the presence of the chemical structures described above. These wide definitions refer to compounds with varying physical and chemical properties, leading to significant differences in their behaviour, area of use and environmental fate.

2.1.2 Terminology

There are two key traits of the chemical structure of a PFAS molecule that forms the foundation of the characterisation: the fluorinated carbon chain and the functional group (i.e head). The carbon chain can differentiate in structure, length and saturation, among others (Figure 1) As the name suggests, PFAS consists of substances with either fully fluorinated (perfluorinated) carbons where all hydrogen atoms are substituted with fluorine, or partially fluorinated (polyfluorinated) carbons where both hydrogen and fluorine occupy the carbon's bonding sites.

PFASs are often divided into the two larger subgroups of non-polymers and polymers, which can be further divided into other subgroups (Buck et al., 2011; Buck et al., 2021). Polymers consist of sequences of one or several monomer units linked together, most commonly composing larger molecules. Non-polymers consist of one chemical unit and are generally of lighter molecular weight.

Further, polymers have complex structures compared to non-polymers, complicating their characterization and evaluation of properties. Non-polymeric PFAS, on the other hand, have been more extensively studied due to the availability of standardised evaluation methods. As a result, some non-polymeric groups have conducted stronger connections to toxicity and risks. Collectively this has resulted in more regulatory measurements and prioritized scientific attention for non-polymeric PFAS.

Amongst the subgroups of non-polymeric PFAS, the most mentioned in environmental investigations and frequently detected in the environment, is perfluoroalkyl acids, PFAA (KEMI, 2022b).

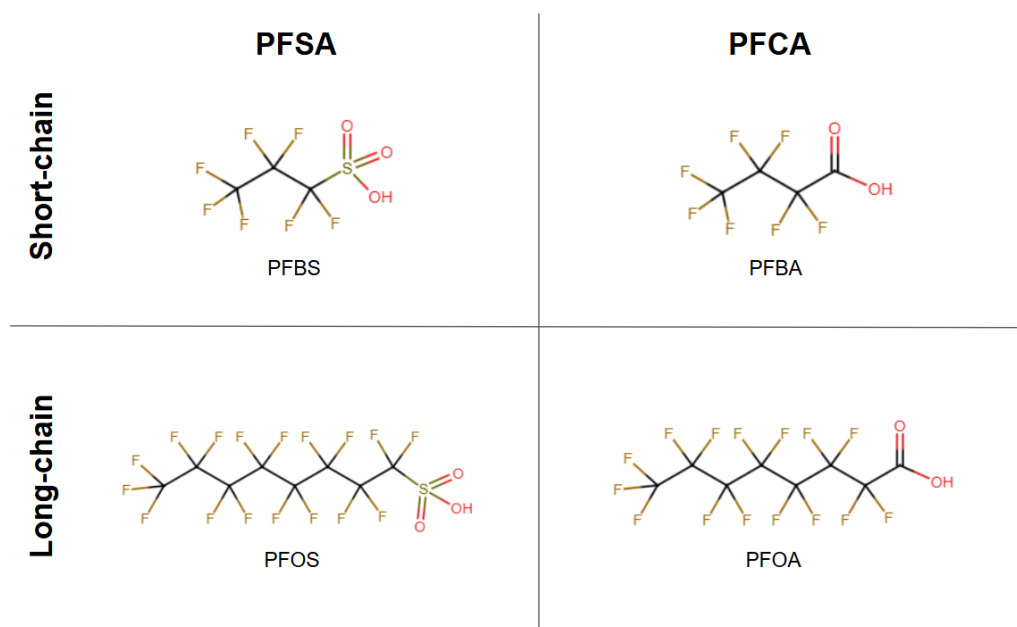


Figure 1. Examples of chemical structures of short- and long-chained PFAAs belonging to the subclasses PFSA and PFCA. (RCSB Protein Data Bank (2021).

The chemical class of PFAA (Figure 1) is further categorized based on their functional groups; perfluorinated sulfonic acids (PFSA), perfluorinated carboxylic acids (PFCA) and perfluorinated phosphonic acids (PFPA). While PFSA and PFCA have been studied for years with applicable and accessible analytic tools, PFPA remains less studied (KEMI, 2022b). PFSA has a fully fluorinated carbon chain with a functional group of sulfonic acid ($-\text{SO}_2\text{OH}$), and this class contains the well documented and studied substance perfluorooctane sulfonic acid (PFOS) (Figure 1). The subclass PFCA also consists of a fully fluorinated carbon chain but is instead connected to a carboxylic acid ($-\text{COOH}$) as its functional group (Figure 1). A recognized example for the PFCA class is the substance perfluorooctanoic acid (PFOA).

PFAS substances can also be categorized as short- or long chained. PFASs with a minimum of six perfluorinated carbons (e.g. PFOS) and PFCAs with at least

seven perfluorinated carbons (e.g. PFOA) are described as long chained (further explained in section 3.2.4).

2.1.3 PFAS Precursors

PFAS is generally described as a persistent group of compounds, resistant to natural degradation (e.g. Petrowski et al., 2024; Zhang et al., 2013). Despite this approximately 20% of PFAS may transform and degrade to some extent under natural conditions (Jiao et al., 2021). These relatively degradable PFASs are called PFAS precursors and can form persistent PFASs over time. For example, Fluorotelomer sulfonic acids (FTSs) contains the functional group of the class of PFSA (sulfonic acid, $\text{-SO}_2\text{OH}$) (KEMI, 2021). Through several reactions, 6:2 FTS can transform into the terminal perfluorohexonic acid (PFHxA), belonging to the PFCA class (figure 2) (Hamid et al., 2019). Degradation processes and related factors are described further in section 2.2.3.

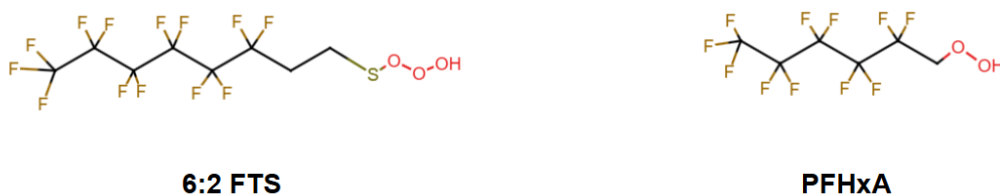


Figure 2. Chemical structures of 6:2 fluorotelomer sulfonic acid (FTS) and perfluorohexonic acid (PFHxA). (RCSB Protein Data Bank (2021)).

2.1.4 Desired Properties and Commercial Use

Important properties of the PFAS molecules in industrial settings include high durability, low surface tension and their amphilic nature (Buck et al., 2012). Common areas of use include electronics, textiles, agricultural biocides and firefighting foams. Industrial uses includes coatings, on cables, reaction vessels and architectural materials. They also appear in everyday products, such as phone screens, personal care products (e.g. shampoo and skincare), makeup and pharmaceuticals (Glüge et al., 2020; OECD, 2020; OECD, 2022; OECD, 2024). As previously stated, the molecule consists of a functional group and a fluorinated carbon chain. The PFAS-head is hydrophilic (water soluble) whereas the tail is both hydrophobic (water repellent) and oleophobic (oil repellent). These chemical traits cause the molecule to position itself in-between two different medias (KEMI, 2022b). With the tail being both oil- and water repellent, when applied to a surface the assembled layer can protect it from various contaminants, including both grease and dirt. The high durability and stability of the PFAS is also very desirable as the molecules can withstand harsh temperatures and chemical conditions, e.g. fires or a strongly acidic or oxidative solutions (Buck et al., 2012). A minimal aqueous surface tension allows for smooth application by effective

spreading and covering of the targeted surface and it can be used on various materials, such as plastics, wood, metals, hard/wet/porous and even oily elements. This fast and smooth coverage of a surface is an ideal trait for fire foam for it to be able to suffocate the fire. These traits make PFAS superior to alternative surfactant like silicones and hydrocarbons (Sharma et al., 2007).

The Organisation for Economic Co-operation and Development (OECD) (2018) provided an extensive list of PFAS that may have been or are currently on the global market, containing nearly 5000 substances. However, mapping the total use of PFAS has been proven complicated due to the lack of consensus of the definition of the chemicals, lack of proper knowledge and the rise of other PFAS through degradation of the containing product (KEMI, 2022a). This has brought the list's relevance in to question for today's market and current usage (Buck et al., 2021).

The first PFASs to appear on the market, often referred to as *conventional* or *legacy* PFASs, were long chained PFAS such as PFOS and PFOA (Calore et al., 2023; Kärman et al., 2019). Their revolutionary properties were however associated with environmental and health related issues. As these risks became evident, legislations were implemented and industries shifted to short-chained alternatives instead (UBA, 2013; Wang et al., 2013). These newer compounds are often referred to as *novel* PFASs, many of them being precursors to short-chained persistent PFASs. In an extensive screening of medias and biotas in the Nordic environment both legacy and novel PFAS substances and their precursors were detected in several mammals, with increasing trends for animals higher up in the food chain (Kärman et al., 2019).

2.2 PFAS in the Environment

PFAS can enter the environment through point sources and nonpoint sources. Examples of point sources are firefighting training sites (Filipovic et al., 2015), landfills (Elldegren, 2023), and industries (Olsen et al., 2003), while nonpoint sources involve atmospheric or waterborne transportation and the breakdown of precursors into persistent compounds (Kurwadkar et al., 2022). The biggest source of PFAS into the Swedish environment is through the use of firefighting foam and the highest risk for human consumption in Sweden is through drinking water from local hot spots, e.g. contaminated by firefighting foams (KEMI, 2022b). These contaminated fire drill areas have shown particularly high levels of PFOS due to historical use prior to regulatory legislation, and FTSs indicating ongoing use (Kärman et al., 2011).

2.2.1 Environmental and Health Related Risks

Many PFASs are bioaccumulative and bind to tissues within organisms, such as proteins in the blood and liver (Gauthier et al., 2013). The accumulation of PFAS is also seen to increase higher up in the food chain, through a process called

biomagnification, leading to greater concentrations in organisms at higher trophic levels (Androulakaki et al., 2022). As these substances do not degrade, continuous release leads to accumulating concentrations in the environment. This increases the risks of exposure and the likelihood of negative effects (Cousins et al., 2019). Exposure in humans can inhibit normal bodily functions and the development of certain organs and has shown negative effects on the immune system (Grandjean et al., 2012). Several studies have also linked PFAS exposure to cancer (Hardell et al., 2014; Garg et al., 2020). The negative effects of exposure also include decreased fertility rates and embryonic development, which can lead to declining populations and disrupt ecological balances (Marziali et al., 2019). Furthermore, the persistence of these substances alone may pose a threat, similar to the concerns surrounding microplastics (Cousins et al., 2020; Miranda et al., 2021).

2.2.2 Mobility and Retention

The structure of PFAS molecules is highly relevant for predicting their movement in different medias, thus, different PFAS substances will distinguish in their environmental fate. For example, neutrally charged PFASs have a higher volatility and may therefore partition into the atmosphere (Wang et al., 2020). In contrast, ionic PFASs, such as PFAAs may accumulate in water or sediments due to the hydrophilic properties of their functional group, or the hydrophobic properties of the fluorinated tail, respectively (Lindstrom et al., 2011; Wang et al., 2010). An important structural trait guarding PFASs transportation is the fluorocarbon chain length. Long-chained PFASs (e.g. PFOA, PFOS, PFNA, PFDA, PFDoDA and PFTeDA) consists of larger hydrophobic portions compared to the hydrophilic group, resulting in increased sorption affinity to particles (Milinovic et al., 2015). Conversely, short-chained PFAS (e.g. PFBS, PFBA and PFHxS), with a larger structural portion being hydrophilic, is likely to partition into the water phase. Short-chained PFASs are therefore more mobile than long-chained.

In soils or sediments, PFASs interact with the surrounding particles and materials through various mechanisms, including hydrophobic interactions, electrostatic forces, physical processes, and interfacial partitioning (Kookana et al., 2023). Apart from the hydrophobic interactions mentioned above, electrostatics interactions between the ionic PFASs and charged surfaces play an essential role. For example, anionic PFAAs may adsorb to positively charged sites, e.g. (hydr-)oxides, or the pH dependent charges of soil organic matter (SOM). PFAAs are generally anionic in natural soil pH (4-9) and are thus attracted to positively charged sites (Kookana et al., 2023). However, the type of headgroup can alter the strength of the sorption. For example, the sulfonate functional group of PFSAAs seem to favour sorption relative to the carboxylic functional group of PFCAs (Campos-Pereira et al., 2018; Nguyen et al., 2020). Furthermore, the uncharged sulfonamide head group (-SO₂-N-), present in e.g. MeFOSA and EtFOSA, favour sorption over anionic analogues.

The interfacial partitioning may be most important for unsaturated soils. As discussed in 2.1.4, PFAS are surface active substances and may therefore

accumulate between the air and water phases in soil pores, contributing to the overall retention of PFAS in the soil (Brusseau, 2019). The organic matter (OM) content is often positively related to sorption of PFAS, though providing less accurate predictions in mineral soils (Milinovic et al. 2015; Campos-Periera et al., 2023). These are some of the key mechanisms influencing PFAS behaviour in soil, though the overall process is highly complex. To set reliable predictions, both chemistry of the individual PFAS and soil properties must be considered.

2.2.3 Precursor Fate and Degradation

The degradation processes of precursors often proceed through multiple reactions and pathways, resulting in different compounds with varying stabilities (intermediates) in each transformation step. These reactions will ultimately yield a terminal PFAS form, if the conditions are in favour. The processes are many, including oxidation, reduction, hydrolysis, and other chemical transformations (Zhang et al., 2017; Washington & Jenkins, 2015). The matrix where these reactions take place is a determining aspect for the reaction's outcome.

Di(fluoroalkyl) phosphate (diPAP) is an example of a group of precursors used in the paper industry. The same diPAP can transform to different terminal PFASs such as Perfluoropentanoic acid (PFPeA), Perfluorohexanoic acid (PFHxA), Perfluoroheptanoic acid (PFHpA), and PFOA (Weidemann et al., 2024). In the matrix of soils, degradation depends on factors including temperature, pH, oxygen availability and microbial composition and activity (Al Amin et al., 2023; Dinglasan et al., 2004). Together with these factors, the nature of the precursor also plays a key role. For example, microbial degradation can be hindered by sorption of the precursors to soil particles (Mejia-Avendaño et al., 2016). Long-chained precursors may therefore exhibit slower transformation rates than their shorter analogues, due to differences in sorption affinity, previously discussed in section 3.2.2.

In addition to sorption, microbial degradation rates are also affected by oxygen availability (Yi et al., 2022). The transformation of fluorotelomer alcohols (FTOHs) illustrates the oxygen dependency of transformation processes that includes FTOHs as intermediates. FTOHs, a subclass of PFAS, are common intermediates in several transformation processes among various precursors, such as FTSs and Et- & Me-FOSAA (Washington et al., 2015; Shaw et al., 2019). Studies comparing transformation rates of FTOHs (specifically 8:2 FTOH) between aerobic and anaerobic systems, found considerably greater rates in the aerobic systems (Zhang et al., 2013; Yu et al., 2016). Oxygen availability is also observed to determine the direction of transformation, where aerobic and anaerobic conditions yield different end-products from the same precursors (Choi et al., 2022).

In summary, surrounding conditions and molecular structure of the precursor interact in numerous ways, creating a complex network of potential PFAS products and transformation pathways.

2.3 Swedish Sewage Sludge

2.3.1 Production and Usage

Sewage sludge is the semi-solid rest product of wastewater treatments, consisting of solid material, separated from the incoming water through various processes (Naturvårdsverket, nd). In 2020, Sweden had 429 permit-required wastewater treatment plants (WWTPs). These facilities produced a total of 208,348 tons dry matter (TS) of sewage sludge per year (SCB, 2022). The major applications for the sludge were on agricultural land (46 percent), landscaped land with normal phosphorus levels (13 percent), landscaped land with high phosphorus levels (10 percent), combustion (2 percent) and forest land (less than 1 percent). The vast majority of Sweden's produced sludge, approximately 85 percent of total net production, is used as a soil additive, i.e. mixed with soil (SCB, 2022).

Sewage Sludge as a Resource

On farmland, sewage sludge is mainly used as a natural source of nutrients (phosphorous in particular) and organic matter (Ekane & Wiklund, 2021). Studies show increased soil stocks of organic matter, decrease of bulk density, while also promoting bioactivity and plant-supporting reactions (Börjesson & Kätterer, 2018). An average application on Swedish farmland is approximately 4 ton TS per hectare every five years, i.e. 0.8 ton per hectare and year (Kärman et al., 2024). It is then mixed into the top 20 centimetres of the applied soil.

Sewage sludge application for land restoration purposes includes covering of waste rock piles and sand deposits for vegetation establishments (Stockholm Vatten och Avfall, 2018). Sewage sludge is also used for forming noise barriers. An evaluation of application and disposal methods by Stockholm Vatten och Avfall (2018) rated application on farmland highest, for high compliance with current laws, minimal environmental impact and contribution to economic performance. Volumes of applications for these purposes was not found during the writing of this report.

2.3.2 Contaminants in Sewage Sludge

A society's production and consumerism are reflected in the wastewater entering the WWTPs and the outgoing sewage sludge (Olofsson et al., 2012). The sludge can therefore be comprised of many undesirable substances. Some compounds end up in the sewage sludge due its use and consumption in society, whereas others are less soluble and may gather in the sludge due to a higher affinity (Lindberg et al., 2006). For example, pharmaceutical products such as hormones and antibiotics have been detected in sewage sludge (Östman et al., 2017). The attention receiving microplastics has also been detected in sewage sludge (Rasmussen et al. 2021). The well documented presence and risks of heavy metal in sewage sludge led to legislations and governmental control, ultimately resulting in declining trends in both sewage sludge and amended soils (Kirchmann et al., 2017). As noted, PFAS is used in various products and processes and is thereby also detected in sewage sludge (Kärman et al., 2019).

PFAS in Swedish Sewage Sludge

Detected PFASs in Swedish sewage sludge are presented in Table 1. The dominating PFASs consists of precursors, such as mono- and diPAPs which are not included in mentioned standard methods (Eriksson et al., 2015; Kärrman et al., 2019; Kärrman et al., 2024). Concentrations below limits of detection, >LOD in Table 1, refers to concentrations smaller than what could be reliably detected or analysed.

The studies evaluating sewage sludge as a source of PFAS to the applied source are sparse. However, Kärrman et al. (2024) reported higher levels of PFAS in soils where sewage sludge has been applied with one ton per hectare and year. Though worth noting that these volumes are higher than the average application on farmland, 0.8 ton hectare per year. The study could not determine accumulation of PFAS in wheat. Another study on plant uptake of PFAS found higher accumulation in vegetables compared to cereals (Mei et al., 2021).

Table 1. Concentrations (ng/g) of different PFAS found in Swedish sewage sludge (Eriksson et al., 2015; Kärrman et al., 2019; Kärrman et al., 2024). The studies analysed 1 to 3 different sewage sludges.

Substance	Concentration Span	Mean Concentration	Included in studies
PFOS	1.1 - 16.5	7.3	3
PFOA	0.6 - 0.9	0.8	2
PFHxA	0.5 - 3.3	1.4	2
PFNA	>LOD - 0.9	0.9	1
PFDA	0.3 - 3.5	1.5	2
PFUnDA	2.8 - 3.5	3.1	1
4:2 FTS	>LOD	>LOD	2
6:2 FTS	0.1 - 0.1	0.1	1
8:2 FTS	0.6 - 0.8	0.7	1
MeFOSAA	1.9 - 10.0	6.0	1
EtFOSAA	7.6 - 9.0	8.3	1
N-MeFOSA	>LOD - 1.3	1.3	1
N-EtFOSA	0.0 - 0.0	>LOD	1
N-MeFOSE	>LOD - 2.4	2.4	1
N-EtFOSE	>LOD - 0.9	0.9	1
FOSAA	22.8 - 22.8	22.8	1
GenX	>LOD	>LOD	2
(A)DONA	>LOD	>LOD	2
6:2 diPAP	2.9 - 9.7	6.9	3
8:2 diPAP	3.0 - 4.2	3.6	2

2.3.3 Sewage Sludge Treatments

Stabilisation

Stabilized sewage sludge has gone through stabilizing treatments to reduce pests and pathogens and improve handling and storage properties. In Sweden, the most common stabilization technique is anaerobic digestion (Åkerblom et al., 2020). This technique involves microbial degradation of organic matter, which also produces biogas that can be utilized as fuel (Hanum et al., 2019). As carbon is removed in the form of gases, the volume of the digested sludge is reduced while nutrients and other remaining compounds become more saturated. In the context of PFAS decontamination, the treatment does not achieve complete degradation of PFAS, i.e. mineralization of PFAS to fluorine ions (Ross et al., 2018). However, concentrations of persistent PFASs such as PFOS and PFOA, has been observed to increase during digestion, likely because of precursor transformations (Yu et al., 2009).

Sanitisation

Sanitation of sewage sludge is done to minimise contamination of salmonella (Johansen et al., 2023). A common method is storing the sewage sludge for 6 months before it is applied to farmland. The storage of sludge takes place outside.

2.4 Regulations and Legislation

2.4.1 Application of Sewage Sludge

Application of sewage sludge on agricultural land is guarded by regulations of the Swedish Board of Agriculture (Jordbruksverket) and the Swedish Environmental Protection Agency (Naturvårdsverket), with support from the Environmental Code (Miljöbalken). SJVFS 2004:62 state regulations regarding nutrients, such as restricting the dose of phosphorus from sewage sludge to a maximum of 22 kg P per hectare and year and 110 kg P per hectare and application event (Jordbruksverket, 2004). The SJVFS 2004:62 also restrict nitrogen, with stricter regulations in certain nitrogen-sensitive areas of Sweden. SNFS 1994:2 include limit values for certain metals of the sludge, the soil and of added amounts of metals to the soil through sludge application (Naturvårdsverket, 1994). However, 54 percent of produced sewage sludge is not applied on farmland and is not regulated by these limit values (SCB, 2022).

Revaq

Revaq (SPC 167) is a certification system, issued by Research Institute of Sweden (RISE), aimed to regulate the flow of toxic compounds entering WWTPs, create sustainable ways of nutrient recycling and monitor related risks (Svenskt Vatten, 2023). 44 Swedish WWTPs were certified year 2023, constituting 47 percent of total Swedish sewage sludge production. The certification demands sewage sludge producers to monitor certain trace elements, whom, when added to soil through the sludge, may have an accumulation rate higher than 0.2 percent per year. As for 2025 the certificate also includes the monitoring of PFAS. The new

demands include yearly analysis of two target analysis packages PFAS4 and PFAS22 (Table 4) where values exceeding 7.5 µg/kg DM and 25 µg/kg DM respectively, shall result in extra monitoring and planning of countermeasures to decrease these values (Svenskt Vatten, 2025). These analysis packages are considered sufficient in means of accuracy and financial availability in assessing the PFAS content. They do not include precursors such as diPAPs, Et-FOSAA or other PFAS substances that has been detected in sewage sludge, but those may be of interest in future test kits (IVL, 2023). For comparison, criteria in Denmark for sewage sludge are set as 400 µg/kg for the sum of PFAS 22 and 10 µg/kg for the sum of PFAS 4 (Lassen et al., 2024).

2.4.2 PFAS Regulations and Guidelines

Though PFAS only raised concerns as of recent years, some regulations and guidelines have been established both on national and EU level, with further regulatory measures being formulated. In 2023 the EU established legally binding limit values for several foods (European Union, 2023). Drinking water and ground water both have limit values set for a sum of set PFAS, PFAS 4 and PFAS 21, and PFAS 11 and PFAS 24 respectively (Livsmedelsverket, 2022). Several other medias such as different water sources and marine biota have legally binding limit values, but for soil, in both sensitive and less sensitive areas, only guide values are set. The guidelines focus on PFOS, 3 µg PFOS/kg TS for sensitive areas (where farmland is included) and 20 µg PFOS/kg TS for less sensitive areas (Pettersson et al. 2015). Stricter values (1.2 µg PFOS/kg TS) are being considered.

Most established regulations concern legacy PFAS, and while the novel alternatives emerged with safe profiles, new research has revealed similar related risks (Fenton et al., 2020). As previously stated, PFAS-related regulations are much evolving. The Swedish chemical agencies and other corresponding agencies in EU are working towards assessing PFAS as a group instead of individual substances, the goal being to eventually phase out PFAS entirely (ECHA, 2023). For example, the Registration, Evaluation and Restriction of Chemicals (REACH) are working towards a ban on PFAS in firefighting foams, the highest source for exposure in Sweden. Though no date of implementation is currently set.

2.5 Analytical Methods

2.5.1 Target Analysis

Reference standards have been developed for some individual PFAS substances with known chemical structures. These can be used to target those specific PFASs in samples in an analytical method called target analysis (Rehman et al., 2023). The method uses high-resolution chromatography together with mass spectrometry (LC-MS/MS) to detect and quantify the targeted substances, with high accuracy and sensitivity (Lai et al., 2019). Specific PFASs are selected according to known environmental prevalence or specific interests (e.g. regulatory or risk assessment). Swedish limit values and guidelines for PFASs are set based on target analysis. These regulatory values are either set for one specific compound, such as PFOS, or for a sum of selected PFASs, such as the widely

used sets of PFAS4 or PFAS22 (LIVSFS 2022:12). These sets contain PFASs with known hazardous effects, such as PFOA, PFNA PFOS and PFHxS, which are all included in PFAS4. The method is however limited to only detect the selected targeted substances and does not give information about the total amount of PFAS in a sample.

2.5.2 Total Oxidisable Precursors Assay

Total Oxidisable Precursor assay, often referred to as TOP assay, is a method that converts oxidizable PFASs (using NaOH and kalium persulfate, $K_2S_2O_8$) into PFAAs which are then analysed through target analysis (Ateia et al., 2023). The method requires duplicate samples, where one is oxidised, transforming PFAS precursors into intermediates (not fully transformed) or terminal oxidation products (that cannot transform further through oxidation). The end products of the oxidized sample are PFASs with carbonylic groups, such as a carboxylic acid (-COOH). The other duplicate sample is left untreated. Concentrations are then measured in both samples through target analysis, using the same targets. By using the untreated concentrations as a reference, values of PFAA precursors can be estimated in the oxidised sample. Limitations for this method include only detecting targeted substances and precursors that can be transformed through oxidation (Liu et al., 2024). The result may therefor exclude unknown or untargeted PFASs and substances resistant to oxidation. For example, a recorded transformation rate through oxidation of the novel PFAS DONA, i.e. ADONA, to the intermediate form PFMOPrA was between 98-20 % (Zhang et al., 2019).

2.5.3 Leaching Procedure

The batch leaching test, also referred to as the shake test, is a standardized laboratory procedure used to assess the leaching potential of contaminants from solid waste materials into aqueous phases. It simulates conditions under which pollutants may be mobilized from solid matrices when in contact with water. The test involves mixing a representative solid sample with deionized water at a defined liquid-to-solid (L/S) ratio, followed by agitation under controlled conditions (Townsend et al., 2003).

Measuring Leachability

The distribution coefficient (K_d) is commonly used to describe a substance's sorption affinity by comparing its concentration in the solid phase to that in the liquid phase under equilibrium conditions (Pettersen et al., 2015; Campos-Pereira et al., 2023). A higher K_d value indicates stronger sorption to the solid phase, whereas a lower value suggests greater mobility in the liquid phase. Given the strong affinity of PFAS compounds for organic matter, it is also common to calculate the distribution coefficient K_{oc} , which provides an estimate of sorption specifically to organic matter.

Both K_d and K_{oc} assume linear adsorption isotherms, without considering any specific binding mechanisms. As discussed earlier, PFAS sorption and mobility

are complex and highly site-specific processes. Nonetheless, these coefficients offer a useful means of comparing the relative sorption affinities of different PFAS compounds under similar conditions.

3. Method

3.1 Literature Search

In the search for sources of information and references of sewage sludge related values, limitations were set to Swedish sources, as these values are highly dependent on societal trends.

3.2 Sample selection and collection

3.2.1 Sewage Sludge

Selection of Wastewater Treatment Plants

Five WWTPs were selected in eastern Sweden, mainly in the Mälaren Valley region. Three selected plants (A-C) represent large municipal facilities responsible for the majority of Sweden's wastewater treatment and sludge production, where the sludge undergoes digestion. The other two (D-E) represent smaller plants where the sludge is not digested (table 2).

Table 2. Description of the capacity, sewage sludge (SS) production and SS treatment of the corresponding Wastewater Treatment Plants (WWTPs) (A-E), where sampled SS were produced. Sanitation duration describes the length of storage time of the sampled sludge.

WWTP	Capacity (pe)	Yearly SS production (ton DM/year)	SS Treatment	Sanitation duration (months)
A	<1000000	<10000	Digestion	3-4
B	<1000000	<10000	Digestion	4-5
C	<1000000	<10000	Digestion	3-4
D	>50000	>3000	Untreated	4
E	>50000	>3000	Untreated	3

Sampling of Sewage Sludge from Stored Stockpiles

The sampled sewage sludge had been stored for sanitisation for approximately 3–5 months at the time of sampling (Table 2). Sampling was conducted during the transition between January and February on rain-free days. A metal sampling rod, 120 cm in length, was used to collect approximately 30–40 cores per replicate. All equipment was rinsed with 99% methanol prior to, and between sampling from different WWTPs. Precautions were made to minimize the risk of PFAS contamination from sampling clothing and equipment. Sealed samples were stored outdoors (temperatures around 0 degrees Celsius) in shade prior to analysis to preserve equal temperatures for all sewage sludges. See sampled volumes in Table 3.

Table 3. Description of volumes of stored stockpiles of sewage sludge that were sampled, sample volumes and the corresponding sample representation of the sampled stockpiles. Note that replicates of SS C-E were all sampled from the same stockpile.

SS Sample	Replicate	Sampled SS Volume (ton)	Volume of sample (g)	Representation of Sample (%)
A	1	790	590	0.00007
	2	889	495	0.00006
	3	690	520	0.00008
B	1	1044	765	0.00007
	2	917	975	0.00011
	3	1137	435	0.00004
C	1	1647	595	0.00004
	2		685	0.00004
	3		860	0.00005
D	1	599	615	0.00010
	2		560	0.00009
	3		855	0.00014
E	1	38	1035	0.00270
	2		750	0.00195
	3		980	0.00255

3.2.2 Soil

The soil needed to have a texture that allowed for easier handling in laboratory settings, as soils with high clay content can be challenging to work with when it comes to obtaining a homogenised mix with sewage sludge. Additionally, it was important that the soil supported biological activity, with adequate nutrient availability and effective gas and water exchange. Lastly, its relevance to practical applications was considered, particularly in relation to the common practice of applying sewage sludge to agricultural fields. The soil was collected from a potato field in Kristianstad, Sweden, where the dominating soil types of the area consists of sand and till (SGU, 2020).

3.3 PFAS analysis

The samples were defrosted at room temperature and homogenized by stirring. Water content was determined by drying the samples at 105 degrees Celsius. To prepare the LS 10 leaching solution, each sample was mixed with ionized water in proportion to its measured water content, following the ISO standard 21268-2. Following 24 hours of agitation, the mixtures were allowed to settle for approximately one hour before the supernatant was decanted into containers for leachate analysis. Approximately 50 grams of each homogenized sample were set aside for analysis of the solid phase.

Laboratory analysis of PFAS and other parameters was conducted by the accredited laboratory SGS Analytics Sweden. The analytical methods of target

PFAS analysis and TOP Assay was conducted using the standard method for examination of sludge, DIN 38414-14, which uses high performance liquid chromatography and mass spectrometric detection (HPLC-MS/MS). Targeted PFAS substances were chosen from SGSs analytical target packages, based on previous PFAS screening of sewage sludge. The chosen targets for both methods are presented in Table 4, together with the PFAS 4 and PFAS 22 – which sums could be calculated from the results. Target analysis (49 targets) and TOP assay (22 targets) were used to analyse PFAS in solid and liquid phase of the five sampled triplicates of sewage sludge and in solid phase only of the soil. The laboratory used the ISO standard 21675:2019 (2019) for extraction of PFAS in the liquid phase.

Other measured parameters included pH, using the method SS-EN ISO 10390:2022, aluminium and iron using EN ISO 54321 and total phosphorus using EN 16171.

Table 4. Table of analysed PFASs and the PFASs included in PFAS 22 (used for TOP Assay) and PFAS 4.

Substance	Target Analysis (49 targets)	TOP Assay (PFAS 22)	PFAS 4
PFOS	X	X	X
PFOA	X	X	X
PFNA	X	X	X
PFHxS	X	X	X
PFBS	X	X	
PFPeA	X	X	
PFHxA	X	X	
PFHpA	X	X	
PFHpS	X	X	
PFPeS	X	X	
PFDS	X	X	
PFNS	X	X	
6:2 FTS	X	X	
PFBA	X	X	
PFDA	X	X	
PFUnDA	X	X	
PFDoDA	X	X	
PFOSA	X	X	
PFTTrDA	X	X	
PFUnDS	X	X	
PFDoDS	X	X	
PFTTrDS	X	X	
4:2 FTS	X		
8:2 FTS	X		
10:2 FTS	X		
HPFHpA	X		

PFTeDA	X		
PFHxDA	X		
PFODA	X		
N-MeFOSAA	X		
N-EtFOSAA	X		
N-MeFOSA	X		
8:2 diPAP	X		
GenX	X		
PFPrS	X		
N-MeFBSAA	X		
PFBSA	X		
(A)DONA	X		
PFECHS	X		
N-MeFBSA	X		
PFHxSA	X		
8:2 FTUCA	X		
P37DMOA	X		
9Cl-PF3ONS	X		
H4-PFUnDA	X		
11Cl-PF3OUdS	X		
N-MeFOSE	X		
N-EtFOSE	X		
N-EtFOSA	X		

3.3.1 Processing of Analytical Results

Estimation and Handling of Non-Detect Values

Reported concentrations were excluded from the results if they fell below the laboratory reporting limit (RL) in all replicates of a given sample – or in all replicates across all samples when evaluating summed values. For the calculation of mean concentrations within individual samples, values below the RL were assigned a value equal to half of the RL. This approach enables the inclusion of all available data in the analysis.

Calculation of K_d & K_{oc}

The distribution coefficient (K_d , l/kg) was calculated using equation 1 from mean concentrations of each analysed PFAS (Swedish Environmental Protection Agency, 2009). Concentration solid is the concentration measured from solid samples (ug/kg TS), Concentration liquid is the concentration measured from the liquid samples (ug/l) and LS is the liquid-to-solid ratio used in the shake test (LS=10).

$$K_d = \frac{\text{Concentration solid} - LS * \text{Concentration liquid}}{\text{Concentration liquid}} \quad (1)$$

K_{oc} (l/kg), describing the sorption to organic matter was calculated using equation 2 and the mean total organic carbon (TOC) concentrations for each PFAS and sample (Calvet, 1989).

$$K_{oc} = \frac{K_d}{f_{oc}} \quad (2)$$

Statistical Analysis

Statistical analyses were conducted using Microsoft Excel. Paired two-tailed t-tests were used to assess significant differences in PFAS concentrations using conventional target analysis and TOP assay. Two-sample t-tests assuming equal variances were applied to compare concentrations between different sewage sludge samples. A significance level of $p < 0.05$ was used to determine statistical significance.

3.4 Incubation

Preparation

Based on the results from Part I, sewage sludge samples A (digested) and E (undigested) were selected for further analysis, as they exhibited the largest differences between concentrations measured using target analysis and the TOP assay, within the digested and undigested SSs. To facilitate the preparation of a light and airy soil–sludge matrix, each SS sample was dried by air-drying. However, due to slower drying of sample E, an oven was used, set to approximately 50 degrees. The water content of both the sewage sludge and the soil was determined by drying the samples at 105 degrees Celsius. A mixture ratio of 1 part sewage sludge (dry weight) to 2 parts soil (dry weight) was chosen to ensure that PFAS concentrations from the SS would remain detectable and not be too diluted in the soil.

Due to the limited quantity of sewage sludge remaining, the prepared portions for each incubation experiment were just sufficient to meet the requirements for the LS 10 leaching test, which demands 90 g of solid material. This left approximately 20 g of each mixture available for solid-phase analysis. Each replicate (3x) of sewage sludge (A and E) was mixed with soil according to the measured dry weights. In total, two sets of six mixtures containing 90 g of solid matter and six mixtures containing 20 g were prepared. Of these, three of the 90 g mixtures and three of the 20 g mixtures were analysed immediately, representing the start of the incubation. The remaining half were sealed with parafilm and stored in darkness at room temperature for a duration of four weeks, after which they were analysed.

Analysis and Processing of Results

Both solid and liquid samples were analysed as described in section 3.3. The processing of reported results follows section 3.3.1.

4. Results

4.1 Part I: PFAS in Sewage Sludge

A measurement uncertainty of ± 0.36 ng/g TS was reported, with a coverage factor of 2, corresponding to approximately 95% confidence. It was noted that the results from TOP Assay may be underestimated due to incomplete oxidation, caused by high contents of oxidizable organic material in the sample.

Reporting limits (RL) varied: <0.2 to <2 ng/g and <10 to <150 ug/l for target analysis of solid and leachate samples, respectively; <0.2 to <1.6 ng/g and <12.5 to 25 ng/l.

4.1.1 Detection Frequency

Solid SS samples analysed with target analysis reported detections over RL for 20 out of 49 analysed PFASs (figure 3). Most frequent PFASs (detected across all SS, A-E) were N-EtFOSAA, 10:2 FTS and PFOS. Other PFASs detected in the majority of sampled SS (three out of five SS) were N-MeFBSAA, N-MeFOSAA and PFDA. Leachate SS samples analysed with target (49) reported detections over RL for 9 out of 49 analysed PFASs (figure 4). Most frequent PFAS (detected in all SS, A-E) was PFHxA.

Among the results from TOP Assay for solid and leachate SS samples, 12 and 11 PFASs respectively, were detected above RL, out of 22 analysed PFASs (figure 3 and 4). Most frequently detected (detected in all SS, A-E) in solid samples (TOP 22) were PFDoDA, PFDA, PFNA, PFBA, PFOA, PFHpA, PFHxA, PFPeA and PFOS. The leachate samples frequently detected PFDA, PFNA, PFBA, PFOA, PFHpA and PFHxA, in all sampled SS (A-E).

Seven PFAS substances (PFPeS, PFHpS, PFDS, PFNS, PFUnDS, PFDoDS and PFTrDS) were not detected above detection limits in any of the analysed samples and will not be included further in the results.

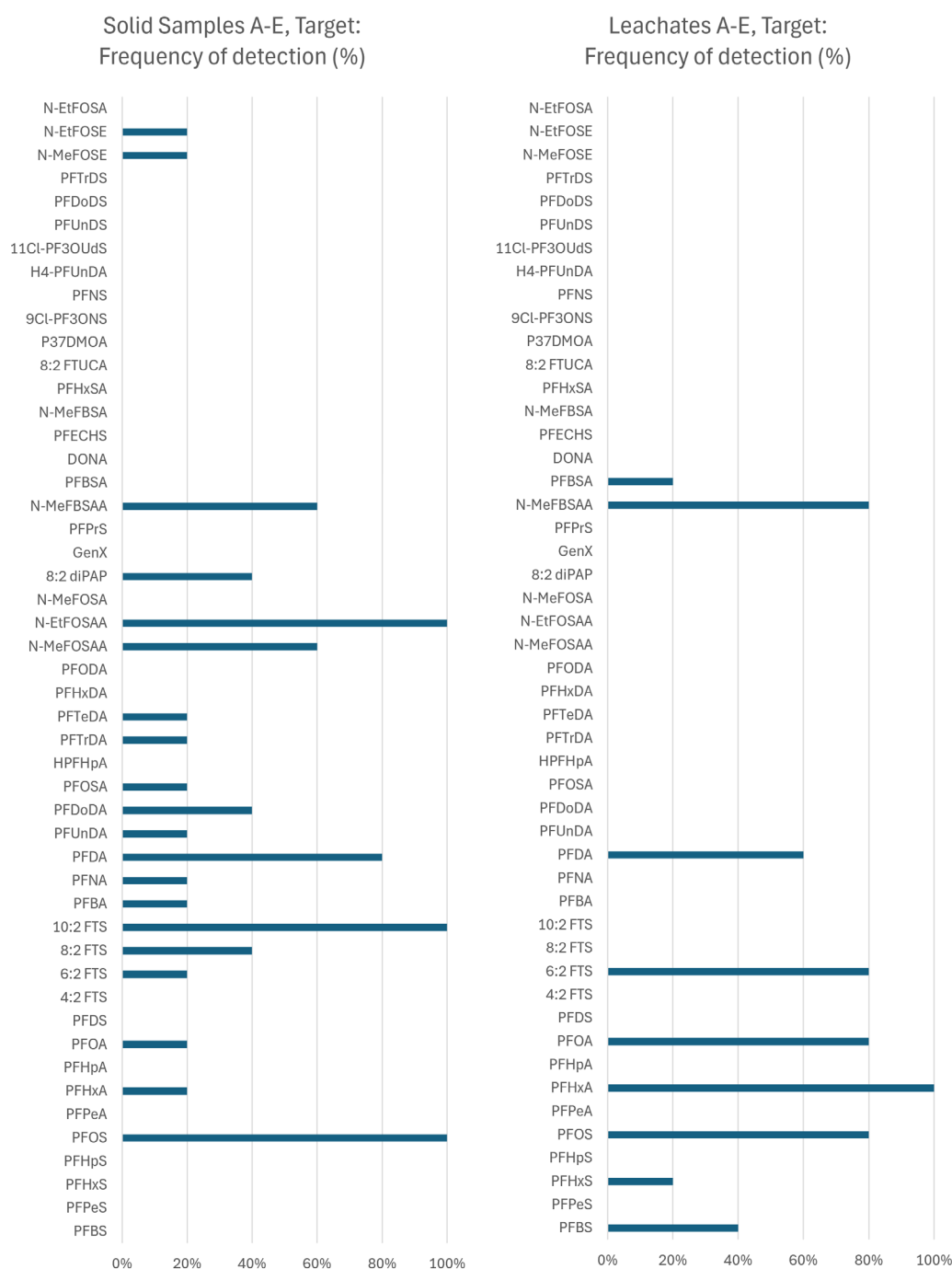


Figure 3. Detection frequency (%) of individual PFAS compounds across sampled SS (A-E), for solids (to the left) and leachates (to the right), analysed using target analysis (49 targets). A compound was considered detected in a given sludge if it was present above the laboratory reporting limits (RL) in at least one of the three replicates analysed. The detection frequency reflects the number of sludge samples (out of five) in which each PFAS met this criterion.



Figure 4. Detection frequency (%) of individual PFAS compounds across sampled SS (A-E), for solids (to the left) and leachates (to the right), analysed using TOP Assay (22 targets). A compound was considered detected in a given sludge if it was present above the laboratory reporting limits (RL) in at least one of three replicates analysed. The detection frequency reflects the number of sludge samples (A-E) in which each PFAS met this criterion.

4.1.2 Solid Sewage Sludge Samples

Target analysis: Solid Samples

PFOS showed the highest mean concentration in sludges A–D (3.3–7.3 ng/g TS), while PFOS, 10:2 FTS (1.9–4.7 ng/g TS), and N-EtFOSAA (2.6–4.4 ng/g TS), all long-chained PFAS, had the higher mean concentrations across all SS (Figure 5 and 6). Undigested sludges D and E exhibited significantly lower total PFAS concentrations ($p=0.0117$) than the digested SS A–C (28.5–46.7 ng/g TS; 36.7–49.3 ng/g TS). When viewing the Figure 6, note that SS E had concentrations over RL for only 3 PFASs: PFOS (1.07 ng/g TS), 10:2 FTS (0.8 ng/g TS) and N-EtFOSAA (1.6 ng/g TS). Low variability among the lower concentrations is due to many values being close to, or under the reporting limits.

The target analysis including 49 PFASs showed significantly higher total concentrations (1.5–26.7 ng/g) compared to the sum of PFAS 4 (0.5–9.7 ng/g) and PFAS 22 (0.5–14 ng/g) ($p=0.0004$ and 0.005 , respectively). No significant difference was observed between PFAS 4 and PFAS 22.

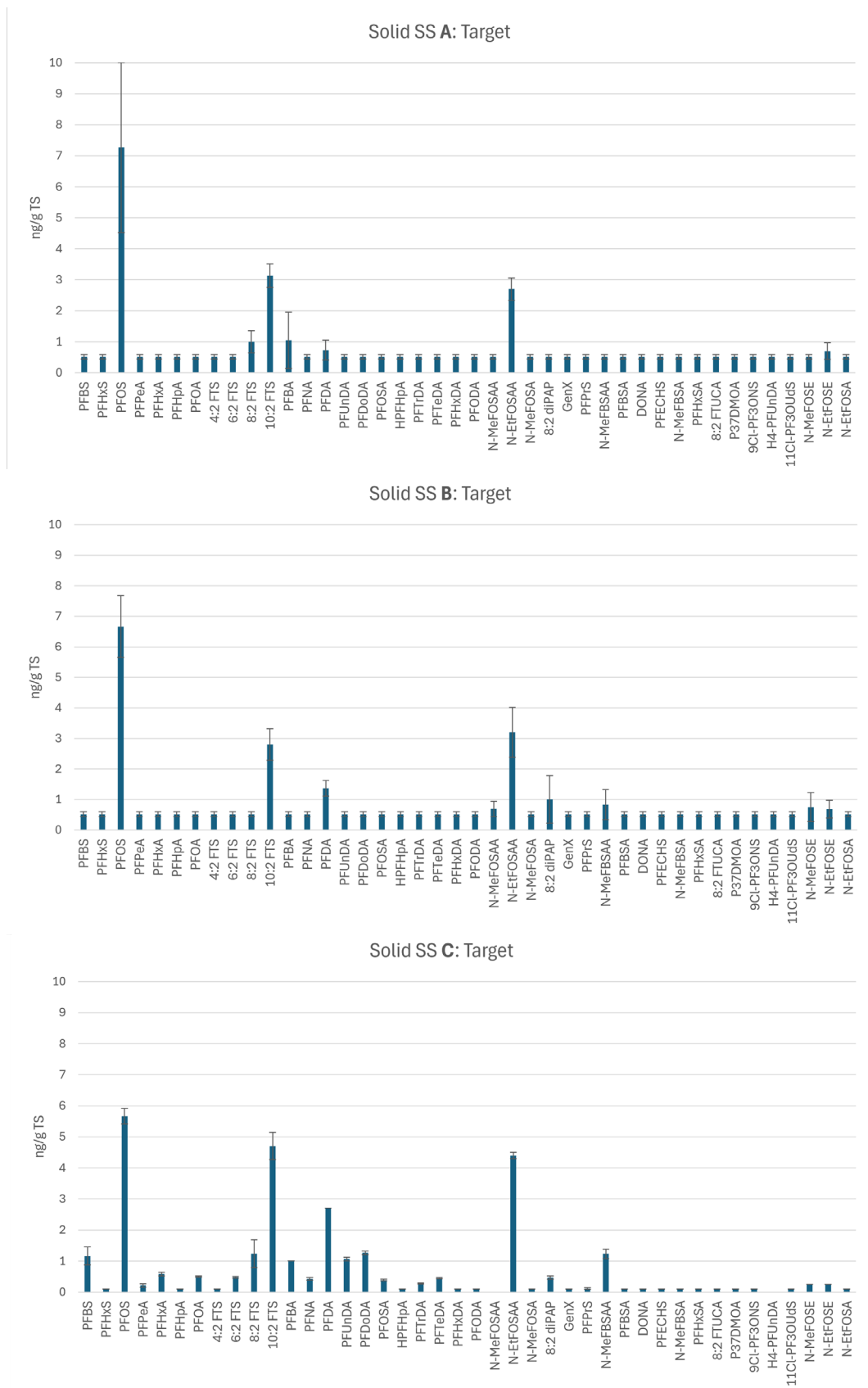


Figure 5. PFAS concentrations (ng/g TS) in solid SS samples A-C, using target analysis (49 targets).

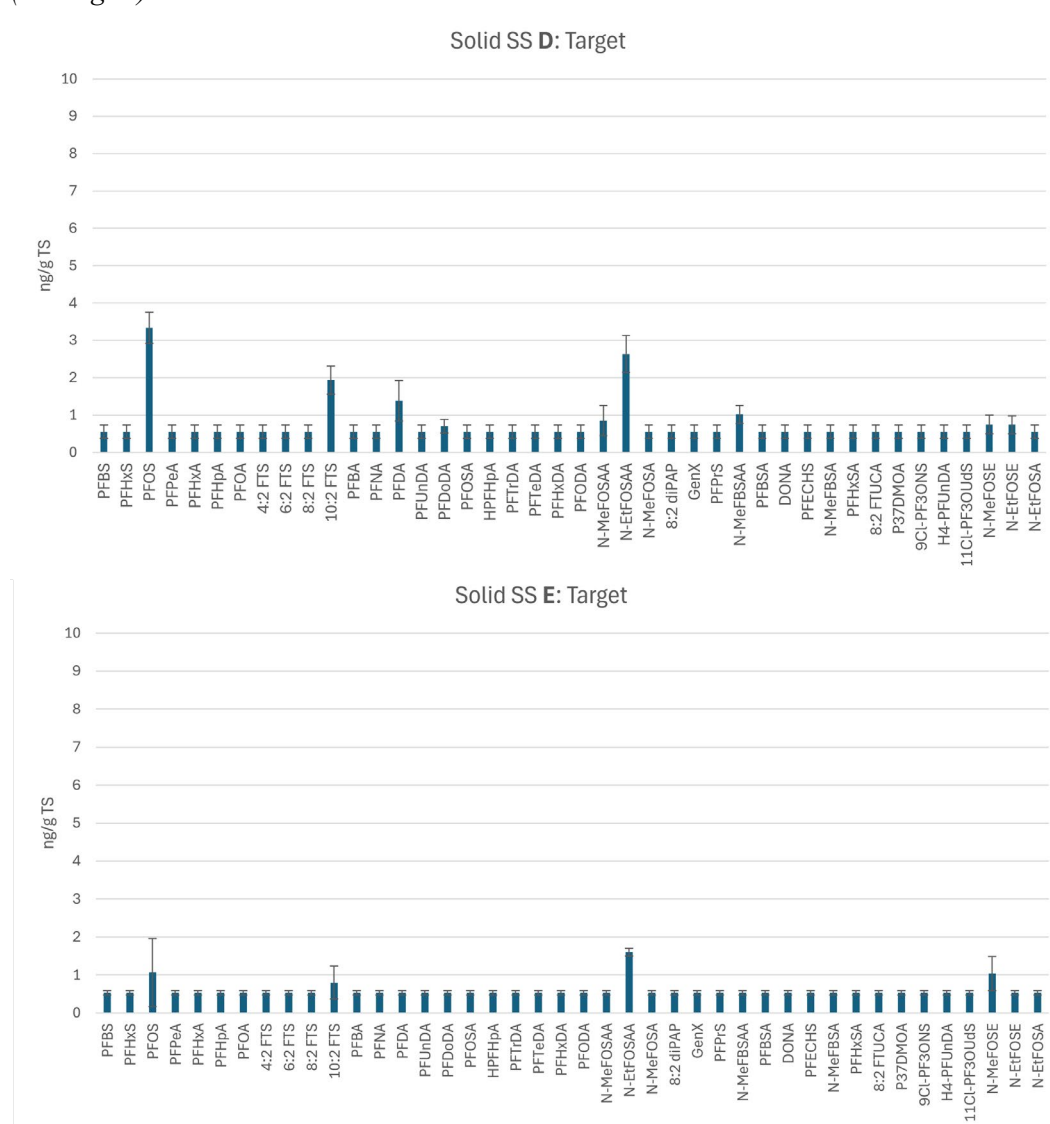


Figure 6. PFAS concentrations (ng/g TS) in solid SS samples D-E, using target analysis (49 targets).

TOP Assay: Solid Samples

Total PFAS concentrations were significantly higher measured from TOP Assay (TOP) compared to the results from target analysis ($p < 0.0001$). On average, total PFAS concentrations measured using TOP were approximately 496% higher than those measured using target analysis (Figure 8). Specifically, concentrations of PFPeA ($p = 0.0015$ – 0.0245), PFHxA ($p = 0.0016$ – 0.0146), PFHpA ($p = 0.0014$ – 0.0129), PFOA ($p = 0.0032$ – 0.0123), PFBA ($p = 0.0010$ – 0.0122), and PFNA ($p = 0.0018$ – 0.0213) increased significantly across all sludge samples, while PFDA ($p = 0.0088$ – 0.0293) increased significantly in all except SS C (Figure 7). In SS C, PFBS ($p = 0.0360$), PFOSA ($p = 0.0035$), and PFTeDA ($p = 0.0026$) also showed significant increases. In contrast, PFOS concentrations were lower using TOP in SS A to C, however not significantly so. A significant difference in total PFAS

concentrations was also observed between digested and undigested SS, with higher concentrations in the undigested samples ($p=0.0124$). Across all samples, PFPeA exhibited the highest mean concentration (13–35 ng/g), followed by PFHxA (11–21 ng/g), PFBA (11–21 ng/g), PFOA (7–10 ng/g), and PFHpA (5–9 ng/g).

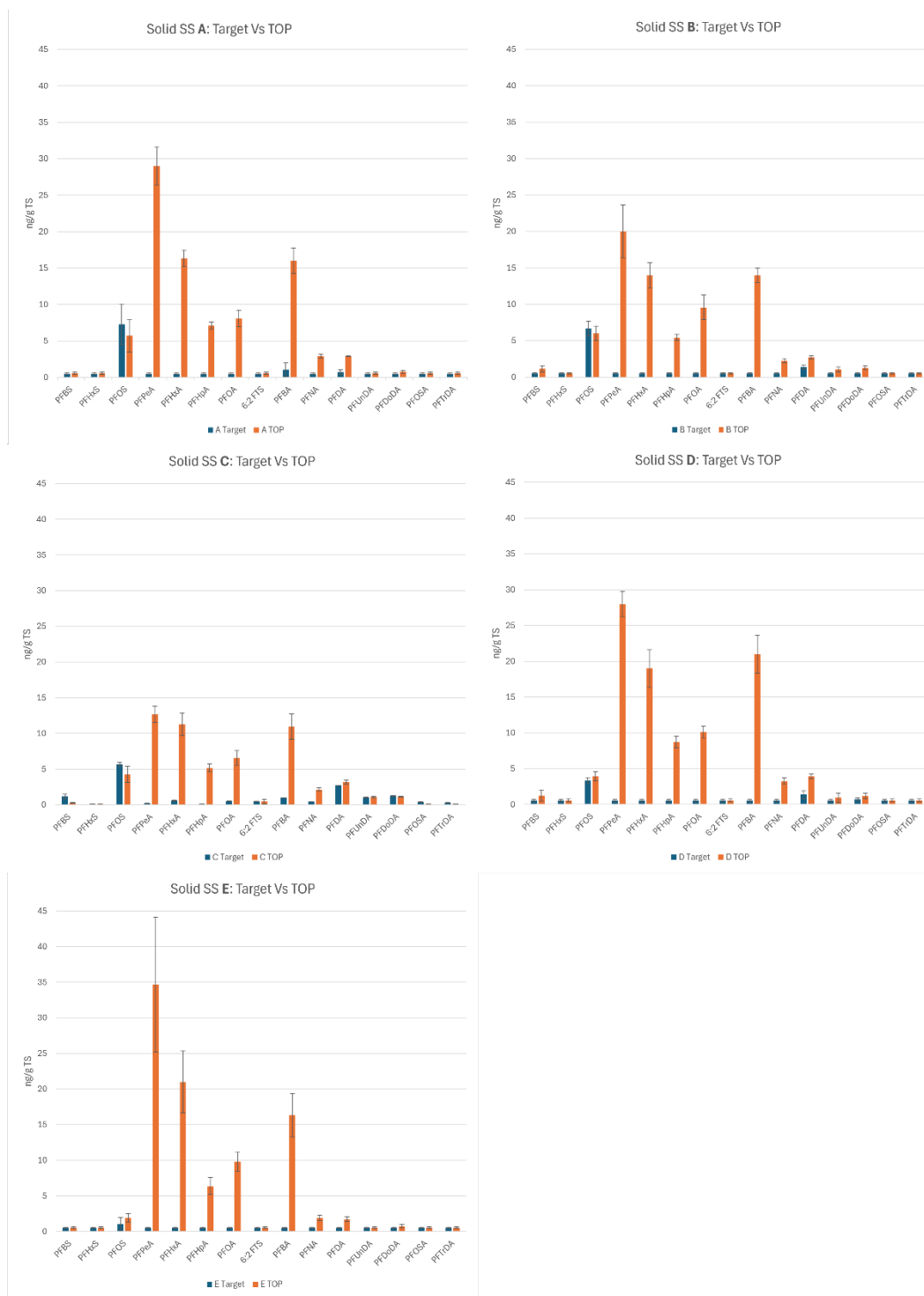


Figure 7. Comparison of PFAS concentrations (ng/g TS) between solid SS samples analysed using TOP Assay (blue) and solid SS samples analysed using target analysis (orange), A-E.



Figure 8. Calculated sums of PFAS 4 (pink) and PFAS 22 (green) from solid samples A-E, using target analysis (darker colour) and TOP Assay (lighter colour), in units ng/g. The total concentrations of target analysis, using 49 targets, above RL is also depicted in dark blue. REVAQ guidelines are set for 7.5 ng/g (PFAS 4) and 20 ng/g (PFAS 22), for comparison.

4.1.3 Leaching of Sewage Sludge Samples

Target analysis: Leachate Samples

N-MeFBSAA was detected at the highest mean concentrations in leachates from SS A–D (96–116 ng/l) (Figure 9 and 10). In samples A and B, PFHxA and PFBA were also among the top three most abundant compounds, with concentrations of 58 and 98 ng/l and 83 and 75 ng/l, respectively.

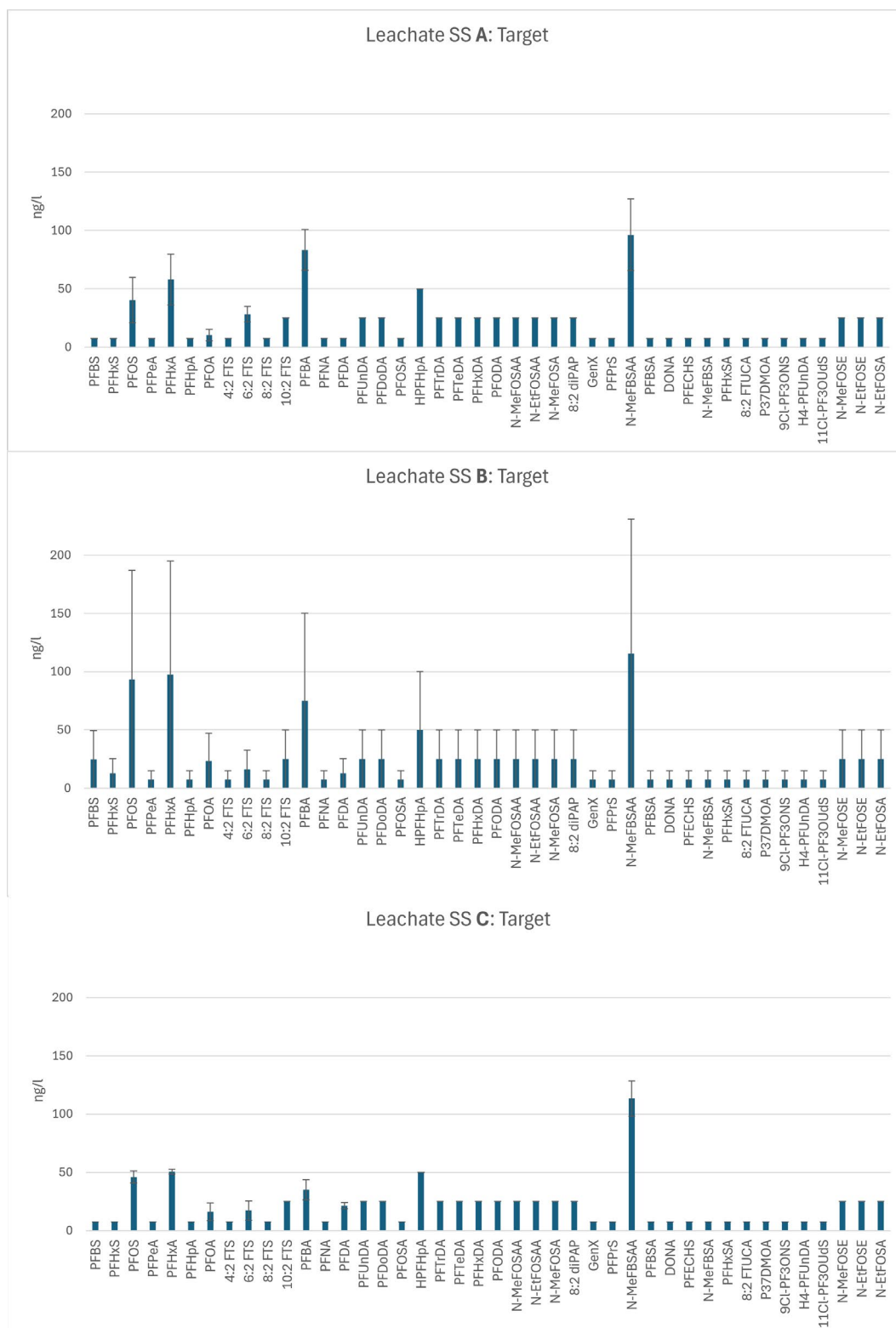


Figure 9. PFAS concentrations (ng/l) in leachate SS samples A-C, using target analysis (49 targets).

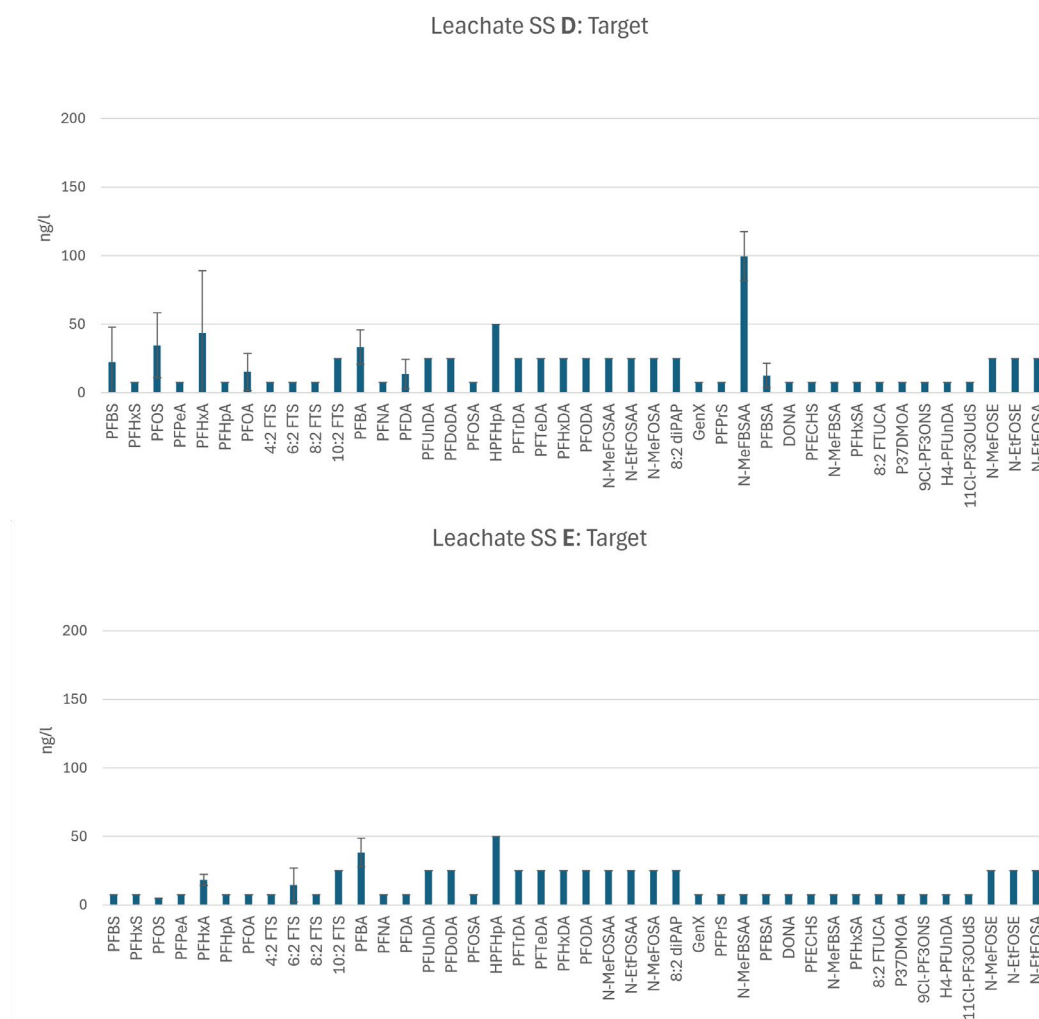


Figure 10. PFAS concentrations (ug/l) in leachate SS samples D-E, using target analysis (49 targets).

TOP Assay: Leachate Samples

Total PFAS concentrations were significantly higher in samples analysed using TOP across all SS ($p=0.0002$). Concentrations of PFPeA and PFBA were significantly higher in SS A–C and E ($p=0.0009$ – 0.0467 and $p=0.0025$ – 0.0783 , respectively), while PFHxA, PFHpA, PFOA, and PFNA increased significantly in SS A, C, and E ($p=0.0035$ – 0.0249 , $p=0.0008$ – 0.0221 , $p=0.0063$ – 0.0254 and $p=0.0009$ – 0.0147 , respectively). PFBS and PFDA showed significant increases in SS A and C ($p=0.0117$ – 0.0298 and $p=0.0154$ – 0.0315), and PFOS and PFDoDA in SS C ($p=0.0422$ and $p=0.0065$). The highest mean concentrations across all SS, were observed for PFPeA (250–1133 ng/l). The second highest concentrations measured in all SS, except SS D, was PFHxA (213–1026 ng/l). In SS D, the second highest concentration was observed for PFBA (290 ng/l). No significant difference was detected for SS D, likely due to high variance among the triplicates, displayed in the standard errors in figure 11.

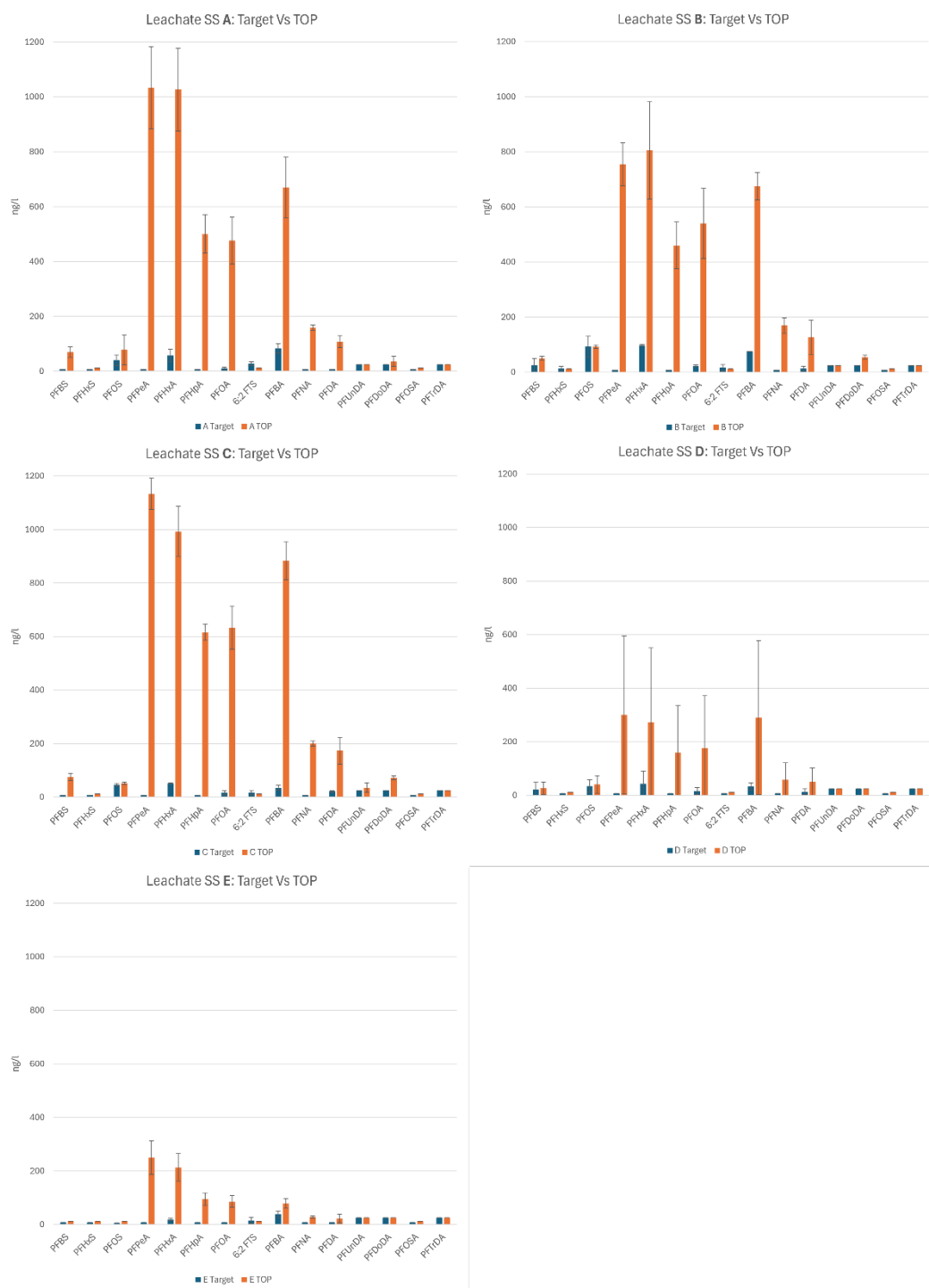


Figure 11. Comparison of PFAS concentrations (ng/l) between leachate SS samples using target analysis (blue) and TOP Assay (orange), A-E.

K_d and K_{oc}

Total organic carbon (TOC) was significantly higher in undigested sludge compared to digested (Table 5). Most PFASs also showed higher K_{oc} values in undigested sludge, although no statistical analysis was performed (Table 7). For

PFOS, 6:2 FTS, PFUnDA, and PFDoDA, K_{oc} values were more similar between sludge types, though this was not statistically confirmed.

Table 5. Mean Total Organic Carbon (TOC) of each sewage sludge sample.

SS Sample	A	B	C	D	E
TOC (% TS)	34.3	34.3	33.0	43.7	38.3

Table 6. Mean Total Solid (TS) of each sewage sludge sample.

SS Sample	A	B	C	D	E
TS (%)	24.5	24.4	24.1	23.3	23.8

Table 7. Calculated K_d Values for each PFAS and group (A-E).

	K_d (l/kg)						
	A	B	C	D	E	Mean	Range
PFBS	-5.4	37.9	-18.7	84.4	88.7	37.4	-18.7 - 88.7
PFOS	182.8	159.8	219.9	199.5	370.4	226.5	159.8 - 370.4
PFPeA	52.6	48.0	3.6	190.8	335.7	126.1	3.6 - 335.7
PFHxA	17.2	21.5	4.2	136.9	230.7	82.1	4.2 - 230.7
PFHpA	12.2	5.3	-4.9	102.1	148.7	52.7	-4.9 - 148.7
PFOA	20.2	22.5	1.1	108.0	273.5	85.1	1.1 - 273.5
6:2 FTS	102.9	95.1	83.6	77.9	88.7	89.7	77.9 - 102.9
PFBA	40.4	31.3	7.2	142.9	515.5	147.5	7.2 - 515.5
PFNA	24.3	9.7	2.0	105.3	150.9	58.4	2.0 - 150.9
PFDA	49.5	33.0	25.1	157.9	182.6	89.6	25.1 - 182.6
PFUnDA	36.9	93.2	63.8	62.6	31.3	57.5	31.3 - 93.2
PFDoDA	33.5	37.2	17.2	87.0	49.7	44.9	17.2 - 87.0

Table 8. Calculated K_{oc} Values for each PFAS and group (A-E).

	K_{oc} (l/kg OC)						
	A	B	C	D	E	Mean	Range
PFBS	-1.9	13.0	-6.2	36.8	34.0	15.2	-6.2 - 36.8
PFOS	62.8	54.9	72.6	87.1	142.0	83.9	54.9 - 142.0
PFPeA	18.1	16.5	1.2	83.3	128.7	49.5	1.2 - 128.7
PFHxA	5.9	7.4	1.4	59.8	88.4	32.6	1.4 - 88.4
PFHpA	4.2	1.8	-1.6	44.6	57.0	21.2	-1.6 - 57.0
PFOA	6.9	7.7	0.4	47.2	104.8	33.4	0.4 - 104.8
6:2 FTS	35.3	32.7	27.6	34.0	34.0	32.7	27.6 - 35.3
PFBA	13.9	10.7	2.4	62.4	197.6	57.4	2.4 - 197.6
PFNA	8.3	3.3	0.7	46.0	57.9	23.2	0.7 - 57.9
PFDA	17.0	11.3	8.3	68.9	70.0	35.1	8.3 - 70.0
PFUnDA	12.7	32.0	21.0	27.3	12.0	21.0	12.0 - 32.0
PFDoDA	11.5	12.8	5.7	38.0	19.1	17.4	5.7 - 38.0

4.2 Part II: Incubation of Sewage Sludge and Soil

The reported measured uncertainty was reported as ± 0.1 . However, due to technical issues and dilution of samples reported from the laboratory, the uncertainty is likely higher.

A significant decrease in PFOS concentrations ($p=0.024$), as well as in the summed concentrations of PFAS 4 and PFAS 22, was observed during the incubation, in the solid samples from sludge A, using target analysis (target 49). No significant changes were detected in the solid samples from sludge E, using target analysis (49 targets). In the results from TOP Assay, significant decreases were found for PFNA ($p=0.017$ in A; $p=0.038$ in E) and PFDoDA ($p=0.007$ in A; $p=0.018$ in E) (Table 12). Additional significant reductions were observed in sample A for PFOA ($p=0.040$), PFDA ($p=0.017$), and PFUnDA ($p=0.036$). In contrast, a significant increase in PFBS ($p=0.028$) and PFOS ($p=0.0019$) was recorded in the SS E-mix using TOP.

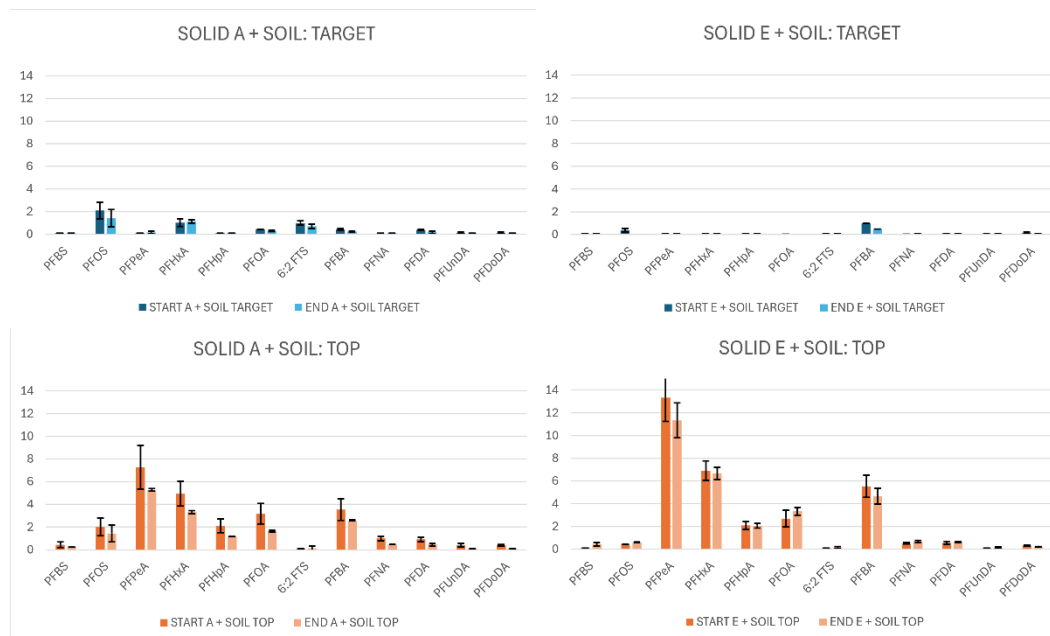


Figure 12. Mean concentrations of PFAS (ng/g) with standard errors, in solid samples of SS A and E, mixed with one third soil. Measurements at the beginning of the incubation (darker colour) and after one month (lighter colour), using both target analysis (blue) and TOP Assay (orange). PFASs with concentrations below the reporting limits in all replicates have been excluded.

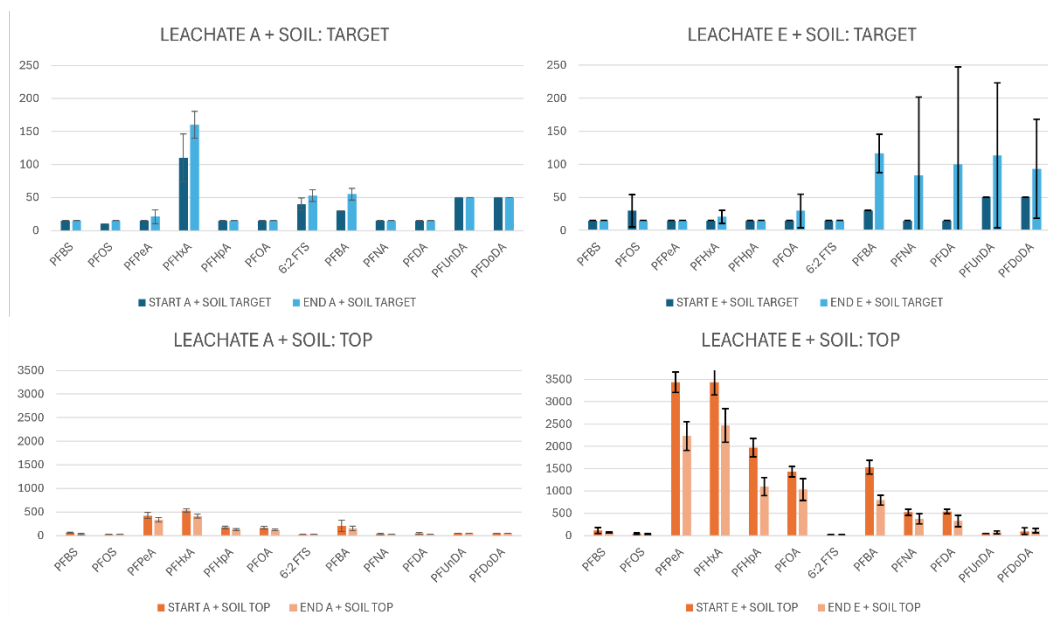


Figure 13. Mean concentrations of PFAS (ng/l) with standard errors, in leachate samples of SS A and E, mixed with one third soil. Measurements at the start of the incubation (darker colour) and after one month (lighter colour), using both target analysis (blue) and TOP Assay (orange). PFASs with concentrations below reporting limits in all replicates have been excluded.

In the leachates analysed with target, significant increase in concentrations were only seen in sample S for the sum of PFASs above RL ($p=0.044$).

The reported significant change of the leachates analysed with TOP was only of decreasing concentrations. This was seen in both A and E for the PFASs PFHxA ($p=0.009$, $p=0.014$), PFHpA ($p=0.041$, $p=0.003$) and PFDoDA ($p=0.004$, $p=0.010$) (Table 13). The significant decrease was also seen in A for PFBS ($p=0.032$), and in E for PFPeA ($p=0.004$), PFOA ($p=0.042$), 6:2 FTS ($p=0.002$) and PFNA ($p=0.044$).

5. Discussion

5.1 Part I: PFAS in Sewage Sludge

5.1.1 PFAS Occurrences and Limitations in Analytical Practices

Concentrations of quantified PFASs in this study are mostly comparable with results from other Swedish studies. Kärrman et al. (2019) and Eriksson et al. (2015) identified diPAPs as the dominant PFAS group in Swedish sewage sludge, with 6:2 diPAP being the most abundant. In contrast, this study where only 8:2 diPAP was included, found relatively low concentrations. However, the 8:2 diPAP concentrations reported by Kärrman et al. (2019) and Eriksson et al. (2015), align with those observed in this study. The analysis highlights that including only 8:2 diPAP does not provide an adequate representation of the total diPAP content in sewage sludge.

The PFAS detected at higher concentrations in the solid samples – such as PFOS, 10:2 FTS, and N-EtFOSAA – were all long-chained, which is consistent with findings from previous studies (Eriksson et al., 2015; Kärrman et al., 2019). The elevated levels of PFOS may reflect its historical use in firefighting foams prior to its ban. Contributions may also come from transformations of PFOS precursors. N-EtFOSAA, a known PFOS precursor with a similarly strong affinity for solids, likely partitions into the sludge in the same manner. The presence of 10:2 FTS, a more recent component in firefighting foams, suggests that current or legacy use may be a source of its accumulation in sewage sludge (Kärrman et al., 2011). 10:2 FTS, the longest FTS compound analysed in this study, further illustrates the positive correlation between chain length and particle affinity.

The results of this study show higher concentrations of conventional PFASs compared to the novel compounds analysed with target analysis, such as GenX and ANOVA. This trend was also observed in the two referenced studies (Kärrman et al., 2019; Eriksson et al., 2015). These findings highlight the persistence of PFASs and the potential transformation of precursors into these compounds. However, the analytical scope for novel PFASs was limited across all studies, including this one, meaning that they may have gone undetected.

The results show that using a broader target list than PFAS 4 and PFAS 11 increased the detected total PFAS concentrations. This suggests that these limited, standardised sets may not be optimal for evaluating PFAS content in sewage sludge. For example, two of the most dominant compounds found in this study – N-EtFOSAA and 10:2 FTS – are not included in either PFAS 4 or PFAS 22. Using standardised target sets, like PFAS 4 and PFAS 22, can support comparability between studies and help forming regulatory guidelines. However, applying the same sets across different matrices, such as groundwater or sewage sludge, can be deceptive. The composition of PFAS contamination varies widely depending on the medium and its exposure. Thus, standardised sets may capture

very different proportions of the total PFAS present. This underlines the importance of context-specific target selection when assessing PFAS contamination.

Values falling below the reporting limit (RL) were included in the calculation of mean concentrations by assigning them a value equal to half the RL. While this approach allows for the inclusion of all replicates, it introduces uncertainty, as the true concentrations remain unknown. To improve accuracy in future studies, it is recommended to either utilize analytical methods with lower RLs or apply modelling techniques capable of handling censored data. Such methods could provide higher certainty evaluations of values below the detection limit.

Precursors and TOP Assay

Despite targeting fewer compounds (22 vs. 49), the TOP Assay measured nearly 500% higher total PFAS concentrations compared to the target analysis. A comparison with target analysis with the substances in PFAS 4 and PFAS 22 target sets revealed an even greater relative increase in total PFAS concentrations. These results align with previous studies, identifying precursors to be the major PFAS group in sewage sludge (Kärman et al. 2019).

Major concentration increase of short-chained PFASs such as PFBA, PFPeA and PFHxA, reflects on the shift of PFAS production and usage from long-chained PFAS to short-chained PFASs and precursors (NICNAS, 2015). The significant increase in PFPeA, PFHxA, PFHpA, and PFOA after oxidation, combined with studies identifying diPAPs as major PFASs in sewage sludge, suggests that these compounds may result from the transformation of 6:2 and 8:2 diPAPs, for which they are known terminal degradation products (Weidemann et al., 2024)

The results indicate that the TOP Assay provides a more comprehensive picture of both the total PFAS content, and the types of terminal transformation products present in sewage sludge compared to conventional target analyses, such as PFAS 4, PFAS 22, or even a broader 49-target screen. By inducing oxidation of precursors, the TOP Assay can simulate environmental transformation processes, such as those that occur when sewage sludge is applied to soil, thereby offering more realistic insight into potential PFAS exposure. A representative screening of PFAS using conventional target analysis acquires insight of the likely PFAS composition of the tested medium. TOP Assay can detect a wider spectrum of PFASs using fewer targets and less prior information about the sludge's PFAS profile.

However, the method has limitations. The TOP Assay is generally associated with higher analytical costs compared to conventional target analysis when assessing the same set of PFAS compounds and is limited to transform oxidisable precursors (SGS Analytics Sweden, 2025; Eurofins Sweden, 2025). The quality of the results is also affected by varying transformation efficiency, depending on precursor structure and reactivity. Additionally, it does not identify the original precursor compounds. This may make it less suitable for source-tracking applications, which can be important for wastewater treatment plants aiming to

manage PFAS inputs. Although the exact time frame for precursor transformation in the environment remains unclear, the likelihood of transformation in soil, aided by microbial activity and natural conditions, supports the environmental relevance of TOP Assay results. Still, careful consideration is needed when selecting which PFASs to target post-oxidation to ensure a representative outcome.

In summary, the findings suggest that the TOP Assay may be an appropriate tool for assessing PFAS contamination in sewage sludge, in combination with an extended target PFAS analysis package. Therefore, regulatory frameworks should be adapted to accommodate data generated through this method, rather than relying solely on conventional target analyses.

PFAS Mobility and Leachability

The results show that the majority of PFASs detected in leachates at the highest concentrations in both the TOP Assay and target analysis were short-chained, supporting the understanding that short-chain PFASs exhibit higher mobility (Milinovic et al., 2015). However, elevated concentrations of PFOA and PFOS were also observed in the leachate samples. This may be attributed to their relatively high levels in the solid phase, which increases the potential for leaching despite their longer chain lengths. The calculated organic carbon–water partition coefficients (K_{oc}) varied substantially across different sewage sludge samples, making it difficult to draw clear distinctions between individual PFAS compounds. Nevertheless, PFOS exhibited consistently higher K_{oc} values compared to most other PFASs. This supports a higher affinity of PFOS to organic matter, aligning with previous studies (Kookana et al., 2023).

The results provide an indication of which PFAS compounds may pose a risk of leaching when sewage sludge is applied to soil. However, it is not possible to draw direct conclusions about leaching behaviour under natural environmental conditions from these results. The liquid-to-solid ratio of 10 gives an indication of leaching during a couple of decades. In natural soils, the presence of air-filled pores creates additional sorption interfaces, as many PFAS compounds preferentially adsorb at air-water interfaces (Brusseau, 2019). This phenomenon can significantly reduce their mobility and retention in the soil matrix and is not accounted for in the LS 10 tests where water saturated conditions prevail.

To more accurately assess actual leaching risks under field conditions, field tests with accurate sewage sludge volumes are recommended.

Digestion

For untreated solid samples, the undigested sewage sludge (D-E) measured lower total PFAS concentrations compared to the digested sludge (A-C). Digestion may facilitate precursor transformation, resulting in higher amounts of terminal PFAS compounds in the digested SS. However, the TOP Assay results revealed higher total PFAS concentrations in the undigested sludge than in the digested sludge. This pattern suggests that a larger proportion of untransformed precursors remain in the undigested sludge. Upon oxidation during the TOP Assay-analysis, these

precursors are converted into terminal PFASs, resulting in a greater increase in PFAS concentrations in undigested sludge compared to digested sludge.

Anaerobic digestion reduces total organic carbon (TOC) during the process (Hanum et al., 2019), which is also seen in this study. Due to PFAS affinity to organic matter, lower TOC may result in fewer available sorption sites for PFAS. This is supported by the higher K_{oc} values observed for most PFASs in undigested sludge (with higher TOC), indicating a stronger sorption affinity to undigested sludge compared to digested sludge. Consequently, digested sludge may either accumulate fewer PFASs during the wastewater treatment process or experience greater PFAS leaching during post-treatment storage and sanitation. This may explain why the undigested sludge exhibited higher total PFAS concentrations following TOP Assay analysis compared to the digested sludge. However, further analysis, including the PFASs that this study did not target, is needed to fully prove this theory. The undigested sludges are produced from WWTPS with lower capacities than the digested sludges. There might be other processes, unknown to this study, beyond digestion that have created these differences.

Implications for Land Application of Sewage Sludge

As previous studies, these results confirm that the tested sewage sludges contain PFAS, indicating that land application of sludge can introduce PFAS into soils. While concentrations of PFAS 4 and PFAS 22 were below the REVAQ guideline values when assessed using conventional target analysis, results from the TOP Assay exceeded these limits (Figure 8). This suggests that standard testing methods may underestimate the total PFAS load introduced to soil through sludge application.

Previous long-term field studies have demonstrated PFAS accumulation in soil following repeated sludge applications, although they did not determine uptake in wheat crops (Kärrman et al. 2024). It is important to note that these studies applied higher sludge quantities than typical farmland applications, potentially overestimating soil concentrations. Further research is needed to assess the impact of realistic sludge application rates, both for agricultural use and in land restoration contexts. As for land restoration areas, the research is particularly sparse.

This study also highlights the importance of effective sewage sludge treatment in reducing the amount of PFAS introduced into the environment through land application. As the composition of wastewater – and thus sewage sludge – mirrors the products and industrial processes used in society, upstream measures are critical for controlling PFAS pollution. Many PFAS precursors have been introduced as substitutes for legacy compounds following regulatory restrictions. However, emerging research indicates that these precursors may also exhibit persistence, bioaccumulation, and toxicity (Miranda et al. 2021). Mitigation of PFAS at its source, by expanding regulations to target both established PFASs and their precursors, as well as incorporating these as targets in investigations, could provide safer and sustainable recycling of materials, such as sewage sludge.

5.2 Part II: Incubation of Sewage Sludge and Soil

The low significance of changes observed in solid samples analysed with target analysis suggests that the incubation period led to minimal detectable differences in PFAS concentrations using this method. However, when analysed using the TOP Assay, a statistically significant decrease in the concentrations of PFNA, PFDoDA, PFOA, PFDA, and PFUnDA was observed, alongside a significant increase in PFBS and PFOS.

Given that these compounds are terminal perfluoroalkyl acids (PFAAs), which are known for their high persistence and resistance to environmental degradation (Cousins et al., 2020), the observed changes are unlikely to result from natural transformation processes during the incubation period. If the observed decrease were of precursors, the shifts could have indicated partial degradation of precursors (not targeted in any of the used methods) during the incubation, that then facilitated the oxidation-transformation in the TOP Assay. However, since the compounds in question are not precursors and do not undergo natural transformation, the observed variations are more plausibly explained by analytical factors. These may include adsorption to container walls or soil particles that reduced extraction efficiency, or inherent sample heterogeneity affecting reproducibility. Further investigation would be needed to confirm the exact causes of these changes.

For the leachate samples, target analysis revealed few statistically significant changes over the incubation period. However, Figure 13 illustrates trends toward increasing concentrations for some PFAS compounds. This may be attributed to the breakdown of organic matter in the sludge-soil mixture, potentially resulting in fewer available sorption sites for PFAS, thereby increasing their mobility and leaching potential. Leachates analysed with the TOP Assay showed significant decreases in certain PFAS concentrations. However, this study cannot isolate a specific factor responsible for these observations. Precursor transformation may influence mobility by altering molecular structure; for example, transformation can result in shorter-chain compounds, which are generally more mobile.

In summary, although some changes in PFAS concentrations were observed during the one-month incubation, the results are inconclusive regarding the underlying mechanisms. The study duration and analytical limitations prevent any definitive conclusions about the lifetimes of precursors.

5.3 Conclusions

This study found PFOS, 10:2 FTS and N-EtFOSAA (Using 49 targets) and PFHpA and PFBA (using TOP Assay) as the main PFAS contaminants in the five sewage sludges. Concentrations were comparable to other studies (e.g. PFOS at 3.3-7.3 ng/g TS). When assessing leachabilities, some patterns in PFAS distribution were observed among PFASs with higher concentrations, for example

PFOS. However, due to the similar distribution coefficients (K_d) and organic carbon partition coefficients (K_{oc}) among many of the lower-concentration PFAS compounds, no definitive trends could be established among them. For future studies, column leaching test is suggested as a tool for investigating leaching behaviours in a more realistic soil setting and estimate influence of soluble PFAS on ground water quality (Kalbe et al., 2014).

No transformation of precursors was observed during the four-week incubation period. It is however not possible to determine that no transformation happened from the results. In conclusion, the analytical methods applied in this study were sufficient to address the core research questions and objectives of the project.

Additionally, the selected PFAS compounds were successfully detected and quantified using both target analysis (49 compounds) and the TOP assay (22 compounds) with acceptable measurement uncertainty. However, in several cases, the reporting limits (RL) were close to the measured concentrations. This may present a limitation in contexts where more precise quantification is required. The reported concentrations of this study were comparable to concentrations reported in previous Swedish studies, indicating that this study provides a good representation of PFAS in Swedish sewage sludge and that the samples were unlikely disturbed by contamination. It shows that the sampling method is sufficient, despite the low representation of the total batch.

However, the PFASs included in both this study and in previous are limited. Future research and PFAS screening of Swedish sewage sludge, focusing more on novel PFASs, could improve knowledge on what targets are relevant for a representative sewage sludge analysis.

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Popular science summary

Per- and polyfluoroalkyl substances (PFAS) are synthetic chemicals valued for their water, grease, and dirt-repellent properties, but many pose environmental and health risks due to their extreme persistence. In Sweden, about 85% of sewage sludge from wastewater treatment is applied to farmland. While it adds nutrients and organic matter, sludge also contains pollutants like PFAS, due to its use in society.

PFAS research and regulation have mainly targeted well-known, persistent compounds like PFOS and PFOA. However, many newer PFAS are precursors, substances that can degrade into persistent PFASs. Studies show precursors can make up a large portion of PFAS in sludge, yet they are often excluded from analysis and regulation, risking underestimation of total PFAS levels in land-applied sludge.

This thesis explored the presence of established PFAS and precursors in Swedish sewage sludge and assessed the limitations of current analytical methods. Sludge from five treatment plants, both digested and untreated, was analysed using two methods: *Target Analysis*, which detects and quantifies a set number of known PFAS (49 in this study), and *TOP Assay*, which oxidizes precursors in the sample, converting them into measurable PFAS acids (22 analysed here). Comparing results before and after oxidation estimates the precursor content.

The study confirms that Swedish sewage sludge contains significant amounts of PFAS, much of which are precursors not captured by current standard analyses or regulations. When sludge is applied to farmland or used in land restoration, the total PFAS load added to the environment is therefore higher than previously measured. These precursors can transform into persistent and potentially harmful PFAS acids. In summary, using sludge as soil amendment introduces a substantial, largely overlooked PFAS burden to the land. It is therefore recommended to include TOP Assay or more substances for target analysis when investigating PFAS in sludge.

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