

PFASs in chemical products and textiles

PM 2/22



Swedish Chemicals Agency

The Swedish Chemicals Agency is supervisory authority under the Government. We work in Sweden, the EU and internationally to develop legislation and other incentives to promote good health and improved environment. We monitor compliance of applicable rules on chemical products, pesticides and substances in articles and carry out inspections. We review and authorise pesticides before they can be used. Our environmental quality objective is A Non-toxic Environment.

© Swedish Chemicals Agency. Stockholm.

Article number: 511 439.

Preface

PFASs (short for per- and polyfluoroalkyl substances) constitute a group of thousands man-made chemicals that are widely used in various technical applications in society due to their unique physical and chemical properties. Since they are chemically and thermally stable as well as repellent to water and oil, they are used in products such as water- and stain repellent textiles, fire-fighting foams, ski waxes, floor polish and bike care products.

REACH (EC 1907/2006) is a regulation of the European Union, adopted to improve the protection of human health and the environment from the risks that can be posed by chemicals, while enhancing the competitiveness of the EU chemicals industry. Restrictions are an instrument to protect human health and the environment from unacceptable risks posed by chemicals. Restrictions are normally used to limit or ban the manufacture, placing on the market or use of a substance, but can impose any relevant condition, such as requiring technical measures or specific labels. A restriction may apply to any substance on its own or in an article, including imported articles.

The dossier proposing the restriction contains background information such as the identity of the substance and justifications for the proposed restrictions. It includes the identified risks, any information on alternatives to the substance and the costs, as well as the environmental and human health benefits, resulting from the restriction.

NILU (Norwegian Institute for Air Research) and Örebro University have conducted this study on PFASs in chemical products and textiles by contract of the Swedish Chemicals Agency as part of the Agency's preparatory work on a broad PFAS restriction under the REACH regulation.

Contact at the Swedish Chemicals Agency was Jenny Ivarsson. Robin Vestergren and Daniel Borg have also contributed to the work. Head of Unit, Kerem Yazar, was responsible for the assignment.

Stockholm, January 2022

Content

Glossary	5
Summary	7
Sammanfattning	8
1 Introduction.....	9
2 Materials and Methods	10
1.1 Chemical products	10
1.2 Textiles	12
1.3 Instrumental analysis	14
1.4 Quality control and quality assurance	16
3 Results.....	18
1.5 Chemical products	18
1.6 Textiles	20
1.7 Comparison of EOF and calculated fluorine content	22
4 Discussion.....	25
5 References	26
Appendix 1	27
Appendix 2	46
Details about the quality assurance for EOF	46

Glossary

4:2 FTOH	4:2 Fluorotelomer alcohol
4x3 PFECA	Perfluoro(2,5,8,10-tetramethyl-3,6,9-trioxaundecanoic) acid. Cas nr: 1212077-14-9
6:2 FTOH	6:2 Fluorotelomer alcohol
8:2 FTOH	8:2 Fluorotelomer alcohol
10:2 FTOH	10:2 Fluorotelomer alcohol
4:2 FTS	4:2 Fluorotelomer sulfonic acid
6:2 FTS	6:2 Fluorotelomer sulfonic acid
8:2 FTS	8:2 Fluorotelomer sulfonic acid
10:2 FTS	10:2 Fluorotelomer sulfonic acid
CIC	Combustion ion chromatography
EOF	Extractable organic fluorine
FOSA	Perfluorooctane sulfonamide
GC-MS	Gas chromatography mass spectrometry
Gen X	Perfluoro-2-methyl-3-oxahexanoic acid
KEMI	Swedish Chemicals Agency
LC-MS	Liquid chromatography mass spectrometry
LOD	Limit of detection
N-Et-FOSE	N-Ethyl perfluorooctane sulfonamidoethanol
NILU	Norwegian Institute for Air Research
N-Et-FOSA	N-Ethyl-heptadecafluorooctane sulfonamide
N-Et-FOSAA	N-Ethyl perfluorooctane sulfonamidoacetic acid
N-Et-FOSE	N-Ethyl-heptadecafluorooctane sulfonamidoethanol
N-Me-FOSA	N-Methyl-heptadecafluorooctane sulfonamide
N-Me-FOSAA	N-Methyl perfluorooctane sulfonamidoacetic acid
N-Me-FOSE	N-Methyl-heptadecafluorooctane sulfonamidoethanol
PAPs	Polyfluoroalkyl phosphate ester
PFAS	Per- and polyfluorinated alkyl substances
PFBA	Perfluorobutanoic acid
PFBS	Perfluorobutane sulfonate
PFCA	Perfluorinated carboxylic acid
PFDS	Perfluorodecane sulfonate
PFDA	Perfluorodecanoate
PFDoDA	Perfluorododecanoate
PFECHS	Perfluoro(perfluoroethyl)cyclohexanesulfonic acid
PFHpA	Perfluoroheptanoate
PFHpS	Perfluoroheptane sulfonate
PFHxDA	Perfluorohexadecanoic acid
PFHxA	Perfluorohexanoate

PFHxS	Perfluorohexane sulfonate
PFNA	Perfluorononanoate
PFNS	Perfluorononane sulfonate
PFOA	Perfluoroctanoate
PFOcDA	Perfluorooctadecanoic acid
PFOS	Perfluorooctane sulfonate
PPPeA	Perfluoropentanoic acid
PFPS	Perfluoropentane sulfonate
PFSA	Perfluorinated sulfonic acids
PFTrDA	Perfluorotridecanoate
PFTeDA	Perfluorotetradecanoate
PFUnDA	Perfluoroundecanoate
TF	Total fluorine

Summary

NILU (Norwegian Institute for Air Research) has in 2020, on behalf of the Swedish Chemicals Agency (KEMI), performed chemical analyses of individual per- and polyfluorinated alkyl substances (PFASs), extractable organic fluorine (EOF) or total fluorine (TF) in chemical products and textiles. The textile products (n=31) included jackets, gloves, shoes, back packs, and tents. The chemical products (n=31) included fire-fighting foams, ski waxes, shoe and textile impregnation/water proofing products, floor and bike care products. All products were purchased in Sweden and shipped to NILU for analysis.

The aim of the study was to identify and quantify the presence of 36 individual PFAS in all samples and to measure the content of EOF/TF to understand to what extent the targeted PFASs contribute to the EOF/TF and to what extent there are non-targeted PFASs in the products. The target analyses included PFOA, its salts, other PFOA related substances, PFOS and PFOS related substances, and a broad spectrum of other PFAS. The EOF/TF was compared to calculated fluorine content based on the target PFAS analyses.

The results show that two of the chemical products (both ski waxes) were above the limit value of 0.025 mg/kg for PFOA and salts of PFOA and that one of them was also above the limit value of 1 mg/kg for 8:2 FTOH and 10:2 FTOH, categorized as PFOA related compounds. The highest individual PFAS concentrations were found for 6:2 FTOH in a textile impregnation sample (1 000 mg/kg) followed by a ski wax, floor polish, textile impregnation and shoe care (15-50 mg/kg). In fire-fighting foams, 6:2 FTS was found at highest concentrations (0.3-10.6 mg/kg). In textile samples, 6:2 FTOH and PFTeDA were found at highest concentrations, all in jackets.

The highest amounts of EOF/TF were found in one textile impregnation sample and two ski wax samples (25 000-85 000 mg F/kg). The calculated fluorine from the targeted PFAS analysis was marginal compared to EOF/TF in samples where EOF/TF was above detection limit. This shows the complexity of the PFAS group and that some chemical products and textiles comprise a wide range of fluorinated compounds, also those not targeted in this study.

Sammanfattning

NILU (Norskt Institut för Luftforskning) har under 2020, på uppdrag av Kemikalieinspektionen, utfört kemiska analyser av individuella per- och polyfluorerade alkylsubstanser (PFAS), extraherbart organiskt fluor (EOF) eller totalt fluor (TF) i textilvaror och kemiska produkter. Textilvarorna (n=31) inkluderar jackor, handskar, skor, ryggsäckar och tält. De kemiska produkterna (n=31) inkluderar brandskum, skidvalla, sko- och textilimpregnering samt golv- och cykelvårdsprodukter. Alla produkter köptes in i Sverige och skickades till NILU för analys.

Målet med studien var att studera förekomsten av 36 individuella PFAS och mängden EOF/TF i alla textilier och kemiska produkter. Mängden EOF/TF jämfördes mot den totala mängden fluor från de individuella PFAS för att få insikt i om andra PFAS kan förekomma i produkterna. De individuella PFAS inkluderar PFOA, dess salter, andra PFOA-relaterade substanser, PFOS och PFOS-relaterade substanser samt ett brett spektrum av andra PFAS.

Resultaten visar att PFOA och dess salter var över gränsvärdet på 0,025 mg/kg i två av de kemiska produkterna (bägge skidvallor) och att en av dessa också var över gränsvärdet på 1 mg/kg för 8:2 FTOH och 10:2 FTOH. De högsta koncentrationerna uppmättes för 6:2 FTOH (15–1 000 mg/kg), alla i kemiska produkter. I brandskum uppmättes 6:2 FTS i högst koncentrationer (0,3–10,6 mg/kg). I textilier uppmättes 6:2 FTOH och PFTeDA i högst koncentrationer.

De högsta nivåerna av EOF/TF uppmättes i en textilimpregnéringsprodukt och två skidvallor (25 000–85 000 mg F/kg). Innehållet av fluor baserat på de individuella PFAS var betydligt lägre jämfört med EOF/TF. Detta visar att PFAS-gruppen är komplex och att några textilier och kemiska produkter innehåller ett brett spektrum av fluorerade ämnen, också utöver de 36 PFAS som var inkluderade i studien.

1 Introduction

Per- and polyfluorinated alkyl substances (PFASs) comprise a wide range of compounds that have been produced and used in a wide range of industrial and consumer applications since the 1950s. These applications include indoor related products such as consumer products; stain-proof coatings on furnishing and carpets, oil resistant coatings on food wrapping, non-stick coating on cooking utensils, water resistance in clothing and outdoor materials etc. This, together with the PFASs' global spread in the environment, bioaccumulation potential, persistence and toxicity have resulted in increasing attention from the scientific community and policymakers. Glüge et al. (2020) identified in total, more than 200 use categories and sub-categories for more than 1 400 individual PFAS. Today, more than 4 700 individual PFAS substances have been identified¹ and are used in a wide range of technical applications and consumer products.

¹ OECD, 2018. Toward a new comprehensive global database of per- and polyfluoroalkyl substances (PFASs): Summary report on updating the OECD 2007 list of per- and polyfluoroalkyl substances (PFASs). ENV/JM/MONO(2018)7. 4 May 2018.

2 Materials and Methods

Textile- and chemical products were purchased in physical stores or in online stores in Sweden by the Swedish Chemical Agency and sent to NILU in Norway for chemical analysis in September-October 2020.

All product samples were screened for 36 targeted PFASs. The presence of unknown PFASs, or other organic compounds containing fluorine, was evaluated by analysing the extractable organic fluorine content (EOF) in the same samples. EOF is reported for samples that have been extracted with an organic solvent (textiles), whereas total fluorine (TF) is reported for diluted samples where no extraction was conducted (chemical products), see Appendix 2.

2.1 Chemical products

At arrival, the chemical products ($n=31$) were unwrapped and inspected (see more details in Table 1).

Two sub-samples were taken from each chemical product to assess the analytical variability and increase confidence of results. The fire-fighting foams were transferred from the original container to a pre-cleaned glass bottle by removing the pressure of the containers before opening. This was done to ensure that the sub-samples were not contaminated by the tubes and valves of the container. The weight of each sub-sample was noted (Table 1).

All chemical products were diluted before analysis without further extraction and clean-up (according to Iso standard DS/CEN/TS 15968). For targeted PFAS analysis, the sub-samples were diluted 100 times with acetonitrile (Fisher Scientific, Fisher Chemical A/0627/17X) while for TF analysis the sub-samples were diluted 1 000 to 10 000 times with acetonitrile. To ensure efficient extraction of organic fluorinated compounds from ski waxes, the sub-samples of ski waxes were first dissolved in 900 μ L of acetone and put on ultrasonic bath for 10 min before dilution with acetonitrile.

One aliquot (0.5 mL) of each sub-sample was sent to Örebro University for determination of TF. The second aliquot (0.5 mL) was added ^{13}C labelled internal standard to determine the amount of the targeted PFASs in the samples (see Table 3).

Table 1: Chemical product samples, description, sample weight (g).

Sample ID	NILU ID	Product	Sample weight (g)
1	120118/01 A	Floor varnish	0.127
1	120118/01 B	Floor varnish	0.136
2	120118/02 A	Floor varnish	0.137
2	120118/02 B	Floor varnish	0.132
3	120118/03 A	Floor polish	0.099
3	120118/03 B	Floor polish	0.102
4A	120118/04 A	Floor varnish	0.114
4A	120118/04 B	Floor varnish	0.130
4B	120118/04 A	Floor varnish	0.110
4B	120118/04 B	Floor varnish	0.130
5	120118/05 A	Floor polish	0.109
5	120118/05 B	Floor polish	0.101

Sample ID	NILU ID	Product	Sample weight (g)
6	120118/06 A	Fire-fighting foam	0.090
6	120118/06 B	Fire-fighting foam	0.098
7	120118/07 A	Fire-fighting foam	0.098
7	120118/07 B	Fire-fighting foam	0.095
8	120118/08 A	Fire-fighting foam	0.102
8	120118/08 B	Fire-fighting foam	0.107
9	120118/09 A	Fire-fighting foam	0.105
9	120118/09 B	Fire-fighting foam	0.103
10	120118/10 A	Fire-fighting foam	0.095
10	120118/10 B	Fire-fighting foam	0.097
11	120118/11 A	Textile impregnation	0.075
11	120118/11 B	Textile impregnation	0.075
12	120118/12 A	Bike care	0.183
12	120118/12 B	Bike care	0.211
13	120118/13 A	Bike care	0.065
13	120118/13 B	Bike care	0.068
14	120118/14 A	Bike care	0.081
14	120118/14 B	Bike care	0.081
15	120118/15 A	Bike care	0.083
15	120118/15 B	Bike care	0.077
16	120118/16 A	Shoe care	0.120
16	120118/16 B	Shoe care	0.100
17	120118/17 A	Shoe care	0.080
17	120118/17 B	Shoe care	0.080
20	120118/20 A	Shoe care	0.105
20	120118/20 B	Shoe care	0.105
21	120118/21 A	Bike care	0.078
21	120118/21 B	Bike care	0.083
22	120118/22 A	Bike care	0.071
22	120118/22 B	Bike care	0.072
23	120118/23 A	Shoe care	0.059
23	120118/23 B	Shoe care	0.065
24	120118/24 A	Shoe care	0.061
24	120118/24 B	Shoe care	0.065
25	120118/25 A	Ski wax	0.100
25	120118/25 B	Ski wax	0.100
26	120118/26 A	Ski wax	0.100
26	120118/26 B	Ski wax	0.100
27	120118/27 A	Ski wax	0.150
27	120118/27 B	Ski wax	0.100
28	120118/28 A	Ski wax	0.078
28	120118/28 B	Ski wax	0.078
29	120118/29 A	Ski wax	0.100

Sample ID	NILU ID	Product	Sample weight (g)
29	120118/29 B	Ski wax	0.100
30	120118/30 A	Ski wax	0.075
30	120118/30 B	Ski wax	0.075
31	120118/31 A	Ski wax	0.100
31	120118/31 B	Ski wax	0.100
32	120118/32 A	Ski wax	0.100
32	120118/32 B	Ski wax	0.100
33	120118/33 A	Textile impregnation	0.103
33	120118/33 B	Textile impregnation	0.103

2.2 Textiles

At arrival, the textile products ($n=31$) were unwrapped and inspected (Table 2). During the analysis, the textiles were hung up on racks and covered with a polypropylene plastic bag to avoid contact with adjacent textiles. This was a precautionary step to avoid cross-contamination between samples in the laboratory. However, the textile products may have been contaminated by adjacent/neighbouring textiles in the shops before arrival to the laboratory.

Two sub-samples were taken from each textile sample to obtain representative samples. When feasible, the sub-samples were taken from an area consisting of a uniform material: even surface, without seams and prints, of the same colour and type of the product. This resulted in parallel samples and allowed to assess the analytical variability and increase confidence of results. When the product did not consist of a uniform material (e.g. shoes and gloves), instead the two sub-samples were taken from two different parts of the products. This aimed to obtain a representative sample of the whole product. The weight and surface area of each sub-sample was noted. See Table 2 for product details, sample amount in area (cm^2) and weight (g), and detailed description of the sample.

Each sub-sample was cut in smaller pieces before extraction in methanol (15-20 mL) using ultrasonication bath for 30 min (according to Iso standard DS/CEN/TS 15968). The amount of methanol was such that the sample was completely covered. After centrifugation, methanol was aliquoted out and the volume was reduced to an end-volume of 2 mL. A dispersive clean-up with 25 mg ENVI-Carb and 50 μL of glacial acetic acid (Powley et al. 2005) was performed before the sample was divided.

One aliquot (0.5 mL) of each sub-sample was sent to Örebro University for determination of EOF. The second aliquot (0.5 mL) was added ^{13}C labelled internal standard (see Table 3) to determine the extracted amount of the targeted PFASs in the samples.

Table 2: Textile samples, description, sampled area (cm²) and weight (g).

Sample ID	NILU ID	Product	Description	Sample area (cm ²)	Sample weight (g)
36	120118/36 A	Shoes	Thin film	46	0.83
36	120118/36 B	Shoes	Thin film	43	1.08
37	120118/37 A	Jacket	White outer layer	100	1.20
37	120118/37 B	Jacket	White outer layer	100	1.20
38	120118/38 A	Poncho	Green, thin fabric	100	0.88
38	120118/38 B	Poncho	Green, thin fabric	100	0.90
40	120118/40 A	Gloves	Inside	43	2.82
40	120118/40 B	Gloves	Outside	68	2.45
41	120118/41 A	Gloves	Inside	57	3.15
41	120118/41 B	Gloves	Outside	100	3.42
42	120118/42 A	Gloves	Inside	55	2.28
42	120118/42 B	Gloves	Outside	90	2.27
43	120118/43 A	Backpack	Front part	100	4.16
43	120118/43 B	Backpack	Front part	100	5.34
44	120118/44 A	Shoes	Thin film	435	0.47
44	120118/44 B	Shoes	Thin film	43	0.45
45	120118/45 A	Shoes	Thin film	45	0.92
45	120118/45 B	Shoes	Thin film	52	0.76
46	120118/46 A	Jacket	Grey	100	1.99
46	120118/46 B	Jacket	Black	100	1.97
47	120118/47 A	Jacket	Orange outer layer	100	1.55
47	120118/47 B	Jacket	Orange outer layer	100	1.60
48	120118/48 A	Tent	Not applicable	100	0.82
48	120118/48 B	Tent	Not applicable	100	0.81
49	120118/49 A	Backpack	Black, front part	100	2.62
49	120118/49 B	Backpack	Black, back part	100	2.60
50	120118/50 A	Shoes	Thin film	38	0.57
50	120118/50 B	Shoes	Thin film	16	0.26
51	120118/51 A	Backpack	Black, front part	100	1.18
51	120118/51 B	Backpack	Black, front part	100	1.17
52	120118/52 A	Tent	Not applicable	100	0.71
52	120118/52 B	Tent	Not applicable	100	0.73
53	120118/53 A	Jacket	Blue and grey	100	4.18
53	120118/53 B	Jacket	Grey	100	4.16
55	120118/55 A	Jacket	Brown, outer layer	100	1.20
55	120118/55 B	Jacket	Brown, outer layer	100	1.20
56	120118/56 A	Jacket	Blue, outer layer	100	1.26
56	120118/56 B	Jacket	Blue, outer layer	100	1.25
57	120118/57 A	Backpack	Grey, thick, front top layer	100	4.53
57	120118/57 B	Backpack	Grey, thick, front top layer	100	4.46
58	120118/58 A	Shoes	Thin film	66	0.36

Sample ID	NILU ID	Product	Description	Sample area (cm ²)	Sample weight (g)
58	120118/58 B	Shoes	Thin film	75	0.39
59	120118/59 A	Shoes	Thin film	43	1.19
59	120118/59 B	Shoes	Thin film	48	1.38
60	120118/60 A	Shoes	Thin film	26	0.23
60	120118/60 B	Shoes	Thin film	32	0.52
61	120118/61 A	Shoes	White film	50	1.14
61	120118/61 B	Shoes	White film	51	1.15
62	120118/62 A	Shoes	White fabric	54	1.20
62	120118/62 B	Shoes	White fabric	43	0.93
63	120118/63 A	Jacket	Purple, outer layer	100	2.27
63	120118/63 B	Jacket	Purple, outer layer	100	2.28
64	120118/64 A	Jacket	White, outer layer	100	1.50
64	120118/64 B	Jacket	White, outer layer	100	1.50
65	120118/65 A	Jacket	Black, outer layer	100	1.44
65	120118/65 B	Jacket	Black, outer layer	100	1.43
66	120118/66 A	Jacket	Not applicable	100	0.96
66	120118/66 B	Jacket	Not applicable	100	0.96
67	120118/67 A	Jacket	Outer layer	100	2.73
67	120118/67 B	Jacket	Outer layer	100	2.71
68	120118/68 A	Jacket	Not applicable	100	3.14
68	120118/68 B	Jacket	Not applicable	100	3.12

2.3 Instrumental analysis

Detection and quantification of individual targeted PFASs was carried out using liquid chromatography coupled to mass spectrometry (LC-MS) and gas chromatography coupled to mass spectrometry (GC-MS). Details about methods are described in Hanssen et al. (2013), and Blom and Hanssen (2015). Names and abbreviations of the targeted PFASs and their analytical method are listed in Table 3.

A detailed description of analysis of EOF are found in Kärrman et al. (2019). In brief, the CIC system had a combustion module and an autosampler (both from Analytik Jena, Germany), an absorber module (920 Absorber Module) and an ion chromatograph (IC; 930 Compact IC Flex), both from Metrohm, Switzerland. The anions were separated with an ion exchange column (Metrosep A Supp 5–150/4), carbonate buffer 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 and argon mixed with water vapor under hydrolytic condition. The hydrogen fluoride (HF) formed during combustion was absorbed in MilliQ water (in the absorber module). The F⁻ concentration was measured with the IC. A five-point calibration curve at 50, 100, 200, 500 and 1 000 µg/L PFOS standards was constructed using the combustion method as samples and exhibited good linearity with R²>0.9999.

Table 3: Overview of targeted PFASs and their instrumental method details.

Abbreviation	Full name	Detection method	LOD* (mg/kg) Textiles	LOD* (mg/kg) Chemical products	Measurement uncertainty (%) ^{a)}
4:2 FTS	4:2 Fluorotelomer sulfonic acid	UHPLC-MS/MS	<0.000009	<0.000037	20
6:2 FTS**	6:2 Fluorotelomer sulfonic acid	UHPLC-MS/MS	<0.000009	<0.000037	15
8:2 FTS**	8:2 Fluorotelomer sulfonic acid	UHPLC-MS/MS	<0.000007	<0.000033	15
10:2 FTS	10:2 Fluorotelomer sulfonic acid	UHPLC-MS/MS	<0.000007	<0.000033	20
PFBS	Perfluorobutane sulfonate	UHPLC-MS/MS	<0.000008	<0.000035	15
PFPS	Perfluoropentane sulfonate	UHPLC-MS/MS	<0.000012	<0.000052	15
PFHxS**	Perfluorohexane sulfonate	UHPLC-MS/MS	<0.000012	<0.000070	15
PFHpS	Perfluoroheptane sulfonate	UHPLC-MS/MS	<0.000016	<0.000131	15
PFOS**	Perfluorooctane sulfonate	UHPLC-MS/MS	<0.000039	<0.000077	15
PFNS	Perfluorononane sulfonate	UHPLC-MS/MS	<0.000018	<0.000077	15
PFDS	Perfluorodecane sulfonate	UHPLC-MS/MS	<0.000018	<0.000077	15
PFHxA**	Perfluorohexanoate	UHPLC-MS/MS	<0.00005	<0.000218	15
PFHpA**	Perfluoroheptanoate	UHPLC-MS/MS	<0.00005	<0.000218	15
PFOA**	Perfluorooctanoate	UHPLC-MS/MS	<0.00005	<0.000218	15
PFNA**	Perfluorononanoate	UHPLC-MS/MS	<0.000056	<0.000245	15
PFDA**	Perfluorodecanoate	UHPLC-MS/MS	<0.000026	<0.000114	15
PFUnDA**	Perfluoroundecanoate	UHPLC-MS/MS	<0.000026	<0.000112	15
PFDoDA**	Perfluorododecanoate	UHPLC-MS/MS	<0.00001	<0.000016	15
PTTrDA	Perfluorotridecanoate	UHPLC-MS/MS	<0.00001	<0.000045	20
PFTeDA**	Perfluorotetradecanoate	UHPLC-MS/MS	<0.00001	<0.000045	15
PFHxDA	Perfluorohexadecanoic acid	UHPLC-MS/MS	<0.00005	<0.000045	20
PFOcDA	Perfluorooctadecanoic acid	UHPLC-MS/MS	<0.0001	<0.000059	20
FOSA**	Perfluorooctane sulfonamide	UHPLC-MS/MS	<0.0001	<0.000394	15
N-Me-FOSAA	N-Methyl perfluorooctane sulfonamidoacetic acid	UHPLC-MS/MS	<0.0001	<0.0001	20

Abbreviation	Full name	Detection method	LOD* (mg/kg) Textiles	LOD* (mg/kg) Chemical products	Measurement uncertainty (%) ^{a)}
N-Et-FOSAA	N-Ethyl perfluorooctane sulfonamidoacetic acid	UHPLC-MS/MS	<0.0001	<0.0001	20
4x3 PFECA	Perfluoro(2,5,8,10-tetramethyl-3,6,9-trioxaundecanoic) acid Cas nr: 1212077-14-9	UHPLC-MS/MS	<0.0001	<0.0001	30
PFECHS	Perfluoro(perfluoroethyl)cyclohexane sulfonic acid	UHPLC-MS/MS	<0.0001	<0.0001	30
Gen X	Perfluoro-2-methyl-3-oxahexanoic acid	UHPLC-MS/MS	<0.0001	<0.0001	30
4:2 FTOH**	4:2 Fluorotelomer alcohol	GC-MS	<0.00226	<0.0226	30
6:2 FTOH**	6:2 Fluorotelomer alcohol	GC-MS	<0.00245	<0.0245	30
8:2 FTOH**	8:2 Fluorotelomer alcohol	GC-MS	<0.00477	<0.0477	30
10:2 FTOH**	10:2 Fluorotelomer alcohol	GC-MS	<0.00402	<0.0402	30
N-Me-FOSA **	N-Methyl-heptadecafluorooctane sulfonamide	GC-MS	<0.00137	<0.0137	30
N-Et-FOSA **	N-Ethyl-heptadecafluorooctane sulfonamide	GC-MS	<0.00137	<0.0137	30
N-Me-FOSE **	N-Methyl-heptadecafluorooctane sulfonamidoethanol	GC-MS	<0.00458	<0.0458	30
N-Et-FOSE **	N-Ethyl-heptadecafluorooctane sulfonamidoethanol	GC-MS	<0.00394	<0.0394	30

**Carbon labelled internal standards.

a) Expanded uncertainty equal to two times the standard deviation s_R , under reproducibility conditions.

2.4 Quality control and quality assurance

A rigorous quality assurance was applied to all samples to avoid contamination during handling of samples and chemical analyses. All equipment used were of one-time use or cleaned thoroughly between samples. All solvents were tested with respect to PFAS content prior to use. One blank sample was included with each batch of samples for extraction. None of the targeted PFASs were detected in the blank samples ensuring no or minimal contamination. Parallels of samples (sub-samples) were always extracted in the same batch. The analytical method detection limit (LOD) for the individual PFAS (Table 3) is based on blanks and calibration curve.

Details about the quality assurance and uncertainties for EOF/TF are presented in Appendix 2.

To assess the efficiency of the applied extraction and clean-up method, three replicates of one textile sample were taken out and spiked with 25 ng of native PFAS standards containing 23 of the 36 targeted PFASs. The spiked replicates were then extracted, cleaned-up and analysed for PFAS as textile samples. The results of these spiked replicates showed good recovery for all PFASs and a low analytical variability between samples (Table 4).

Table 4: PFAS concentrations in spiked sample (120118/48, tent). The sample was spiked with 25 ng native PFAS standard mix. The reported amounts are in ng.

PFAS compound	Amount of PFAS (ng/sample)	Stdev	%RSD			
	Parallel I	Parallel II	Parallel III	Average		
4:2 FTS	35.7	27.8	28.0	30.5	4.52	15
6:2 FTS	26.8	26.7	26.4	26.6	0.20	0.7
PFBS	23.3	24.5	25.1	24.3	0.89	3.7
PFPS	25.5	24.7	25.4	25.2	0.44	1.8
PFHxS	26.3	24.5	25.2	25.3	0.90	3.6
PFHpS	24.6	24.7	26.5	25.3	1.04	4.1
PFOS	23.8	24.4	24.3	24.2	0.35	1.5
PFNS	22.6	24.3	23.4	23.5	0.84	3.6
PFDS	25.0	26.5	24.9	25.5	0.91	3.6
PFBA	25.4	25.2	25.2	25.3	0.12	0.5
PFPeA	25.7	25.1	25.3	25.3	0.34	1.3
PFHxA	26.0	26.2	25.8	26.0	0.21	0.8
PFHpA	26.7	26.7	27.2	26.8	0.29	1.1
PFOA	26.2	25.0	25.3	25.5	0.66	2.6
PFNA	24.8	27.1	25.4	25.7	1.19	4.6
PFDA	26.5	26.9	25.9	26.4	0.52	1.9
PFUnDA	24.9	24.2	25.8	25.0	0.80	3.2
PFDoDA	28.7	27.7	27.7	28.0	0.63	2.2
PFTrDA	34.3	34.3	33.3	34.0	0.59	1.7
PFTeDA	26.0	24.8	26.4	25.7	0.83	3.2
PFHxDA	23.5	21.4	22.0	22.3	1.10	4.9
PFOcDA	23.9	25.8	25.6	25.1	1.06	4.2
FOSA	26.4	25.7	25.2	25.8	0.61	2.4

3 Results

The quantified concentrations of the targeted PFASs and EOF in the individual samples are presented in Table A1 and A2 in Appendix 1.

The results are compared to three categories of limit values given in the EU restrictions, according to Regulation (EU) 2019/1021 of the European Parliament and of the Council of 20 June 2019 on persistent organic pollutants (the POPs Regulation):

- PFOA and salts of PFOA: $\leq 0,025 \text{ mg/kg}$, or $\leq 1 \text{ mg/kg}$ for samples and technical products that contain micro-powder of PTFE;
- PFOA related substances: $\leq 1 \text{ mg/kg}$;
- PFOS and PFOS related substances: $\leq 10 \text{ mg/kg}$ in substances or in mixtures. $\leq 1 \mu\text{g/m}^2$ of the coated material for textiles or other coated materials.

3.1 Chemical products

The concentration of the individual PFAS measured in chemical products are presented in Table A1 in Appendix 1. Of the 36 targeted PFASs, 16 were detected in at least one of the chemical product samples, while the other 20 targeted PFASs were below detection limit in all chemical products.

Two of the 31 chemical product samples exceeded the limit values for PFOA and salts of PFOA (0.025 mg/kg): i) sample 25 (ski wax), and ii) sample 32 (ski wax). The concentration of 8:2 FTOH and 10:2 FTOH, categorized as PFOA related compounds, were also above the limit value in sample 25. The concentration of PFOA and salts of PFOA in sample 26 (ski wax) was just below the limit value (0.025 mg/kg).

Several PFAS, not covered by the EU restrictions, were detected in the chemical product samples. The amount of 6:2 FTS in fire-fighting foams (samples 6-10) ranged from 0.3 to 10.6 mg/kg (Figure 1). Also 4:2 FTS and PFHxS were above the detection limits in these fire-fighting foam samples. Among the volatile PFAS, 6:2 FTOH was detected in several samples, such as three floor varnish/lacquer (sample 1, 4 and 5), one shoe care product (sample 23), one ski wax (sample 28) and two textile impregnations (sample 11 and 33) (Figure 1). The highest concentration of an individual PFAS measured in this study was 6:2 FTOH in sample 33 A and B (textile impregnation), 588 and 1 038 mg/kg, respectively (Figure 1).

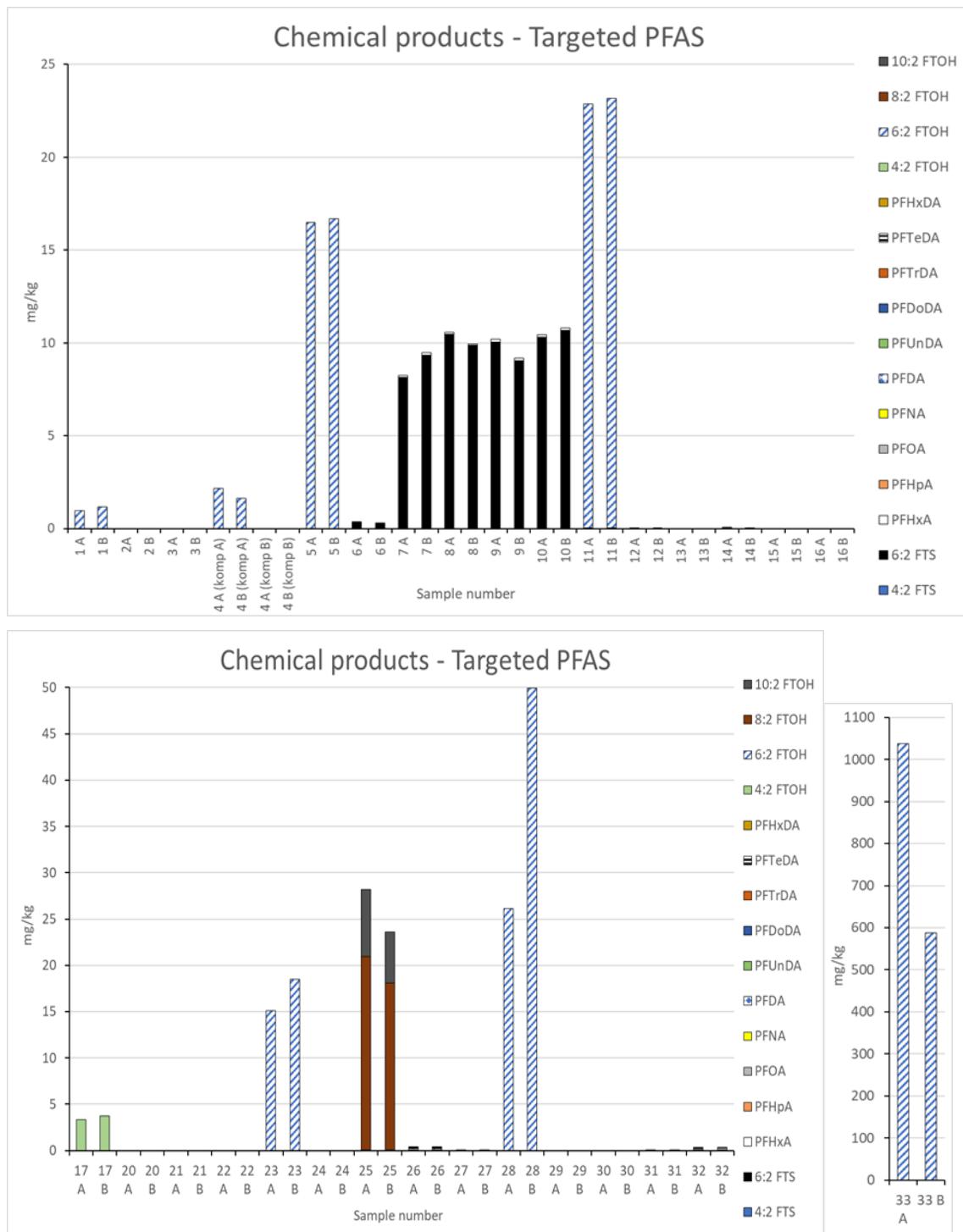


Figure 1: Concentrations of targeted PFASs detected in chemical products (mg/kg). Sample 33 shown separately due to high concentrations of 6:2 FTOH.

The TF in chemical products ranged from <0.03 to 85 680 mg F/kg sample. The TF was below detection limit (<LOD) in 60 % of the chemical products. The highest amount of TF was measured in two of the ski wax samples (sample 26 and 32) and in one impregnation (sample 11), all exceeding 25 000 mg F/kg sample. See the separate sub-chapter below for comparison with calculated fluorine content.

3.2 Textiles

The concentration for the individual PFAS measured in textile products are presented in Table A2 in Appendix 1.

17 of the targeted 36 PFASs were detected in at least one of the 31 textile samples (Figure 2). However, none of the measured concentrations exceeded the limit values according to the POPs Regulation. PFCAs were the dominating group of the detected PFAS, but the highest individual concentrations were found for 6:2 FTOH (sample 46 and 66) and PFTeDA (sample 55 and 67), all in jackets (Figure 2). The highest concentrations of the targeted PFASs (sum of targeted PFASs) were detected in the same four jackets (sample 46, 67, 55 and 66). The reported EOFs in textile samples (<0.07-61.8 mg F/kg sample) were significantly lower than EOF values in the chemical products. Despite that, only 23 % of the textile samples were below the detection limit (<LOD) for EOF compared to 61 % of the chemical products.

The highest EOFs in textile samples were measured in four jackets (sample 46, 63, 64, 66) and one shoe (sample 61). See the separate sub-chapter below for comparison with calculated fluorine content.

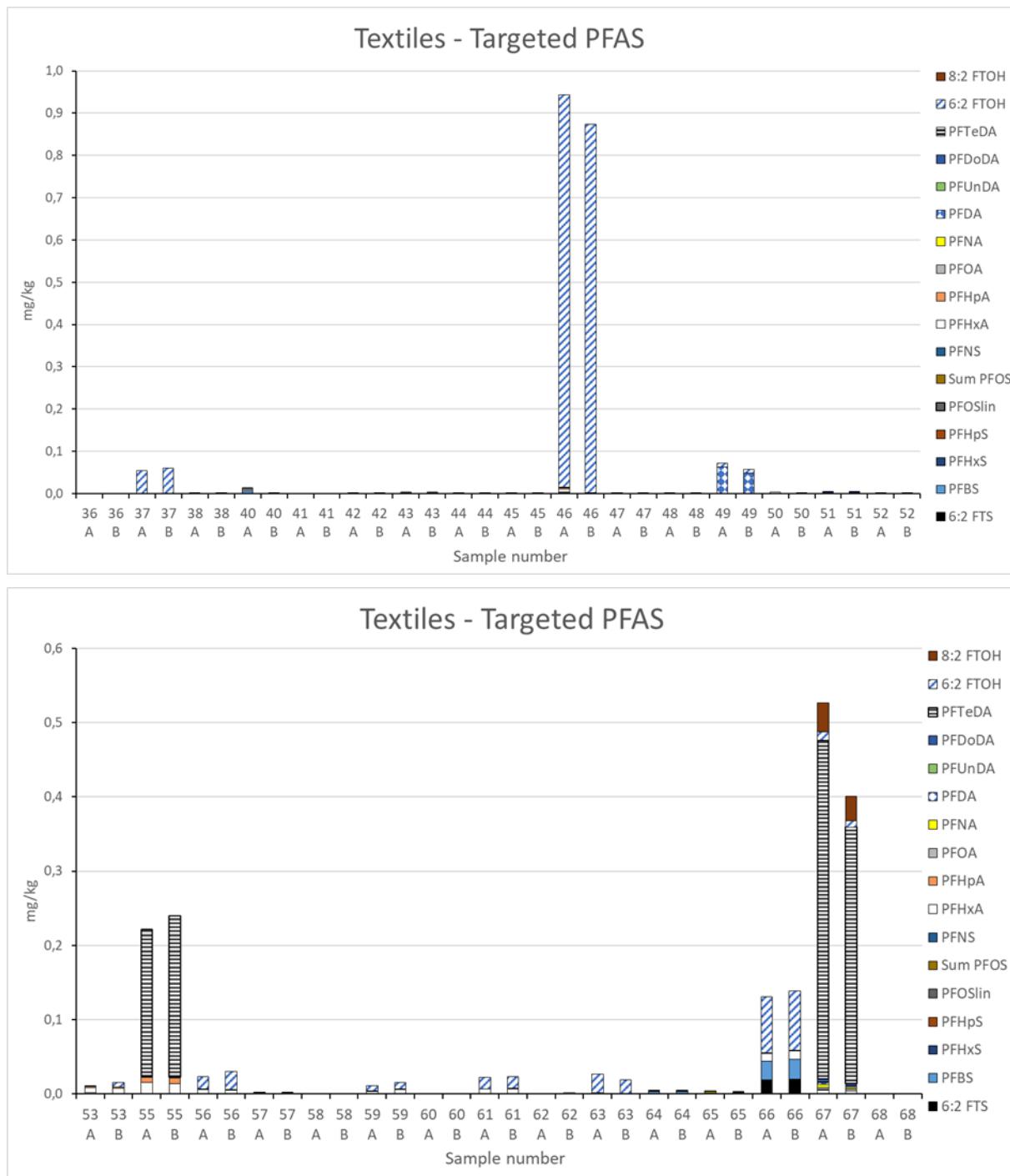


Figure 2: Concentrations of targeted PFASs detected in the textile samples (mg/kg).

The PFAS concentrations in the two sub-samples from each textile product that were analysed in parallel had low variability, with the exception for one glove (sample 40) and one shoe (sample 50). For the glove, the concentrations of the detected PFAS were higher in the sub-sample taken from the palm compared to the sub-sample from the back of the palm. For the shoe, only PFHxA was detected and then to higher concentrations in sub-sample A compared to sub-sample B.

3.3 Comparison of EOF and calculated fluorine content

The measured EOF/TF amounts were compared to a calculated fluorine content based on the target PFAS concentrations (Figure 3 A-B (products) and Figure 4 A-B (textiles)). The amount of fluorine in PFAS compounds is on average 65 % of the total molecular weight. The calculated fluorine content in the individual samples presented in Figure 3 and Figure 4 is therefore obtained by multiplying the sum targeted PFAS concentration in the individual samples with 65 %.

The results show that when EOF/TF is above detection limit, the calculated fluorine content from target PFAS is marginal to the measured EOF/TF. The calculated fluorine content explains 0-18 % of the measured EOF/TF for textile samples and 0-35 % for chemical product samples. The highest contribution for textile samples were found for samples 46, 55 and 67 (jackets) that had the highest PFAS concentrations (4.5-18 %). Of the chemical products only the sample with the highest measured concentrations (sample 33) had a significant contribution to measured TF.

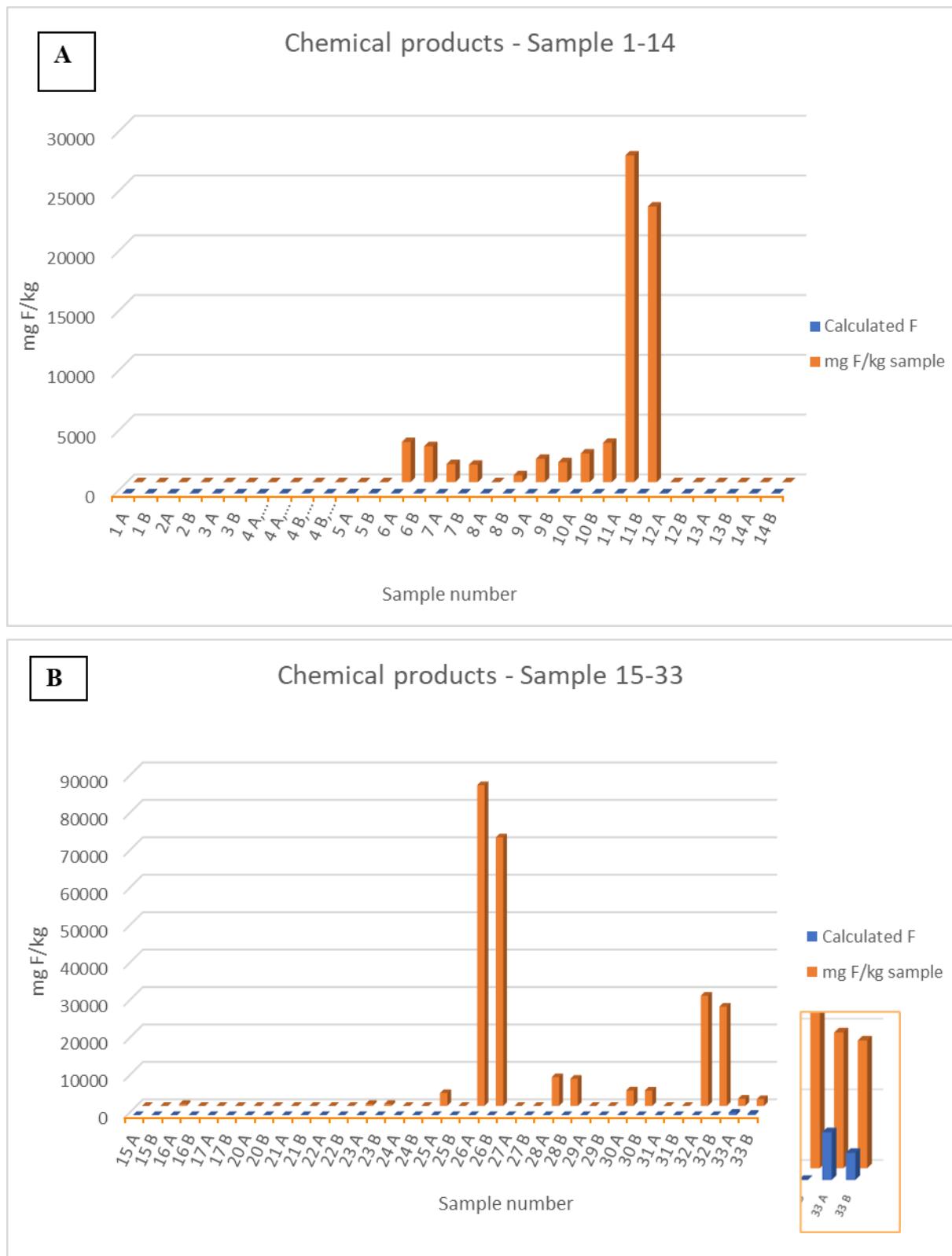


Figure 3: A (upper) and B (lower): Comparison of calculated fluorine concentrations based on target PFAS analyses (blue bar) and measured TF/EOF (orange bar) in extracts from chemical products. Upper right corner in figure 3 B shows sample 33 (textile impregnation) on a smaller scale.

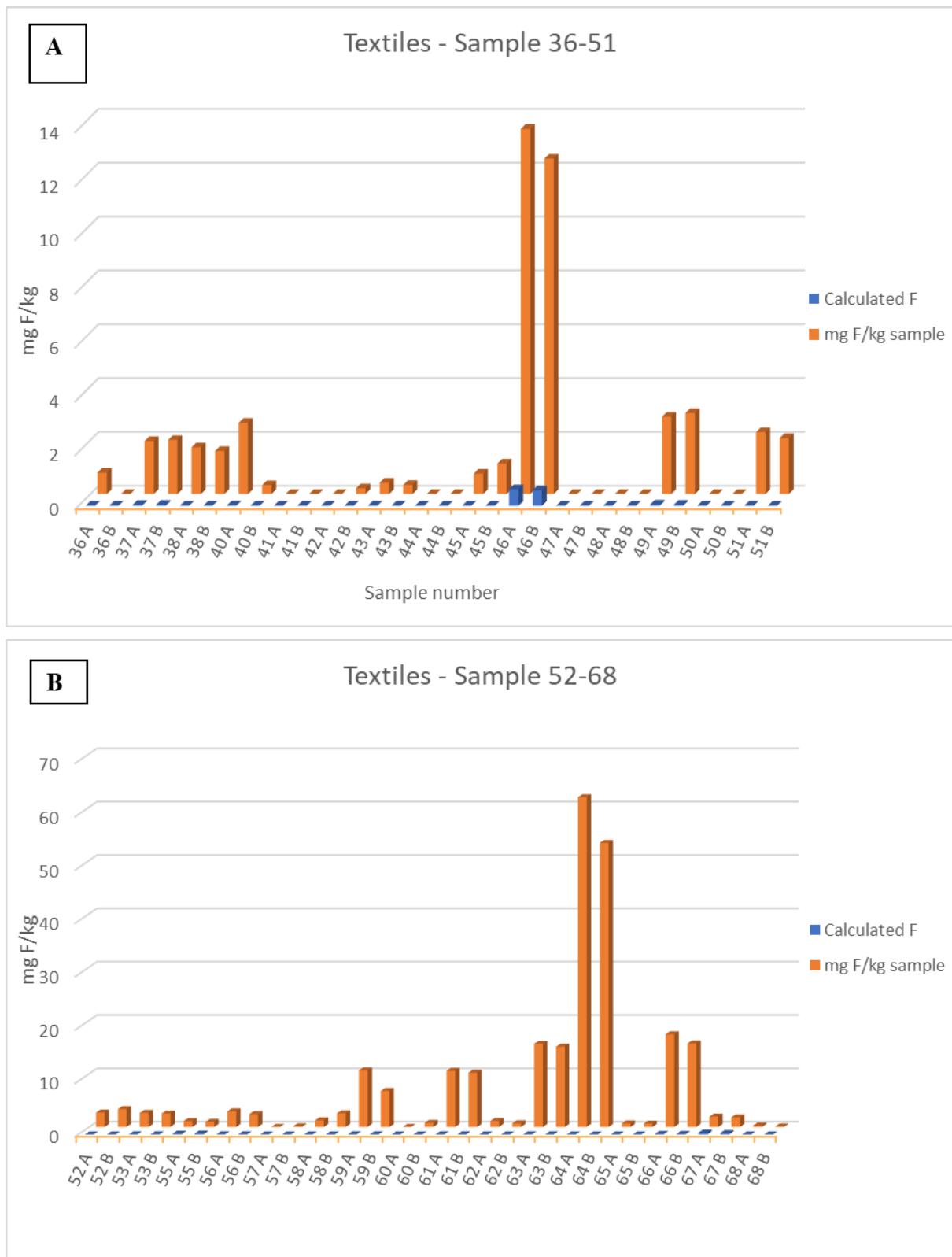


Figure 4: A (upper) and B (lower): Comparison of calculated fluorine concentrations based on target PFAS analyses (blue bar) and measured TF/EOF (orange bar) in the extracts from textiles.

4 Discussion

The highest concentrations of individual PFAS and TF/EOF were measured in chemical products, specifically in ski wax and textile impregnation samples. The highest individual PFAS was 6:2 FTOH, but also 8:2 and 10:2 FTOH were measured in high concentrations in one ski wax sample and 6:2 FTS in four of the five fire-fighting foams (8.1-10.6 mg/kg). The 6:2 FTS concentrations in the four fire-fighting foam (sample 7-10) were comparable to detected concentrations in KEMI (2015). A concentration equal to 10 mg/kg equals 0.001 %, which is the reporting limit for PFOS in fire-fighting foam. This means that the samples above 10 mg/kg are above the reporting limit value. The percentage of 6:2 FTS in the fire-fighting foams in this study are maximum 0.001 % and are probably residues from the manufacturing process or due to storage degradation (KEMI, 2015).

In total, eight different ski waxes were analyzed, and PFAS were detected in six of them. The largest number of PFAS compounds and also the highest TF concentration was found in ski wax (sample 26), where ten of the eleven analyzed PFCAs² were detected. However, the PFOA concentration in this sample was below the limit value and the concentrations of the detected PFAS compounds in this sample were lower than in sample 25 and 32 that had PFOA concentrations higher than the limit value. Despite elevated concentrations of individual PFAS in two of the ski waxes (25 and 32), the calculated fluorine content from the detected PFAS was marginal compared to the measured TF. The sample with the highest EOF (sample 26) is not associated with elevated concentrations of the targeted PFASs. This shows that ski waxes comprise a wide range of fluorinated compounds, also those not targeted in this study.

Among the textile samples, the highest concentrations were measured in four jackets. The predominant PFAS in these jackets was 6:2 FTOH (sample 46 and 66) and PFTeDA (sample 55 and 67). None of the targeted PFASs in textile samples were however above the reporting limit. PFSAs and PFOS were most prominent in textile samples. For the jacket samples 46, 55 and 67, the calculated fluorine content from detected PFAS compounds explain 4.5-18 % of the measured EOF. For all the other textile samples, the targeted PFASs results only explain 0-1 % of the fluorine content from EOF.

For samples where the measured EOF is higher than the calculated fluorine content based on the target PFAS analyses, there is an unknown source of organic fluorine in the sample. This can originate from not analysed or even unknown PFASs and/or other fluorine-containing organic compounds. Not analysed PFASs, i.e. not part of the targeted analysis in this study, could for example be acrylates, polyfluoroalkyl phosphate ester (PAPs) and long chain FTOHs.

The results of this study reflect the complexity of the PFAS group. There are PFAS reported for both chemical products and textiles that are not part of the regulations, and EOF results reveal the presence of organic fluorine of unknown origin.

² PFCA detected: PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoDA, PFTrDA, PFTeDA and PFHxDA

5 References

- Blom, C., Hanssen, L. (2015) Analysis of per- and polyfluorinated substances in articles. Copenhagen, Nordic Council of Ministers (Nordiske Arbejdspapirer, 2015:911).
- Fang, S, Plassmann, MM and Cousins, IT. (2020) Levels of per- and polyfluoroalkyl substances (PFAS) in ski wax products on the market in 2019 indicate no changes in formulation. Environ. Sci.: Processes Impacts, 22: 2142.
- Glüge, J., M. Scheringer, I.T. Cousins, J.C. DeWitt, G. Goldenman, D. Herzke, R. Lohmann, C.A. Ng, X. Trier, and Z. Wang. (2020) An overview of the uses of per- and polyfluoroalkyl substances (PFAS). Environ Sci Process Impacts, 22(12): 2345-2373.
- Hanssen, L., Dudarev, A., Huber, S., Odland, J.O., Nieboer, E., Sandanger, T.M. (2013) Partition of perfluoroalkyl substances (PFASs) in whole blood and plasma, assessed in maternal and umbilical cord samples from inhabitants of arctic Russia and Uzbekistan. Sci. Total Environ., 447: 430-437.
- Kärrman, A., Wang, T., Kallenborn R., (2019) PFASs in the Nordic environment Screening of Poly- and Perfluoroalkyl Substances (PFASs) and Extractable Organic Fluorine (EOF) in the Nordic Environment. Copenhagen, Nordic Council of Ministers (TemaNord, 2019:515).
- KEMI (2015) Chemical Analysis of selected Fire-fighting Foams in the Swedish Market (PM 6/15). Retrieved from URL:
<https://www.kemi.se/download/18.6df1d3df171c243fb23960dd/1591097411709/pm-6-15.pdf>
- KEMI (2021). Interlaboratory Comparison of Extractable Organofluorine (EOF). Analysis of water, effluent and sludge (PM 5/21). Retrieved from URL:
<https://www.kemi.se/en/publications/pms/2021/pm-5-21-interlaboratory-comparison-of-extractable-organofluorine-eof.pdf>
- Powley, CR, George SW, Ryan TW, Buck RC. (2005) Matrix effect-free analytical methods for determination of perfluorinated carboxylic acids in environmental matrixes. Anal. Chem., 77: 6353–6358.
- Wiener, B., Yeung, L., Marchington, E.B., D'Agostino, L.A. (2013) Organic fluorine content in aqueous film forming foams (AFFFs) and biodegradation of the foam component 6: 2 fluorotelomer-mercaptoalkylamido sulfonate (6: 2 FTSAs). Environ. Chemistry, 10(6): 486.

Appendix 1

Table A1: PFAS concentrations in sub-samples of chemical products (mg/kg).

mg/kg	4:2 FTS	6:2 FTS	8:2 FTS	10:2 FTS	PFBS	PFPS	PFHxS	PFHpS	PFOSlin	Sum PFOS	PFNS	PFDS
120118/26 B	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/27 A	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/27 B	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/28 A	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/28 B	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/29 A	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/29 B	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/30 A	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/30 B	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/31 A	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/31 B	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/32 A	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/32 B	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/33 A	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077
120118/33 B	<0.000037	<0.000037	<0.000033	<0.000033	<0.000035	<0.000052	<0.000070	<0.000131	<0.000077	<0.000077	<0.000077	<0.000077

Red	PFOA-related substances	max. 1 mg/kg
Pink	PFOA & PFOA-salts	max 0.025 mg/kg or 1 mg/kg for products containing PTFE micro powder
Orange	PFOS and its derivates	max. 10 mg/kg

mg/kg	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFTrDA	PFTeDA	PFHxDA	PFOcDA
120118/29 A	<0.000218	<0.000218	<0.000218	<0.000245	<0.000114	<0.000112	<0.000016	<0.000045	<0.000045	<0.000045	<0.000059
120118/29 B	<0.000218	<0.000218	<0.000218	<0.000245	<0.000114	<0.000112	<0.000016	<0.000045	<0.000045	<0.000045	<0.000059
120118/30 A	<0.000218	<0.000218	<0.000218	<0.000245	<0.000114	<0.000112	<0.000016	<0.000045	<0.000045	<0.000045	<0.000059
120118/30 B	<0.000218	<0.000218	<0.000218	<0.000245	<0.000114	<0.000112	<0.000016	<0.000045	<0.000045	<0.000045	<0.000059
120118/31 A	<0.000218	0.0004	0.0043	<0.000245	0.0016	<0.000112	<0.000016	<0.000045	<0.000045	<0.000045	<0.000059
120118/31 B	<0.000218	0.0003	0.0050	<0.000245	0.0019	<0.000112	<0.000016	<0.000045	<0.000045	<0.000045	<0.000059
120118/32 A	0.0452	0.0172	0.166	0.0181	0.0122	0.0044	<0.000016	<0.000045	<0.000045	<0.000045	<0.000059
120118/32 B	0.0517	0.0222	0.199	0.0155	0.0131	0.0032	<0.000016	<0.000045	<0.000045	<0.000045	<0.000059
120118/33 A	0.0389	<0.000218	<0.000218	<0.000245	<0.000114	<0.000112	<0.000016	<0.000045	<0.000045	<0.000045	<0.000059
120118/33 B	0.0206	<0.000218	<0.000218	<0.000245	<0.000114	<0.000112	<0.000016	<0.000045	<0.000045	<0.000045	<0.000059

Red	PFOA-related substances	max. 1 mg/kg
Pink	PFOA & PFOA-salts	max 0.025 mg/kg or 1 mg/kg for products containing PTFE micro powder
Orange	PFOS and its derivates	max. 10 mg/kg

mg/kg	FOSA	N-Me-FOSAA	N-Et-FOSAA	4x3 PFECA	PFECHS	GenX	4:2 FTOH	6:2 FTOH	8:2 FTOH	10:2 FTOH	N-Me-FOSA	N-Et-FOSA	N-Me-FOSE	N-Et-FOSE	mg F/kg sample
120118/27 A	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	<0.0245	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	<0.03
120118/27 B	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	<0.0245	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	<0.03
120118/28 A	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	26.2	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	7677
120118/28 B	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	49.9	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	7217
120118/29 A	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	<0.0245	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	<0.03
120118/29 B	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	<0.0245	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	<0.03
120118/30 A	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	<0.0245	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	4126
120118/30 B	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	<0.0245	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	4084
120118/31 A	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	<0.0245	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	<0.03
120118/31 B	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	<0.0245	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	<0.03
120118/32 A	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	<0.0245	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	29430
120118/32 B	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	<0.0245	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	26522
120118/33 A	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	1038	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	1920
120118/33 B	<0.000394	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0226	588	<0.0477	<0.0402	<0.0137	<0.0137	<0.0458	<0.0394	1808

Red	PFOA-related substances	max. 1 mg/kg
Pink	PFOA & PFOA-salts	max 0.025 mg/kg or 1 mg/kg for products containing PTFE micro powder
Orange	PFOS and its derivates	max. 10 mg/kg

Table A.2: PFAS concentrations in sub-samples of textiles (mg/kg).

mg/kg	4:2 FTS	6:2 FTS	8:2 FTS	10:2 FTS	PFBS	PFPS	PFHxS	PFHpS	PFOSin	Sum PFOS	PFNS	PFDS
120118/65 A	<0.000009	<0.000009	<0.000007	<0.000007	<0.000008	<0.000012	<0.000012	<0.000016	0.00011	0.00011	<0.000018	<0.000018
120118/65 B	<0.000009	<0.000009	<0.000007	<0.000007	<0.000008	<0.000012	<0.000012	<0.000016	0.00010	0.00010	<0.000018	<0.000018
120118/66 A	<0.000009	0.01865	<0.000007	<0.000007	0.02530	<0.000012	<0.000012	<0.000016	0.00004	0.00004	<0.000018	<0.000018
120118/66 B	<0.000009	0.02009	<0.000007	<0.000007	0.02624	<0.000012	<0.000012	<0.000016	0.00004	0.00004	<0.000018	<0.000018
120118/67 A	<0.000009	<0.000009	<0.000007	<0.000007	<0.000008	<0.000012	<0.000012	<0.000016	0.00011	0.00011	<0.000018	<0.000018
120118/67 B	<0.000009	<0.000009	<0.000007	<0.000007	<0.000008	<0.000012	<0.000012	<0.000016	0.00011	0.00011	<0.000018	<0.000018
120118/68 A	<0.000009	<0.000009	<0.000007	<0.000007	<0.000008	<0.000012	<0.000012	<0.000016	0.00007	0.00007	<0.000018	<0.000018
120118/68 B	<0.000009	<0.000009	<0.000007	<0.000007	<0.000008	<0.000012	<0.000012	<0.000016	0.00008	0.00008	<0.000018	<0.000018

Red	PFOA-related substances	max. 1 mg/kg
Pink	PFOA & PFOA-salts	max 0.025 mg/kg or 1 mg/kg for products containing PTFE micro powder
Orange	PFOS and its derivates	max. 10 mg/kg

mg/kg	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFTrDA	PFTeDA	PFHxDA	PFOcDA
120118/36 A	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/36 B	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/37 A	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/37 B	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/38 A	0.000812	<0.00005	0.000129	0.000116	0.000019	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/38 B	0.000703	<0.00005	0.000136	0.000117	0.000031	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/40 A	0.003372	<0.00005	0.002377	0.000071	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/40 B	0.000220	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/41 A	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/41 B	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/42 A	<0.00005	<0.00005	0.00008	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/42 B	<0.00005	<0.00005	0.00030	0.000444	0.000119	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/43 A	0.002544	0.00104	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/43 B	0.002310	0.00120	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/44 A	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/44 B	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/45 A	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	0.000053	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/45 B	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	0.000076	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/46 A	0.008184	0.00247	0.00046	0.000354	<0.000026	0.000071	0.00006	<0.00001	<0.00001	<0.00005	<0.0001
120118/46 B	0.001055	0.00024	0.00008	0.000091	<0.000026	0.000036	0.00005	<0.00001	<0.00001	<0.00005	<0.0001
120118/47 A	0.000040	<0.00005	0.00011	0.000070	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/47 B	0.000036	<0.00005	0.00008	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/48 A	0.000076	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/48 B	0.000089	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/49 A	0.000095	<0.00005	0.00008	<0.000056	0.062480	0.000034	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/49 B	0.000099	<0.00005	0.00012	<0.000056	0.047746	0.000035	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001

mg/kg	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFTrDA	PFTeDA	PFHxDA	PFOcDA
120118/50 A	0.002918	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/50 B	0.000896	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/51 A	<0.00005	<0.00005	0.00022	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/51 B	<0.00005	<0.00005	0.00016	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/52 A	0.000307	<0.00005	0.00017	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/52 B	0.000267	<0.00005	0.00016	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/53 A	0.007128	0.00174	0.00011	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/53 B	0.007199	0.00169	0.00011	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/55 A	0.015303	0.00733	0.00041	0.000289	0.000487	0.000109	0.00050	<0.00001	0.19685	<0.00005	<0.0001
120118/55 B	0.014177	0.00711	0.00037	0.000205	0.000458	0.000144	0.00050	<0.00001	0.21661	<0.00005	<0.0001
120118/56 A	0.006356	0.00033	0.00022	0.000102	0.000085	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/56 B	0.005403	0.00035	0.00021	0.000103	0.000101	0.000036	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/57 A	0.000240	0.00028	0.00064	<0.000056	0.000065	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/57 B	0.000239	0.00027	0.00061	<0.000056	0.000065	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/58 A	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/58 B	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/59 A	0.003320	0.00048	0.00010	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/59 B	0.005741	0.00070	0.00009	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/60 A	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/60 B	<0.00005	<0.00005	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/61 A	0.006595	0.00053	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/61 B	0.007167	0.00061	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/62 A	0.000266	<0.00005	0.00017	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/62 B	0.000400	<0.00005	0.00018	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/63 A	0.000763	0.00007	0.00012	0.000070	0.000037	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/63 B	0.000487	<0.00005	0.00007	0.000057	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/64 A	0.000737	<0.00005	0.00010	0.000075	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/64 B	0.000806	<0.00005	0.00013	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001

mg/kg	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFTrDA	PFTeDA	PFHxDA	PFOcDA
120118/65 A	0.000240	0.00035	0.00086	0.001231	<0.000026	0.000155	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/65 B	0.000211	0.00025	0.00077	0.000931	<0.000026	0.000116	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/66 A	0.010973	0.00076	<0.00005	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/66 B	0.011403	0.00082	0.00012	0.000159	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/67 A	0.005112	0.00050	0.00272	0.004576	0.002214	0.000990	0.00403	<0.00001	0.45571	<0.00005	<0.0001
120118/67 B	0.003729	0.00035	0.00229	0.002269	0.001535	0.000610	0.00336	<0.00001	0.34487	<0.00005	<0.0001
120118/68 A	0.000025	<0.00005	0.00014	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001
120118/68 B	0.000028	<0.00005	0.00015	<0.000056	<0.000026	<0.000026	<0.00001	<0.00001	<0.00001	<0.00005	<0.0001

Red	PFOA-related substances	max. 1 mg/kg
Pink	PFOA & PFOA-salts	max 0.025 mg/kg or 1 mg/kg for products containing PTFE micro powder
Orange	PFOS and its derivates	max. 10 mg/kg

mg/kg	FOSA	N-Me-FOSAA	N-Et-FOSAA	4x3 PFECA	PFECHS	GenX	4:2 FTOH	6:2 FTOH	8:2 FTOH	10:2 FTOH	N-Me-FOSA	N-Et-FOSA	N-Me-FOSE	N-Et-FOSE	mg F/kg sample
120118/66 A	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.00226	0.07560	<0.00477	<0.00402	<0.00137	<0.00137	<0.00458	<0.00394	17.4
120118/66 B	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.00226	0.08024	<0.00477	<0.00402	<0.00137	<0.00137	<0.00458	<0.00394	15.6
120118/67 A	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.00226	0.01154	0.0391	<0.00402	<0.00137	<0.00137	<0.00458	<0.00394	1.95
120118/67 B	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.00226	0.00884	0.0330	<0.00402	<0.00137	<0.00137	<0.00458	<0.00394	1.81
120118/68 A	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.00226	<0.00245	<0.00477	<0.00402	<0.00137	<0.00137	<0.00458	<0.00394	0.264
120118/68 B	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.00226	<0.00245	<0.00477	<0.00402	<0.00137	<0.00137	<0.00458	<0.00394	0.089

Red	PFOA-related compounds	max. 1 mg/kg
Pink	PFOA & PFOA-salts	max 0.025 mg/kg or 1 mg/kg for products containing PTFE micro powder
Orange	PFOS and its derivates	max. 10 mg/kg

Appendix 2

Details about the quality assurance for EOF

The background fluoride levels of the combustion ion chromatography (CIC) varied from day to day; the background fluoride indicated as instrumental (boat) blank was found to be 8 ng F (geomean of 9 replicates). The analysis of organofluorine in samples was initiated only when the RSD of three sequential combustion blanks (empty sample boat analysis) was below 5 %. An additional combustion blank was run after every fifth samples to monitor for carry-over. The combustion blank response (average of combustion blanks before and after the sample) was subtracted from the sample responses, before further data processing. A PFOA standard of 240 ng F/mL was injected in between every 10 samples to evaluate the stability of the system; the measured mean value of the standard injection was 251 ng F/mL (R.S.D.: 13 %, n=10); intra-day variability: at most 14 % and inter-day variability: 15 %). The method has been evaluated in the “Interlaboratory Comparison of Extractable Organofluorine (EOF) – Analysis of water, effluent and sludge” (KEMI. 2021).

Uncertainties of EOF results

As the chemical products were not extracted but only diluted with organic solvents and no separation of inorganic fluoride were done before analysis, this analysis should be regarded as total fluorine (TF) analysis for the chemical products. The contribution from inorganic fluoride is however assumed to be small, based on previous studies. For example, Weiner et al. 2013 conducted a mass balance analysis of aqueous film forming foam (AFFF) and showed that inorganic fluoride contributed to at most 8 % of to the total fluorine of the AFFF samples. In addition, while inorganic fluoride might be co-extracted during extraction of a sample, results from previous studies have suggested that inorganic fluoride is not extracted from the sample when organic solvents (methanol or acetonitrile) are used (KEMI, 2021). Results from a spike test of 1 µg of inorganic fluoride on paper followed by extraction with either methanol or acetonitrile, showed that none of the extracts had detectable fluoride signal (<5 ng). Therefore, it is assumed that no inorganic fluoride was extracted from the textile samples in this study.

As for EOF analysis, when different solvents were used for extraction, then the measurement of EOF even in the same product may be different, because of different extraction efficiencies of different solvents on different compounds. Therefore, special care should be given onto what extraction solvents was/were used when comparing EOF results across studies.



Swedish Chemicals Agency

Box 2, SE-172 13 Sundbyberg
+46 8 519 41 100

Visitors' and delivery address
Esplanaden 3A, Sundbyberg

kemi@kemi.se
www.kemikalieinspektionen.se