



Screening of per- and polyfluoroalkyl substances (PFAS) in sediment and water close to paper industries

Screening av per- och polyfluoralkylsubstanser (PFAS) i sediment och vatten i närheten av pappersindustrier

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Screening of per- and polyfluoroalkyl substances (PFAS) in sediment and water close to paper industries

Screening av per- och polyfluoralkyl substanser (PFAS) i sediment och vatten i närheten av pappersindustrier

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Sammanfattning Per- och polyfluoralkylsubstanser (PFAS) är en stor grupp antropogena föreningar med bred användning, till exempel vid tillverkning av fett- och vattenavvisande papper. Flera PFAS har visat sig utgöra en risk för miljön och människor på grund av deras persistens, rörlighet, toxicitet och bioackumulerande egenskaper. Pappersproduktionen i Sverige omfattar olika typer av produkter såsom kartong, flytande kartong, skrivpapper, säckpapper, hygienpapper, etcetera. Nuvarande och historisk användning av PFAS i svensk pappersindustri kopplat till utsläpp till miljön är inte känd. Syftet med denna studie var att bedöma om pappersindustrier kan vara en viktig punktkälla för PFAS i miljön genom analys av PFAS i sediment och vatten i närheten av pappersindustrier. Totalt ingick 60 sedimentprover från 13 platser i Sverige provtagna under tidsperioden 2015–2022, och åtta ytvattenprover från tre platser tagna 2021. Analysen omfattade totalt 68 PFAS (icke-polymerer) och extraherbart organiskt fluor (EOF). Resultaten visar ett stort koncentrationsområde i sediment nära pappersindustrier i Sverige, från under detektionsgränsen (LOD) till 1181 ng/g torrsvikt. Antalet och typen av detekterade PFAS varierade också. De PFAS-klasser som hittades i högst koncentration i ytsediment var polyfluoralkylfosfatdiestrar (diPAP, max 819 ng/g), fluoroktansulfonamidättisyra (FOSAA, max 137 ng/g) och ämnet N-etylperfluoroktansulfonamidfosfatdiester (diSAmPAP, max 134 ng/g d). Koncentrationerna för diSAmPAP är semi-kvantifierade då matris och isomerer störde kvantifieringen. En generell ökning av koncentrationerna med ökande sedimentdjup kunde ses och den maximala koncentrationen i sediment från olika djup nådde 1084 ng/g för diPAP, 65 ng/g för FOSAA och 682 ng/g för diSAmPAP. De klasserna som vanligtvis mäts i undersökningar, PFCA och PFSA, nådde maximalt 6 och 7 ng/g i ytsediment, och 11 och 6 ng/g i sedimentkärnor. EOF följde samma trend som PFAS-koncentrationerna, med några få undantag, och varierade mellan <LOD till 1301 ng/g. Ytvatten från tre platser visade detekterbara koncentrationer av PFCA, PFSA och FTSA upp till 0,05 ng/ml, men kunde inte kopplas till	

pappersindustrierna i denna studie. Baserat på PFAS-analysen av sediment är åtta av de 13 platserna tydligt påverkade av punktkälla och fem kan klassificeras som lågkontaminerade (summa PFAS-68 <20 ng/g) även om påverkan från närliggande industrier inte helt kan uteslutas.

Summary

Per- and polyfluoroalkyl substances (PFAS) is a large group of anthropogenic compounds that is widely used, for example to manufacture grease- and water-repellent paper. Several PFAS have been found to pose a risk to the environment and humans due to their persistency, mobility, toxicity and bioaccumulative potential. Paper production in Sweden covers widely different products such as cardboard, liquid cardboard, writing paper, sack paper, hygiene paper (tissue), etc. In addition, there are also numerous closed paper industries. Current and historical use of PFAS in paper industries in Sweden and consequent emissions to the environment are not known. The purpose of this study was to assess if paper industries could be an important point source for PFAS in the environment by analysis of PFAS in sediment and water taken nearby paper industries. In total, 60 sediment samples from 13 sites in Sweden sampled during the time period 2015-2022, and eight surface water samples from three sites taken in 2021, were included in the study. A total of 68 non-polymer PFAS and extractable organofluorine (EOF) was targeted. The results from the screening study shows a wide range of the sum target PFAS-68, between below limit of detection (LOD) to 1181 ng/g dry weight (d.w.) in sediments close to paper industries in Sweden. The number and type of PFAS detected also varied. The PFAS-classes found in highest concentration in surface sediments were polyfluoroalkyl phosphate diesters (diPAP) (maximum 819 ng/g d.w.), fluorooctanesulfonamidoacetic acid (FOSAA) (maximum 137 ng/g d.w.), and N-ethyl perfluorooctane sulfonamido ethyl phosphate diester (diSAmPAP) (maximum 134 ng/g d.w.). DiSAmPAP was only semi-quantified due to poor separation of structural isomers and interferences from the sediment matrix. A general increase in concentrations with increasing sediment depth could be seen for the contaminated sites, and the maximum concentration in sediments of different depths reached 1084 ng/g d.w. for diPAP, 65 ng/g d.w. for FOSAA, and 682 ng/g d.w. for diSAmPAP. The commonly measured classes of perfluoroalkyl acids (PFCA) and perfluoroalkyl sulfonic acids (PFSA) reached a maximum of 6 and 7 ng/g d.w. in surface sediments, and 11 and 6 ng/g d.w. in sediment cores, respectively. The EOF levels followed the same trend as target PFAS concentrations, with a few exceptions, and ranged between <LOD to 1301 ng/g d.w.. Surface water from three sites showed detectable concentrations of PFCA, PFSA och FTSA up to 0.05 ng/mL, but the occurrence could not be connected to the paper industries. Based on the target results of surface sediments, five out of the 13 sites can be classified as low contaminated (sum target PFAS-68 <20 ng/g d.w.) and the remaining eight sites are clearly affected by a PFAS point source.

1. Introduction

Per- and polyfluoroalkyl substances (PFAS) have unique physicochemical properties such as stability, water-, oil- and dust repellence (KemI, 2015) and are used in a wide range of commercial and industrial applications. Several PFAS have been found to be persistent, mobile, toxic and bioaccumulative and therefore pose a risk to the environment and humans (Silva et al., 2021; Fenton et al., 2021).

PFAS are used in numerous industrial processes, in high-temperature applications, as surfactants, to provide non-friction surfaces, and water- and oil-repellency. In the paper industry PFAS are used to produce grease- and water-repellent products. Paper products that might contain PFAS includes food packages (plates, popcorn bags and pizza cartons) and other packaging materials such as cartons, containers and masking paper. According to government registers and other inventory lists there are numerous PFAS used in the global paper industry market including polymers/polymer raw materials, mainly polyfluorinated/perfluorinated (meth)acryl polymers and monomers, and poly/perfluorinated alkyl thiols, poly/perfluorinated alkyl sulfonamide derivatives, and poly/perfluorinated alkyl phosphorus compounds (KemI 2015). A shift in the general production of PFAS has occurred to more short-chain alternatives, for example 6:2 fluorotelomers, perfluorobutane sulfonyl fluoride (PBSF)-based PFAS, and perfluoropolyethers (PFPE), which are alternative process chemicals for fluoropolymer production (Wang et al. 2013).

Studies on environmental emissions as a result of paper manufacturing are scarce. Recent screening studies from Norway reported high levels of PFAS in sediment nearby a closed paper industry (Norwegian Environment Agency, 2019; Langberg et al., 2019). There are currently 49 active pulp and paper mills in Sweden. The paper production consists of widely different products such as cardboard, liquid cardboard, writing paper, sack paper, hygiene paper (tissue), etc. In addition, there are also a number of closed paper industries. Current and historical use of PFAS in Swedish paper industries are not known and there is no information regarding environmental occurrence of PFAS due to emissions from the paper industries. The purpose of this study was to assess if paper industries could be an important point source for PFAS to the environment in Sweden by analysis of PFAS in sediment and water taken nearby paper industries. Since possible target PFAS are numerous, and analytical standards are partly lacking, the assessment was done by combining target analysis of selected PFAS with analysis of extractable organofluorine (EOF).

2. Material and methods

2.1 Target analytes and chemicals

A total of 68 non-polymer PFAS (referred to as PFAS-68) were targeted in the study, divided into the subgroups perfluoroalkyl acids (PFCAs, n=15), perfluoroalkyl sulfonic acids (PFSAs, n=11), perfluoroalkylsulfonamides (FASA, n=7), fluorooctanesulfonamidoacetic acids (FOSAA, n=3), polyfluorotelomer sulfonic acids (FTSA, n=6), polyfluoroalkyl phosphate diesters (diPAP, n=8), perfluoroether acids/sulfonic acids (PFECA/PFESA, n=4), N-ethyl perfluorooctane sulfonamido ethyl phosphate diester (diSAmPAP), perfluoroethylcyclohexane sulfonic acid (PFECHS), perfluoroalkyl phosphonic acid (PFPA, n=3), perfluoroalkyl phosphinic acid (PFPiA, n=3), fluorotelomer acids (FTCA, n=3), and fluorotelomer unsaturated acids (FTUCA, n=3). For full list of target analytes, see Appendix A. In addition, EOF was targeted. Native and labeled standards were from Wellington Laboratories (Guelph, Canada).

2.2 Samples

Sediment core samples were taken during 2021-2022 and others were retrieved from frozen archives. In total, 60 samples from 13 sites in Sweden sampled during the time period 2015-2022 are presented here. The samples originated from both fresh water and marine recipients located outside active paper factories and from different sediment depths. Since most samples were selected from left-over samples from previous projects, the core slice measures (sampling depths) differ. Detailed information of the samples is found in Appendix B. Sediment was stored at – 20 °C and was freeze-dried prior to extraction. Surface water (n=8) was taken in 2021 from three sites (Appendix C), and was stored in polypropylene containers at – 20 °C.

2.3 Sample preparation

2.3.1 Sediment

All samples were extracted in duplicates, where the first replicate was intended for target PFAS analysis and was subsequently fortified with isotopically labelled internal standards (IS). The second replicate was analysed for EOF and was therefore not fortified with IS since the labelled standards will also contribute to EOF in the sample. Approximately 0.5 g freeze-dried sediment was treated with alkaline methanol (0.2 M) before solid-liquid extraction with methanol using three consecutive portions and a total of 10 mL methanol. The extract solution was neutralized by addition of formic acid. Clean-up was performed using ENVI-carb (100 mg, Supelco) and weak anion exchange (300 mg Oasis WAX, Waters Corporation, Milford, USA) sorbents. Extra washing steps were applied during the WAX clean-up (5 mL 0.01% aqueous ammonium hydroxide, 3 mL laboratory produced ultra-pure water, 2 mL aqueous ammonium acetate buffer (pH 4) and 2 mL 20% methanol in ultra-pure water) to remove water soluble interferences, and specifically inorganic fluoride. Elution was done with 4 mL 0.1% NH₄OH/methanol, that was evaporated to a final volume of 500 µL

2.3.2 Surface water

All samples were extracted in duplicates, in the same way as for sediment samples to allow for target PFAS analysis and a separate EOF analysis with no contribution from isotopic labeled standards. Unfiltered surface water (500 mL) was extracted using solid phase extraction (SPE) with Oasis WAX cartridges (Waters Corporation, Milford, USA 500 mg, 6 mL). In brief, the samples were adjusted with formic acid to pH 4 and loaded on SPE cartridges, that were subsequently washed with 20 mL 0.01% aqueous ammonium hydroxide, 10 mL laboratory produced ultra-pure water, 10 mL aqueous ammonium acetate buffer (pH 4) and 10 mL 20% methanol in ultra-pure water. Elution was done with 10 mL 0.1% NH₄OH/methanol, that was evaporated to a final volume of 200 µL.

2.4 Instrumental analysis and quality control

2.4.1 Target PFAS

Targeted analysis was carried out on an Acquity UPLC coupled to a Xevo TQ-S triple quadrupole mass spectrometer, equipped with a BEH C18 column (1.7 µm, 100 × 2.1 mm). Confirmatory analysis and semi-quantification of diSAmPAP was done on an Acquity UPLC coupled to a Waters G2-XS-QToF instrument. All instrumentations and columns were from Waters Corporation, Milford, Massachusetts, USA. The mobile phase consisted of 2 mM ammonium acetate in water and methanol. For the PAP analysis 5 mM 1-methylpiperidine was used as an additional additive. An isolator column was inserted after the solvent mixer before the injector to separate any potential contamination from the UPLC system from the injected sample. The system was operated in negative electrospray ionization (ESI-) mode. The source and desolvation temperatures were set at 150 °C and 400 °C, respectively. The desolvation and cone gas flows (nitrogen) were set at 800 L/h and 150 L/h, respectively. The capillary voltage was set at 0.7 kV. Some modifications were done for the PAP analysis; desolvation temperatures and capillary voltage were set at 200 °C and 2.9 kV, respectively. Quantification was performed using labeled internal standards and minimum a four-point linear calibration curve, that ranged from 0.02 to 50 ng/mL, depending on the analyte. Each batch of samples contained one or several procedural blanks and a fortified sample. The procedural blank was used for calculating limit of detection, together with the lowest concentration standard in the calibration curve. Recovery of all analytes were monitored in the fortified sample, together with the internal standard recoveries.

2.4.2 EOF

A combustion ion chromatography (CIC) system with a combustion module from Analytik Jena, (Germany), and an ion chromatograph from Metrohm (Switzerland) was used to quantify extractable organofluorine (EOF). The anions were separated with an ion exchange column (Metrosep A Supp 5–150/4), carbonate buffer (64 mmol/L sodium carbonate and 20 mmol/L sodium bicarbonate) as eluent and isocratic elution. The autosampler injected 100 µL of the extract on a quartz boat. The boat was inserted into the oven (1000–1050 °C) under a flow of oxygen (300 mL/min), argon (100 mL/min), and argon mixed with water vapor (100 mL/min) under hydrolytic conditions monitored by a flame sensor followed by 2 minutes of post-combustion

time with the flow of oxygen (400 mL) only. The hydrogen fluoride (HF) formed during combustion was absorbed in ultrapure water (in the absorber module). The F^- concentration was measured via conductivity.

The concentration of EOF is expressed in ng/g (sediment) or ng/mL (water) and represents the organofluorine content. The sample treatment methods ability to remove inorganic fluoride has been discussed elsewhere (Kärman et al. 2021). A five-point calibration curve of 50-1000 $\mu\text{g/L}$ using PFOA as standard was used for quantification. Empty boat combustions were made before and after each sample and the average background was subtracted from the sample signal. Procedural blanks were run for monitoring background contamination.

3. Results and discussion

PFAS included in the classes PFPA, PFPiA and PFECA/PFESA could not be detected in any of the samples, nor was PFECHS detected. Concentrations (ng/g d.w.) of the other PFAS classes and EOF are presented here and are given in Appendix D and E.

3.1 Surface sediments

The results from the screening study shows a wide range of the sum target PFAS-68, between <LOD to 1181 ng/g dry weight (d.w.) in sediments close to paper industries in Sweden. Figure 1 shows an overview of the sum PFAS-67 (excluding diSAmPAP) in all surface sediments from the 13 sites presented in this study and corresponding concentrations are presented in Appendix D. The homologue distribution of detected PFAS also varied between sites (Figure 1). It should be noted that the samples included in the group “surface sediments” comprises different depths and interpretation should therefore be done carefully.

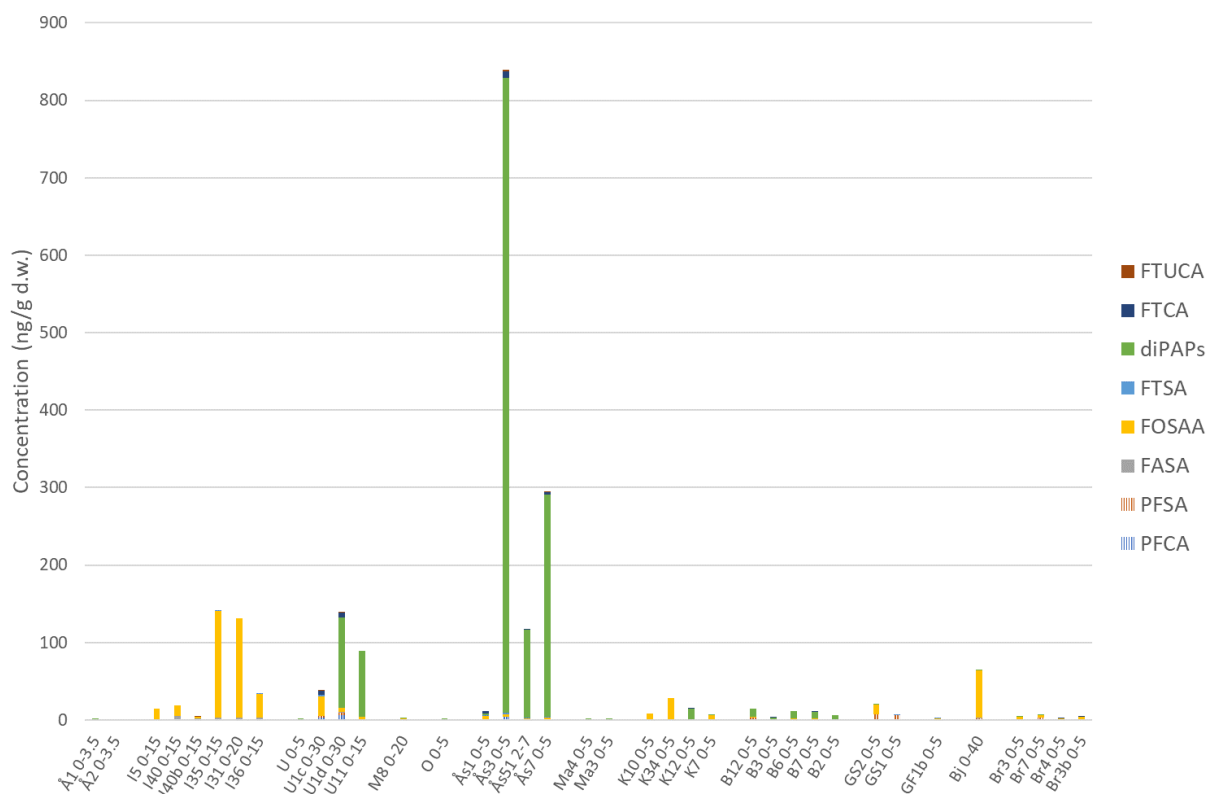


Figure 1. Concentrations (ng/g dry weight) of sum PFAS-67 and the contribution of different PFAS classes in surface sediments. The range given in the name corresponds to the sediment depth (cm). For sample information, see Appendix B.

The most commonly measured classes PFCA and PFSA reached a maximum of 6 and 7 ng/g d.w. in surface sediments, respectively. The PFAS-classes found in highest concentration in surface

sediments were diPAP (maximum 819 ng/g d.w.), FOSAA (maximum 137 ng/g d.w.), and diSAmPAP (maximum 134 ng/g d.w.). Reported diSAmPAP (Figure 2) could only be semi-quantified using high resolution mass spectrometry due to difficulties with interferences and presence of structural isomers. Five sites show lower levels compared to the others and can be classified as low contaminated (sum target PFAS-68 <20 ng/g d.w.) namely Ångermanälven (Å), Ortviken (O), Mariestadsfjärden (Ma4, Ma3), Glan Fiskeby (GF), and Bråviken (Br) (see Appendix F for enlarged figures). It can not be ruled out that these sites are not affected by nearby industries, especially since some show a pattern with diPAP and diSAmPAP that are similar to the highly contaminated sites.

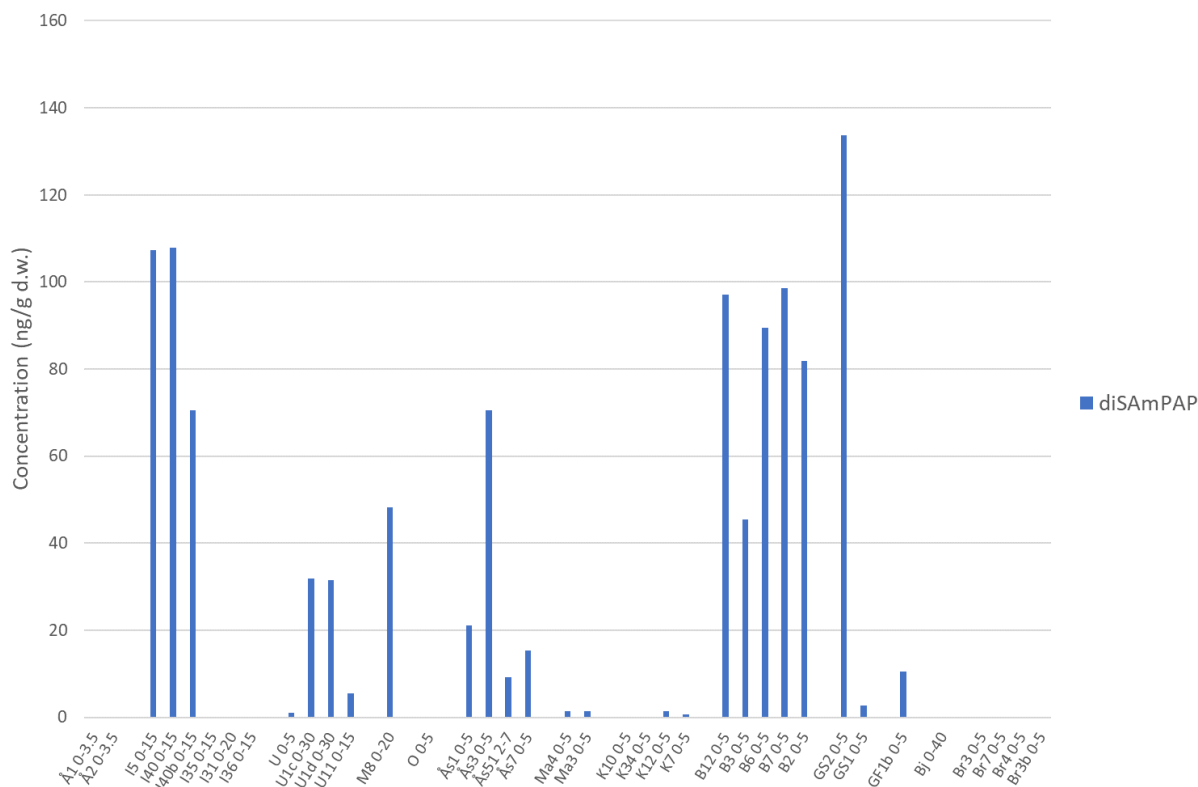


Figure 2. Semi-quantified concentrations (ng/g dry weight) of diSAmPAP in surface sediments. The range given in the name corresponds to the sediment depth (cm). For sample information, see Appendix B.

EOF reporting limits were based on procedural blank levels and the organofluorine background contamination varied between 116 and 466 ng/g for the surface sediment analysis. The samples with the highest target PFAS-68 concentrations also showed highest EOF concentrations (Iggesund (I), Umeälven (U), Åsfjorden (Å)). Most other detected concentrations were close to the reporting limit (~400 ng/g). The sample from Munksund (M8) deviated with an EOF value of

632 ng/g d.w. and PFAS-68 of 52 ng/g d.w., the latter consisting of mostly semi-quantified diSAmPAP (48 ng/g).

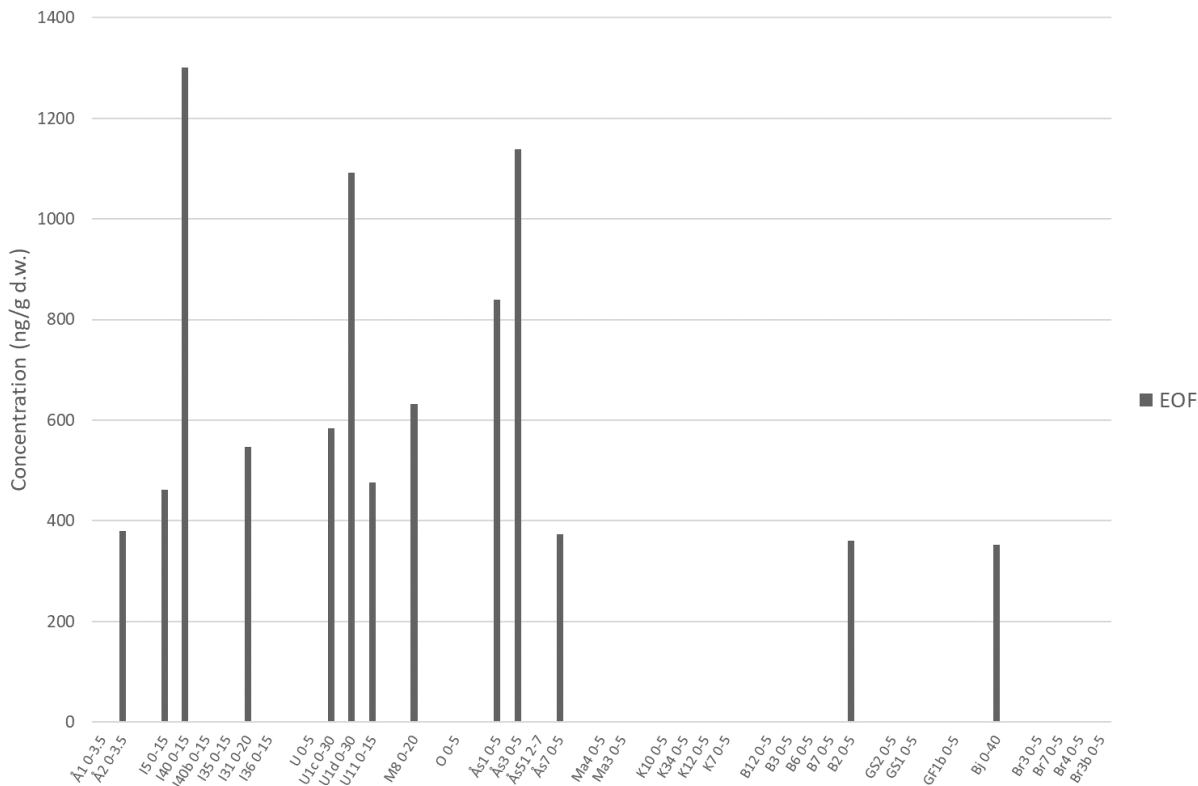
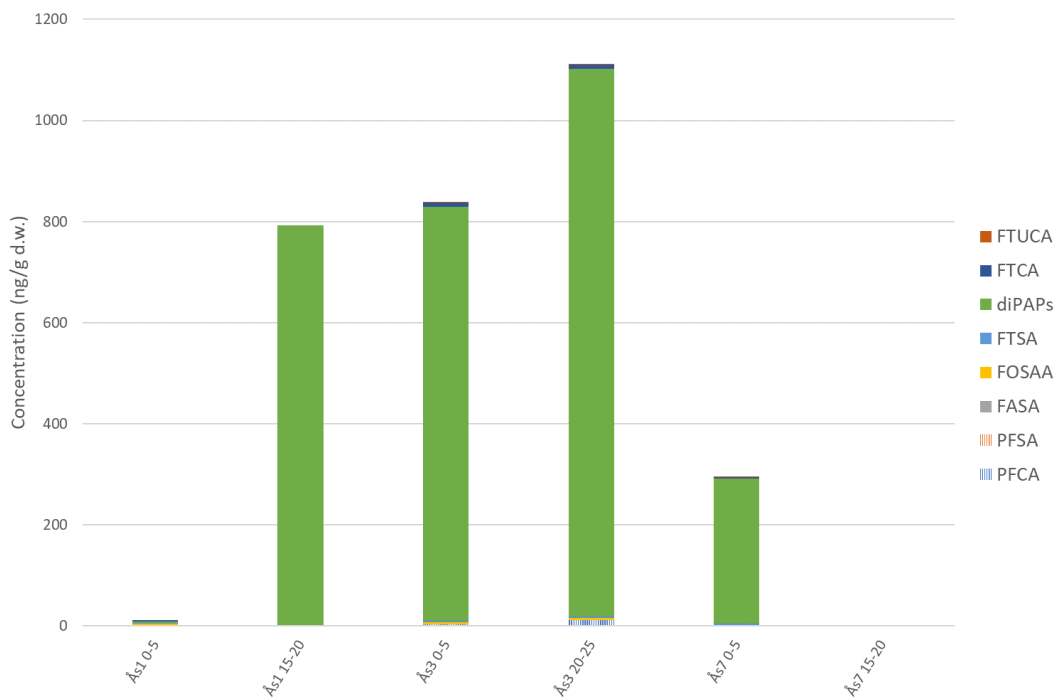


Figure 3. Concentrations (ng/g dry weight) of EOF in surface sediments. The range given in the name corresponds to the sediment depth (cm). For sample information, see Appendix B.

3.2 Sediment cores

The influence of depth on PFAS concentrations is displayed in Figure 4 for the sediment core sections that were available in the current study. All together, 13 sediment core profiles with varying depths and section lengths, from eight sites were analyzed. The PFAS-classes found in highest concentration in sediment profiles were diPAP (maximum 1084 ng/g d.w.), FOSAA (maximum 65 ng/g d.w.), and diSAmPAP (maximum 682 ng/g d.w.) (Figures 4-5). An increase in concentrations with increasing sediment depth could be seen for the contaminated sites, suggesting a higher historical emission of PFAS. None of the five sites that from the surface sediments could be classified as low contaminated showed elevated depth concentrations of target PFAS (see Appendix F for enlarged figures). No clear trend of change in PFAS classes over time, based on concentrations in sediment from different depths, could be seen.

A



B

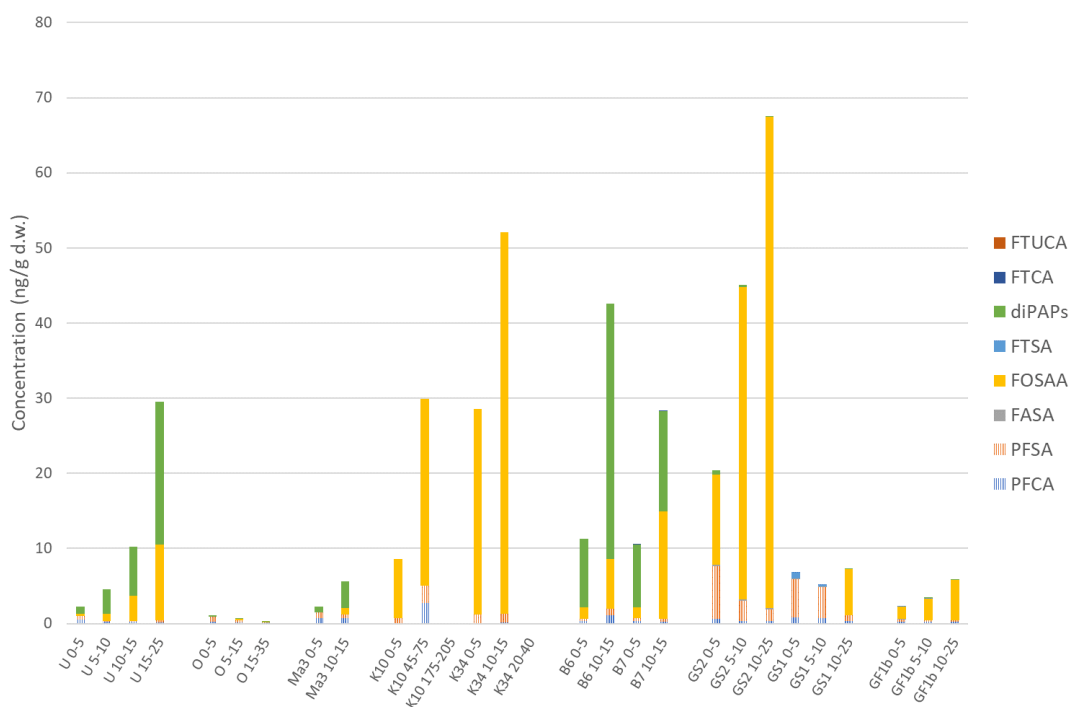


Figure 4. Concentrations (ng/g dry weight) of sum PFAS-67 and the contribution of different PFAS classes in core sediment profiles, divided in two graphs (A, B). The range given in the name corresponds to the sediment depth (cm). For sample information, see Appendix B.

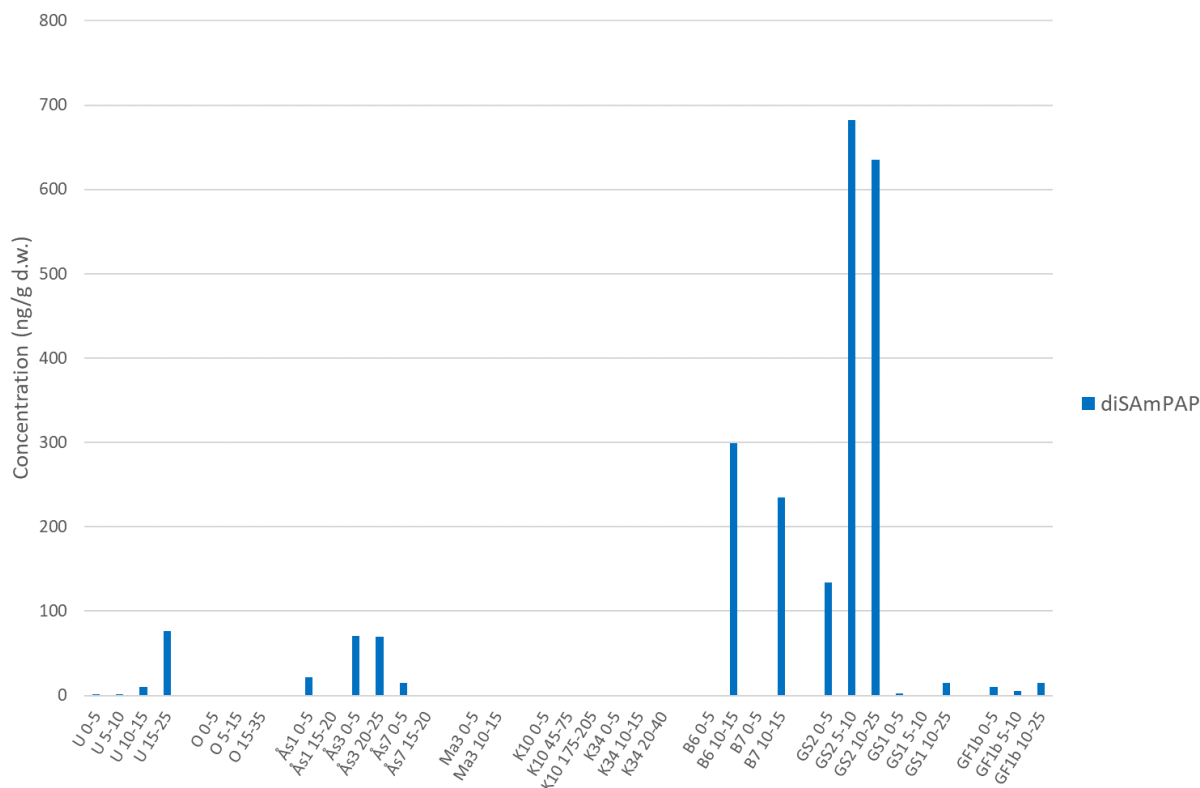


Figure 5. Semi-quantified concentrations (ng/g dry weight) of diSAmPAP in core sediment profiles. The range given in the name corresponds to the sediment depth (cm). For sample information, see Appendix B.

The EOF background contamination resulted in reporting limits for core sediment samples between 211 and 408 ng/g. EOF only partly followed the same trend as target PFAS concentrations (Figure 6). Three deep sediment samples showed low levels of EOF (217-469 ng/g d.w.) even though relatively low target PFAS levels were measured (Ortviken (O) 15-35 cm, Kattfjorden (K34) 20-40 cm, Byviken (B6) 10-15 cm). EOF in the top layer (0-5 cm) of site Åsfjorden 1 (Ås1) had higher EOF compared to the deeper layer (15-20 cm) even though the target PFAS results showed the opposite. The identity of EOF is not known and could originate from a large variety of chemicals containing fluorine, including inorganic fluoride that is not removed during the clean-up. However, another explanation to the latter observation could be inhomogeneity of the sediment sample.

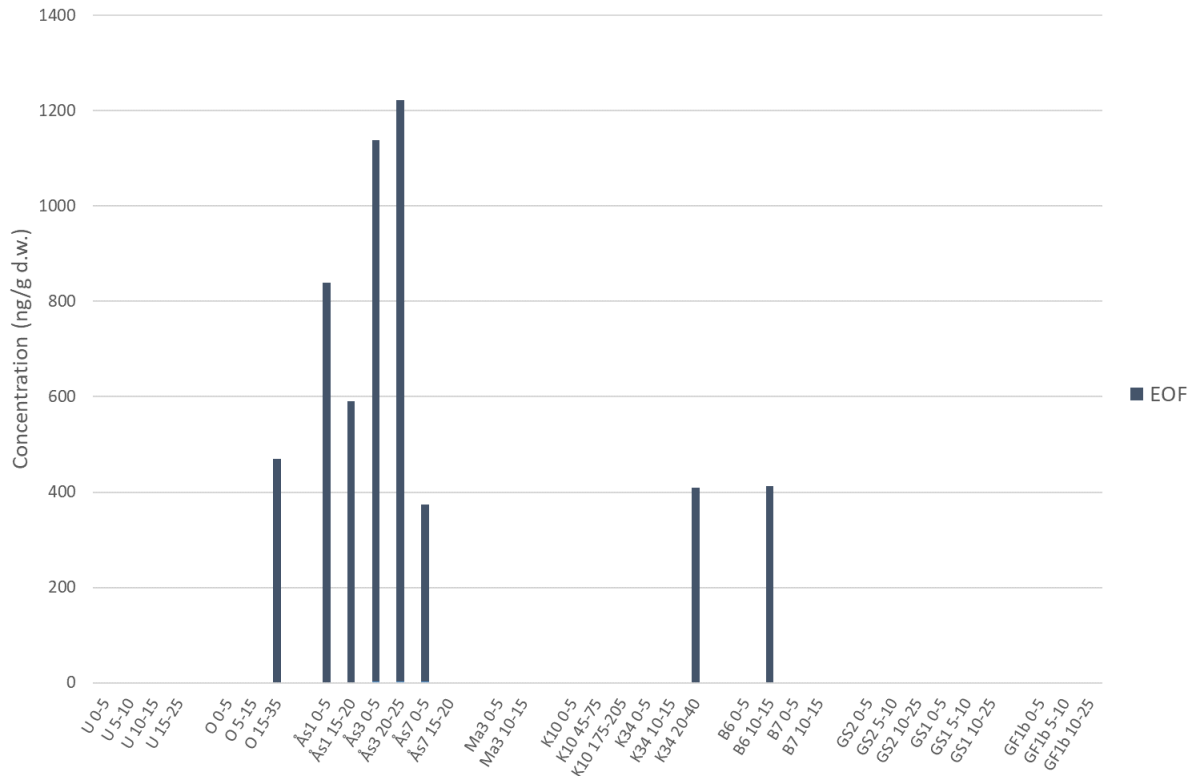


Figure 6. Concentrations (ng/g dry weight) of EOF in core sediment profiles. The range given in the name corresponds to the sediment depth (cm). For sample information, see Appendix B.

3.2 Surface water

Many PFAS are water soluble and surface water samples were taken from three sites in order to assess possible current emissions or influence from ongoing activities. All three sites showed detectable concentrations of PFCA, PFSA och FTSA (Figure 7) up to 0.05 ng/mL (Appendix D). Sum of target PFAS at site Glan Skärblacka was similar upstream and downstream the industrialized area which indicates no influence from the industry. There was a clear difference between up- and downstream at site Glan Fiskeby, with increasing concentrations of foremost 6:2 FTSA and PFHxA. It is not clear if there is an association between those two PFAS and the paper industry at the site. One likely explanation could be that there are other sources contributing to the surface water levels, for example activities involving firefighting foams. Finally, there was a clear difference between surface water concentrations at Bråviken, with higher concentrations away from the industry area compared to close to the emission outlets. The samples with higher concentrations consisted of PFCA but also PFSA (PFHxS, PFPeS, PFOS) summing up to 0.04 ng/mL.

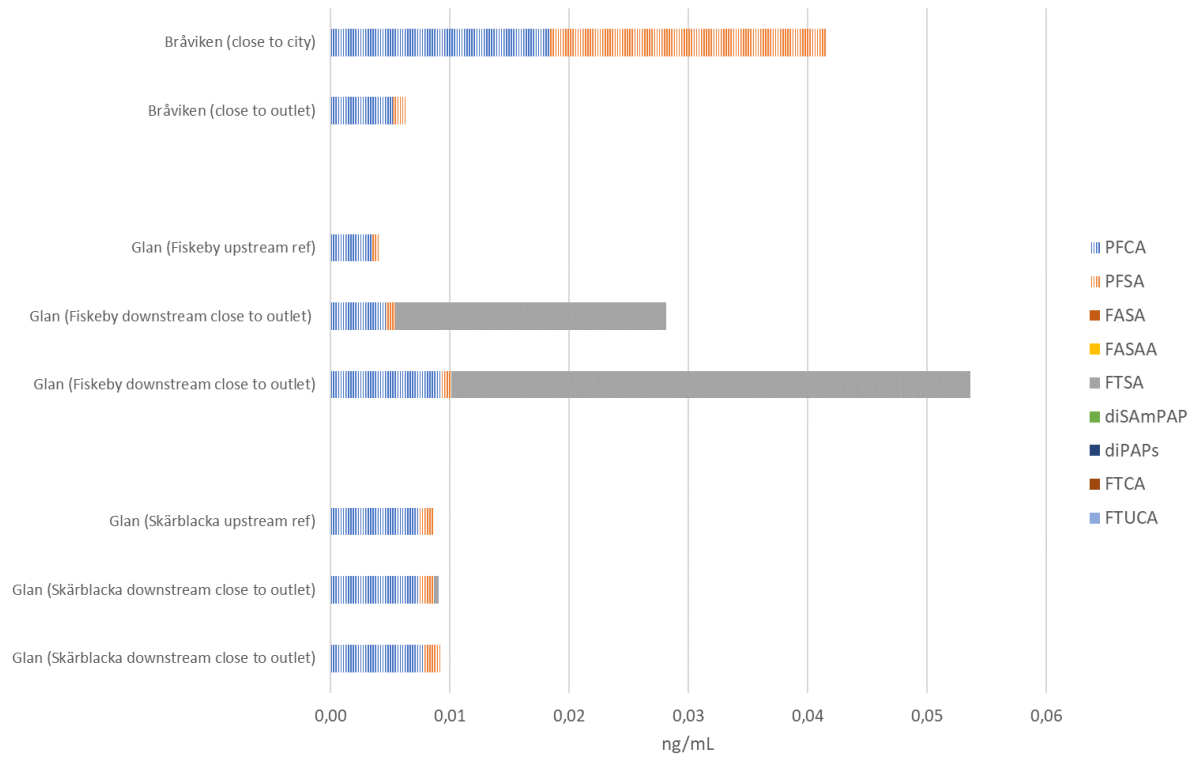


Figure 7. Concentrations (ng/mL) of PFAS in surface water. For sample information, see Appendix C.

4. Conclusion

All sediments included in the study showed PFAS pollution. Samples from 13 sites close to paper industries in Sweden showed that eight sites are clearly more affected by PFAS compared to the other remaining five sites. This difference between sites can be seen by measurement of mainly three classes of PFAS; diPAP, FOSAA, and diSAmPAP. The sites in current study showing elevated PFAS concentrations are in the same range as the recipient lake sediment concentrations outside a closed paper factory (6.1-207 $\mu\text{g}/\text{kg}$, sum PFAS-29) and the river sediments from the factory area (2450 $\mu\text{g}/\text{kg}$) in Norway (Langberg et al., 2021).

The PFAS profile in sediments disclose different types of emissions. The sites with the lowest PFAS concentrations were dominated by PFOS and long-chain PFCAs. Precursor compounds, especially diPAP, FOSAA and diSAmPAP, dominated the homologue profile for all affected studied sites in Sweden, which indicates that there is a point source nearby those sample sites. Both FOSAA and diSAmPAP belongs to precursors that can transform to PFOS and are considered as historical PFAS, since PFOS and related chemicals are restricted. DiPAP have been reported in food packaging materials, from Sweden and elsewhere (Trier et al. 2011, KemI 2021). The contaminated site in Norway was dominated by the precursor compounds FTSA and FASA (including derivatives). The high prevalence of diPAP and lack of detected FASA in current study indicates use of different PFAS that can be related to paper products and manufacturing processes. Other sources for PFAS in the sediments besides the nearby paper factories cannot be entirely ruled out. Another important emission source of PFAS is aqueous film forming foams (AFFF). Sediment core samples from an AFFF-contaminated lake in Sweden showed PFAS concentrations between $<1 \mu\text{g}/\text{kg}$ dw and $76 \mu\text{g}/\text{kg}$ d.w. (Mussabek et al. 2019). Contamination from AFFF is usually dominated by PFOS, PFHxS and FTSA.

Concentrations of diSAmPAP were only semi-quantified in the present study due to interferences from the sediment matrix and the issue of unseparated structural isomers. The identity of diSAmPAP was confirmed by high resolution mass spectrometry.

EOF was used in the present study to evaluate possible influence by non-targeted PFAS. One sample with relatively low PFAS-68 (52 ng/g d.w.) showed an EOF value of 632 ng/g d.w. (Munksund (M8)). The identity of the EOF is not known and it cannot be ruled out that it is non-PFAS compounds causing the signal. The corresponding concentration of the unknown(s) cannot be calculated but a compound containing 50% fluorine (for comparison; PFOA has 69% and diSAmPAP 27%) would have a corresponding concentration of 1264 ng/g d.w..

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Appendix A

List of included PFAS in the study

	Perfluorobutanoic acid (PFBA)
	Perfluoropentanoic acid (PFPeA)
	Perfluorohexanoic acid (PFHxA)
	Perfluoroheptanoic acid (PFHpA)
	Perfluorooctanoic acid (PFOA)
	Perfluorononanoic acid (PFNA)
	Perfluorodecanoic acid (PFDA)
Perfluoroalkyl acid (PFCA)	Perfluoroundecanoic acid (PFUnDA)
	Perfluorododecanoic acid (PFDoDA)
	Perfluorotridecanoic acid (PFTrDA)
	Perfluorotetradecanoic acid (PFTeDA)
	Perfluoropentadecanoic acid (PFPeDA)
	Perfluorohexadecanoic acid (PFHxDA)
	Perfluoroheptadecanoic acid (PFHpDA)
	Perfluorooctadecanoic acid (PFOcDA)
	Perfluorobutane sulfonic acid (PFBS)
	Perfluoropentane sulfonic acid (PFPeS)
	Perfluorohexane sulfonic acid (PFHxS)
	Perfluorooctane sulfonic acid (PFOS)
Perfluoroalkyl sulfonic acid (PFSA)	Perfluorononane sulfonic acid (PFNS)
	Perfluorodecane sulfonic acid (PFDS)
	Perfluoroundecane sulfonic acid (PFUnDS)
	Perfluorododecane sulfonic acid (PFDoDS)
	Perfluorotridecane sulfonic acid (PFTrDS)
	Perfluorotetradecane sulfonic acid (PFTeDS)
	Perfluorobutane sulfonamide (FBSA)
Fluoroalkylsulfonamide (FASA)	N-Methyl perfluorobutane sulfonamide (MeFBSA)
	Perfluorohexane sulfonamide (FHxSA)

	N-Methyl perfluorohexane sulfonamide (MeFHxSA)
	Perfluorooctane sulfonamide (FOSA)
	N-Methyl perfluorooctane sulfonamide (MeFOSA)
	N-Ethyl perfluorooctane sulfonamide (EtFOSA)
	Perfluorooctane sulfonamidoacetic acid (FOSAA)
Fluorooctanesulfonamidoacetic acid (FOSAA)	N-Methyl perfluorooctane sulfonamidoacetic acid (MeFOSAA)
	N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA)
	4:2 Fluorotelomer sulfonic acid (4:2 FTSA)
	6:2 Fluorotelomer sulfonic acid (6:2 FTSA)
Fluorotelomer sulfonic acid (FTSA)	8:2 Fluorotelomer sulfonic acid (8:2 FTSA)
	10:2 Fluorotelomer sulfonic acid (10:2 FTSA)
	12:2 Fluorotelomer sulfonic acid (12:2 FTSA)
	14:2 Fluorotelomer sulfonic acid (14:2 FTSA)
	4:2 Fluorotelomer phosphate diester (4:2 diPAP)
	6:2 Fluorotelomer phosphate diester (6:2 diPAP)
	8:2 Fluorotelomer phosphate diester (8:2 diPAP)
Polyfluoroalkyl phosphate diesters (diPAP)	6:2/8:2 Fluorotelomer phosphate diester (6:2/8:2 diPAP)
	8:2/10:2 Fluorotelomer phosphate diester (8:2/10:2 diPAP)
	10:2 Fluorotelomer phosphate diester (10:2 diPAP)
	10:2/12:2 Fluorotelomer phosphate diester (10:2/12:2 diPAP)
	12:2 Fluorotelomer phosphate diester (12:2 diPAP)
N-ethyl perfluorooctane sulfonamido ethyl phosphate diester	diSAmPAP
	Hexafluoropropylene oxide dimer acid (HFPO-DA)(GenX)
Perfluoro ether acids / sulfonic acids (PFECA/PFESA)	3H-perfluoro-3-[(3-methoxy-propoxy)propanoic acid] (ADONA)
	6:2 chlorinated polyfluorinated ether sulfonate (6:2 Cl-PFESA)
	8:2 chlorinated polyfluorinated ether sulfonate (8:2 Cl-PFESA)
Perfluoroethylcyclohexane sulfonic acid	PFECHS
	Perfluorohexyl phosphonic acid (PFHxPA)

Perfluoroalkyl phosphonic acid (PFPA)	Perfluorooctyl phosphonic acid (PFOPA) Perfluorodecyl phosphonic acid (PFDPA)
Perfluoroalkyl phosphinic acid (PFPIA)	Bis(perfluorohexyl) phosphinic acid (C6/C6 PFPIA) Perfluoro (hexyloctyl) phosphinic acid (C6/C8 PFPIA) Bis(perfluorooctyl) phosphinic acid (C8/C8 PFPIA)
Fluorotelomer acids (FTCA)	3:3 Fluorotelomer carboxylic acid (3:3 FTCA) 5:3 Fluorotelomer carboxylic acid (5:3 FTCA) 7:3 Fluorotelomer carboxylic acid (7:3 FTCA)
Fluorotelomer unsaturated acids (FTUCA)	6:2 Fluorotelomer unsaturated carboxylic acid (6:2 FTUCA) 8:2 Fluorotelomer unsaturated carboxylic acid (8:2 FTUCA) 10:2 Fluorotelomer unsaturated carboxylic acid (10:2 FTUCA)

Appendix B

Information on sediment samples included in the study

Sample	Marine / Fresh	Site	Closest city	Close to factory	Depth (cm)	Sampling date	Part of sampling campaign and code *	ORU code
Å1 0-3.5	F	Ångermanälven	Kramfors	Dynäs Mondi Packaging AB	0-3.5	2015-08-15	RT001F; V3	MH-21-009:001
Å2 0-3.5	F	Ångermanälven	Kramfors	Dynäs Mondi Packaging AB	0-3.5	2015-08-15	RT002F; V4	MH-21-009:002
I5 0-15	M	Gårdsfjärden (downstream Iggesund)	Iggesund	Holmen Iggesund	0-15	2015-05-28	Fin15_i005_04_000_015A	MH-21-009:003
I40 0-15	M	Byfjärden (close to Iggesund)	Iggesund	Holmen Iggesund	0-15	2016-05-05	Fin15_i040_01_000_015A	MH-21-009:005
I40b 0-15	M	Byfjärden (close to Iggesund)	Iggesund	Holmen Iggesund	0-15	2016-05-05	Fin15_i040_01_000_015B	MH-21-009:006
I35 0-15	M	Gårdsfjärden (downstream Iggesund)	Iggesund	Holmen Iggesund	0-15	2015-10-29	fin15_i035_02_000_015A	MH-21-009:042
I31 0-20	M	Iggesundsfjärden (downstream Iggesund)	Iggesund	Holmen Iggesund	0-20	2015-05-28	fin15_i031_02_000_020A	MH-21-009:045
I36 0-15	M	Byfjärden (close to Iggesund)	Iggesund	Holmen Iggesund	0-15	2015-10-29	fin15_i036_01_000_015A	MH-21-009:047
U 0-5	M	Umeälven	Umeå	SCA Obbola	0-5	2021-05-04		MH21-016(018):2 S 0-5
U 5-10	M	Umeälven	Umeå	SCA Obbola	5-10	2021-05-04		MH21-016(018):2 S 5-10

U 10-15	M	Umeälven	Umeå	SCA Obbola	10-15	2021-05-04		MH21-016(018):2 S 10-15
U 15-25	M	Umeälven	Umeå	SCA Obbola	15-25	2021-05-04		MH21-016(018):2 S 15-25
U1c 0-30	M	Umeälven	Umeå	SCA Obbola	0-30	2015-09-04	Fin15_w001_01_000_030C	MH-21-009:007
U1d 0-30	M	Umeälven	Umeå	SCA Obbola	0-30	2015-09-04	Fin15_w001_01_000_030 D	MH-21-009:008
U11 0-15	M	Umeälven	Umeå	SCA Obbola	0-15	2015-09-04	Fin15_w011_01_000_015 D	MH-21-009:009
M8 0-20	M	Munksund	Piteå	SCA Munksund	0-20	2015-10-07	Fin15_z008_01_000_020B	MH-21-009:010
O 0-5	M	Ortviken	Sundsvall	SCA Ortviken	0-5	2021-05-06		MH-21-009:028
O 5-15	M	Ortviken	Sundsvall	SCA Ortviken	5-15	2021-05-06		MH-21-009:029
O 15-35	M	Ortviken	Sundsvall	SCA Ortviken	15-35	2021-05-06		MH-21-009:030
B12 0-5	F	Byviken	Säffle	Nordic Paper	0-5	2020-08-26	Provpunkt D (12)	MH-21-012:386
B6 0-5	F	Byviken	Säffle	Nordic Paper	0-5	2020-08-26	Provpunkt D (6)	MH-21-012:368
B6 10-15	F	Byviken	Säffle	Nordic Paper	10-15	2020-08-26	Provpunkt D (6)	MH-21-012:370

B2 0-5	F	Byviken	Säffle	Nordic Paper	0-5	2020-08-01	Provpunkt D (2)	MH-21-012:364
B7 0-5	F	Byviken	Säffle	Nordic Paper	0-5	2020-08-26	Provpunkt D (7)	MH-21-012:372
B7 10-15	F	Byviken	Säffle	Nordic Paper	10-15	2020-08-26	Provpunkt D (7)	MH-21-012:374
B3 0-5	F	Byviken	Säffle	Nordic Paper	0-5	2020-08-26	Provpunkt D (3)	MH-21-012:366
Ås1 0-5	F	Åsfjorden	Grums	Gruvöns bruk (BillerudKorsnäs)	0-5	2020-08-03	Provpunkt E (1)	MH-21-012:392
Ås1 15-20	F	Åsfjorden	Grums	Gruvöns bruk (BillerudKorsnäs)	15-20	2020-08-03	Provpunkt E (1)	MH-21-012:394
Ås3 0-5	F	Åsfjorden	Grums	Gruvöns bruk (BillerudKorsnäs)	0-5	2020-08-01	Provpunkt E (3)	MH-21-012:396
Ås3 20-25	F	Åsfjorden	Grums	Gruvöns bruk (BillerudKorsnäs)	20-25	2020-08-01	Provpunkt E (3)	MH-21-012:398
Ås51 2-7	F	Åsfjorden	Grums	Gruvöns bruk (BillerudKorsnäs)	2-7	2020-08-03	Provpunkt E (51)	MH-21-012:478
Ås7 0-5	F	Åsfjorden	Grums	Gruvöns bruk (BillerudKorsnäs)	0-5	2020-08-02	Provpunkt E (7)	MH-21-012:402
Ås7 15-20	F	Åsfjorden	Grums	Gruvöns bruk (BillerudKorsnäs)	15-20	2020-08-02	Provpunkt E (7)	MH-21-012:404
K10 0-5	F	Kattfjorden	Skoghall	Stora Enso	0-5	2020-08-30	Provpunkt F (10)	MH-21-012:500
K10 45-75	F	Kattfjorden	Skoghall	Stora Enso	45-75	2020-08-30	Provpunkt F (10)	MH-21-012:502

K10 175-205	F	Kattfjorden	Skoghall	Stora Enso	175-205	2020-08-30	Provpunkt F (10)	MH-21-012:504
K34 0-5	F	Kattfjorden	Skoghall	Stora Enso	0-5	2020-08-31	Provpunkt F (34)	MH-21-012:548
K34 10-15	F	Kattfjorden	Skoghall	Stora Enso	10-15	2020-08-31	Provpunkt F (34)	MH-21-012:550
K34 20-40	F	Kattfjorden	Skoghall	Stora Enso	20-40	2020-08-31	Provpunkt F (34)	MH-21-012:552
K12 0-5	F	Kattfjorden	Skoghall	Stora Enso	0-5	2020-08-04	Provpunkt F (12)	MH-21-012:508
K7 0-5	F	Kattfjorden	Skoghall	Stora Enso	0-5	2020-08-04	Provpunkt F (7)	MH-21-012:496
M3 0-5	F	Mariestadsfjärden	Katrinefors	Metsä Tissue	0-5	2020-08-06	Provpunkt K (3)	MH-21-012:587
M3 10-15	F	Mariestadsfjärden	Katrinefors	Metsä Tissue	10-15	2020-08-06	Provpunkt K (3)	MH-21-012:588
M4 0-5	F	Mariestadsfjärden	Katrinefors	Metsä Tissue	0-5	2020-08-06	Provpunkt K (4)	MH-21-012:589
GS2 0-5	F	Glan (Skärblacka downstream 2)	Norrköping	Skärblacka bruk (BillerudKorsnäs)	0-5	2021-06-28		MH-21-009:017
GS2 5-10	F	Glan (Skärblacka downstream 2)	Norrköping	Skärblacka bruk (BillerudKorsnäs)	5-10	2021-06-28		MH-21-009:018
GS2 10-25	F	Glan (Skärblacka downstream 2)	Norrköping	Skärblacka bruk (BillerudKorsnäs)	10-25	2021-06-28		MH-21-009:019
GS1 0-5	F	Glan (Skärblacka downstream 1)	Norrköping	Skärblacka bruk (BillerudKorsnäs)	0-5	2021-06-28		MH-21-009:020

GS1 5-10	F	Glan (Skärblacka downstream 1)	Norrköping	Skärblacka bruk (BillerudKorsnäs)	5-10	2021-06-28		MH-21-009:021
GS1 10-25	F	Glan (Skärblacka downstream 1)	Norrköping	Skärblacka bruk (BillerudKorsnäs)	10-25	2021-06-28		MH-21-009:022
GF1b 0-5	F	Glan (Fiskeby downstream 1b)	Norrköping	Fiskeby bruk	0-5	2021-06-28		MH-21-009:023
GF1b 5-10	F	Glan (Fiskeby downstream 1b)	Norrköping	Fiskeby bruk	5-10	2021-06-28		MH-21-009:024
GF1b 10-25	F	Glan (Fiskeby downstream 1b)	Norrköping	Fiskeby bruk	10-25	2021-06-28		MH-21-009:025
Bj2 0-40	F	Björken	Ställdalen	Ahlstrom-Munksjö Ställdalen	0-40	2022	rfs_Björken 25.2	MH-21-009:031
Bj1 10-15	F	Björken	Ställdalen	Ahlstrom-Munksjö Ställdalen	10-15	2022	rfs_Björken 25.1	MH-21-009:032
Bj3 10-15	F	Björken	Ställdalen	Ahlstrom-Munksjö Ställdalen	10-15	2022	rfs_Björken 25.3	MH-21-009:033
Br3 0-5	F	Bråviken	Norrköping	Holmen Braviken Paper	0-5	2021-10	rfs21_n003_06_000_005_A	MH-21-009:034
Br7 0-5	F	Bråviken	Norrköping	Holmen Braviken Paper	0-5	2021-10	rfs21_n007_07_000_005_A	MH-21-009:036
Br4 0-5	F	Bråviken (close to outlet)	Norrköping	Holmen Braviken Paper	0-5	2021-10	rfs_n004_Holmen 6500140:573485	MH-21-009:040
Br3b 0-5	F	Bråviken (upstream)	Norrköping	Holmen Braviken Paper	0-5	2021-10	rfs_n003_Holmen 6500050:569290	MH-21-009:041

* Fin15 = Norrlin et al. (2016). Kartläggning och riskklassning av fiberbankar i Norrland.

Provpunkt D-K = Larsson et al. (2021) Förorenade sedimentområden i Väneren.

RT = Dahlberg et al. (2020)

rfs = Regeringsuppdraget förorenade sediment, RUF5

Appendix C

Information on water samples included in the study

Site	Closest city	Close to factory	Sampling date	Original Sample code	ORU Code
Glan (Skärblacka downstream close to outlet)	Norrköping	Skärblacka bruk (BillerudKorsnäs)	2021-06-28	6493923:552338	MH-21-009:013
Glan (Skärblacka downstream close to outlet)	Norrköping	Skärblacka bruk (BillerudKorsnäs)	2021-06-28	6493923:552338	MH-21-009:014
Glan (Skärblacka upstream ref)	Norrköping	Skärblacka bruk (BillerudKorsnäs)	2021-06-28	6493621:552913 (N:E)	MH-21-009:011
Glan (Fiskeby downstream 1b close to outlet)	Norrköping	Fiskeby bruk	2021-06-28	6494747:567192	MH-21-009:012
Glan (Fiskeby downstream 1a close to outlet)	Norrköping	Fiskeby bruk	2021-06-28	6495437:565338	MH-21-009:026
Glan (Fiskeby upstream ref)	Norrköping	Fiskeby bruk	2021-06-28	6494847:563402	MH-21-009:027
Bråviken	Norrköping	Holmen Braviken Paper	2021-10	Surface water_Holmen_n003	MH-21-009:038
Bråviken	Norrköping	Holmen Braviken Paper	2021-10	n004_surface water_Holmen	MH-21-009:039

Appendix D

Concentrations (ng/g d.w.) of PFAS and EOF in sediment

	EOF	∑PFCA-15	∑PFSA-11	∑FASA-7	∑FOSAA-3	∑FTSA-6	∑diPAP-8	di-SAmPAP*	∑FTCA/FTUCA-6
LOD		0.04-0.3	0.04-0.1	0.1	0.1	0.1	0.1	1	0.1
Å1 0-3.5	<116	0.18	<lod	<lod	<lod	0.23	1.16	0.14	<lod
Å2 0-3.5	380	0.10	<lod	<lod	<lod	<lod	0.34	0.16	<lod
I5 0-15	462	0.53	0.56	0.35	13.8	<lod	<lod	107	<lod
I40 0-15	1301	0.70	0.21	4.67	13.5	<lod	<lod	108	<lod
I40b 0-15	<466	1.12	0.19	0.30	2.84	<lod	<lod	70.5	0.14
I35 0-15	<350	0.48	0.84	1.91	137	<lod	<lod	<lod	<lod
I31 0-20	547	0.54	1.01	1.20	129	<lod	<lod	<lod	<lod
I36 0-15	<350	0.18	1.22	1.44	30.9	<lod	<lod	<lod	<lod
U 0-5	<211	0.50	0.47	<lod	0.32	<lod	0.95	1.10	<lod
U 5-10	<211	0.21	0.18	<lod	0.88	<lod	3.29	1.90	<lod
U 10-15	<211	0.22	0.17	<lod	3.30	<lod	6.58	10.5	<lod
U 15-25	<211	0.18	0.26	<lod	10.1	<lod	19.0	76.1	<lod
U1c 0-30	583	3.59	1.73	<lod	25.5	1.31	<lod	32.0	6.11
U1d 0-30	1092	5.82	3.75	0.40	5.57	<lod	117	31.5	6.47
U11 0-15	477	0.21	<lod	<lod	3.76	<lod	85.7	5.44	<lod
M8 0-20	632	0.25	<lod	<lod	1.82	<lod	1.31	48.3	<lod
O 0-5	<211	0.28	0.65	<lod	<lod	<lod	0.13	<lod	<lod
O 5-15	<211	0.11	0.34	<lod	0.14	<lod	0.13	<lod	<lod
O 15-35	469	0.11	<lod	<lod	<lod	<lod	0.12	<lod	<lod
B12 0-5	<282	<lod	<lod	<lod	0.67	<lod	2.76	45.4	<lod
B6 0-5	<282	0.37	0.32	<lod	1.50	<lod	8.30	98.7	<lod
B6 10-15	<282	0.30	0.34	<lod	1.56	<lod	9.08	89.5	<lod
B2 0-5	412	1.08	0.89	<lod	6.63	<lod	34.0	299	<lod
B7 0-5	<282	0.28	0.31	<lod	14.3	<lod	13.4	235	<lod
B7 10-15	<211	1.49	1.57	0.17	0.94	<lod	10.5	97.2	<lod
B3 0-5	360	<lod	<lod	<lod	1.02	<lod	5.07	81.8	<lod
Ås1 0-5	838	1.10	<lod	<lod	4.20	0.80	2.71	21.2	2.51
Ås1 15-20	591	<lod	<lod	<lod	0.70	0.58	792	<lod	<lod
Ås3 0-5	1221	11.4	0.80	<lod	4.29	1.99	1083	69.2	9.47
Ås3 20-25	1136	3.71	<lod	<lod	3.65	2.43	819	70.6	9.78
Ås51 2-7	<282	1.34	0.10	<lod	0.67	0.67	114	9.3	0.14
Ås7 0-5	372	1.43	<lod	<lod	1.25	1.79	287	15.4	3.31
Ås7 15-20	<282	<lod	<lod	<lod	<lod	<lod		0.19	<lod
K10 0-5	<282	0.72	0.52	<lod	0.84	<lod	3.56	11.5	<lod
K10 45-75	<282	0.38	0.45	<lod	0.11	<lod	0.77	1.38	<lod
K10 175-205	<282	<lod	0.71	<lod	7.90	<lod	<lod	<lod	<lod
K34 0-5	<282	2.7	2.3	<lod	24.9	<lod	<lod	<lod	<lod

K34 10-15	<282	<lod	<lod	<lod	<lod	<lod	<lod	<lod	<lod
K34 20-40	<282	<lod	1.23	<lod	27.4	<lod	<lod	<lod	<lod
K12 0-5	<282	0.12	1.15	<lod	50.8	<lod	<lod	<lod	<lod
K7 0-5	409	<lod	<lod	<lod	<lod	<lod	<lod	<lod	<lod
M3 0-5	<282	0.65	0.49	<lod	0.28	0.14	13.4	<lod	<lod
M3 10-15	<282	0.19	0.36	<lod	5.83	<lod	0.39	<lod	<lod
M4 0-5	<282	0.71	0.75	<lod	<lod	<lod	0.81	1.40	<lod
GS2 0-5	<408	0.60	7.08	0.12	12.0	<lod	0.56	134	<lod
GS2 5-10	<408	0.34	2.71	0.18	41.6	<lod	0.28	682	<lod
GS2 10-25	<408	0.35	1.49	0.22	65.4	<lod	<lod	636	<lod
GS1 0-5	<408	0.83	5.05	0.12	<lod	0.82	<lod	2.79	<lod
GS1 5-10	<408	0.76	4.09	0.12	<lod	0.31	<lod	<lod	<lod
GS1 10-25	<408	0.32	0.76	<lod	6.15	<lod	<lod	14.6	<lod
GF1b 0-5	<408	0.23	0.35	<lod	1.64	<lod	<lod	10	<lod
GF1b 5-10	<408	0.20	0.19	<lod	2.93	<lod	<lod	5.5	<lod
GF1b 10-25	<408	0.20	0.21	<lod	5.32	<lod	<lod	15	<lod
Bj2 0-40	352	1.38	0.83	0.99	60.5	<lod	0.26	<lod	<lod
Bj1 10-15	<350	0.21	0.77	0.14	2.43	<lod	<lod	<lod	<lod
Bj3 10-15	<350	0.13	0.28	0.16	9.04	<lod	<lod	<lod	<lod
Br3 0-5	<350	0.37	0.84	<lod	2.74	<lod	0.15	<lod	<lod
Br7 0-5	<350	0.37	2.96	<lod	2.42	<lod	0.12	<lod	<lod
Br4 0-5	<350	0.49	0.33	<lod	0.92	<lod	<lod	<lod	<lod
Br3b 0-5	<350	0.29	0.67	<lod	3.26	<lod	0.18	<lod	<lod

* Concentrations are semi-quantitative

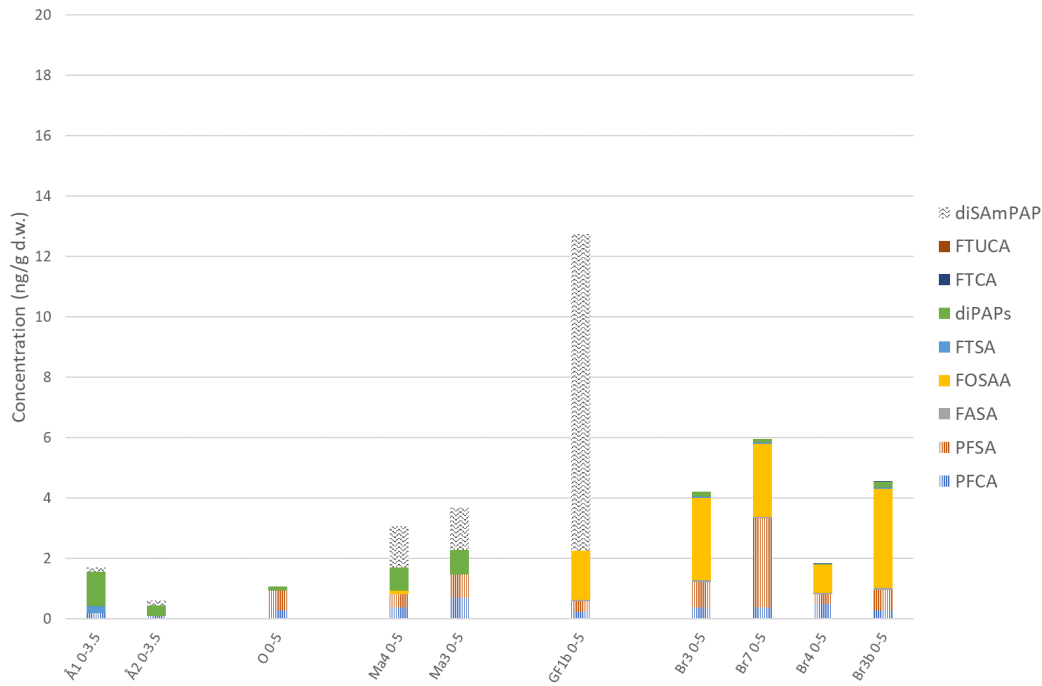
Appendix E

Concentrations (ng/mL) of PFAS in water

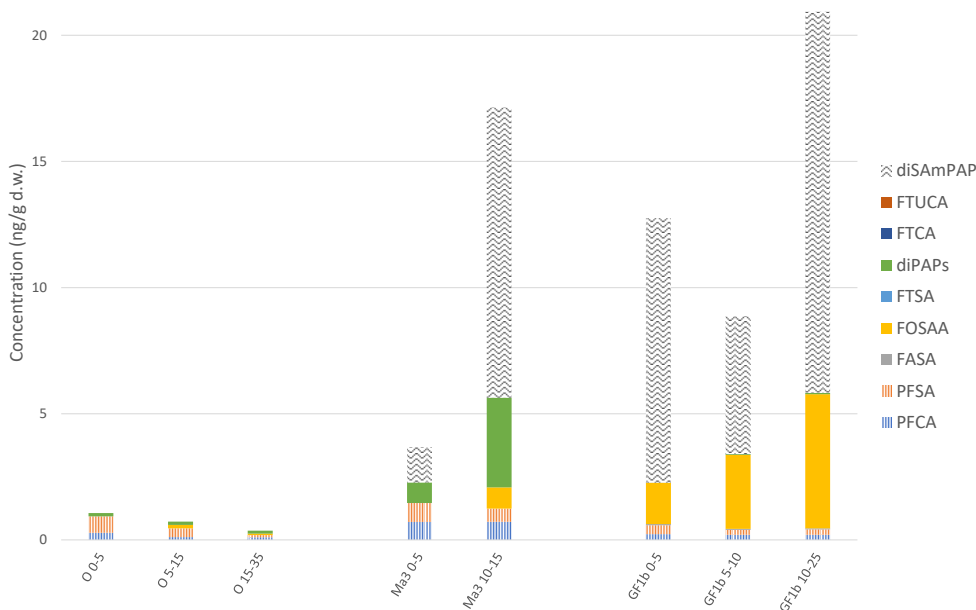
	Σ PFCA-15	Σ PFSA-11	Σ FTSA-6
Glan (Skärblacka downstream close to outlet)	0,0078	0,0014	<0,001
Glan (Skärblacka downstream close to outlet)	0,0073	0,0013	<0,001
Glan (Skärblacka upstream ref)	0,0074	0,0012	<0,001
Glan (Fiskeby downstream close to outlet)	0,0092	0,0009	0,044
Glan (Fiskeby downstream close to outlet)	0,0046	0,0008	0,023
Glan (Fiskeby upstream ref)	0,0035	0,0006	<0,001
Bråviken (close to outlet)	0,0052	0,0010	<0,001
Bråviken (close to city)	0,0183	0,0232	<0,001

Appendix F

Concentrations of PFAS-68 in sediment samples from the five least contaminated sites.



Concentrations (ng/g dry weight) of sum PFAS-68 and the contribution of different PFAS classes in surface sediments from the five least contaminated areas in this study.



Concentrations (ng/g dry weight) of sum PFAS-68 and the contribution of different PFAS classes in core sediment profiles from the three least contaminated areas in this study.