

Survey, screening and analyses of PFCs in consumer products

Dorte Herzke (NILU), Stefan Posner and Elisabeth Olsson

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Swerea IVF AB
Box 104
SE-431 22 Mölndal
Sweden
Telephone +46 (0)31 706 60 00
Fax +46 (0)31 27 61 30
www.swereaivf.se

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Preface

The Norwegian Pollution Control Authority (SFT) has commissioned a survey carried out by Swerea IVF (Sweden) together with Norwegian Institute for Air Research (NILU) that aims to identify and quantify possible sources of PFC in Norway in industrial manufacturing and applications used by the Norwegian population in daily life.

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Summary

Perfluorinated compounds (PFC) are used in numerous industrial and consumer products because of their special chemical properties, for instance the ability to repel both water and oil. A broad variety of PFCs are introduced into the Norwegian market both from industrial use like fire fighting foams and paints and in treated consumer products like textiles and coated paper. Our present knowledge of the exact chemical PFC compositions in preparations using perfluorinated compounds is limited. This makes it challenging to evaluate human exposure and the amounts of waste containing treated products. It is a growing concern that these potentially harmful compounds now are found as widespread global environmental pollutants.

SFT (The Norwegian Pollution Control Authority) has commissioned Swerea IVF (Sweden), together with NILU (Norway) to screen possible sources of PFC in Norway in industrial manufacturing and household applications. The results of the screening are summarised in this brief report.

Industrial samples and samples for personal use were collected mainly in Norway, but also in Sweden. Only five of the 34 analysed industrial materials and consumer products contained none of the polyfluorinated substances that were analysed but this does not exclude the occurrence of unknown PFCs. Notable is that PFOS, which is banned in Norway since 2007, was found in amounts close to or exceeding the regulation¹ in 4 of the 34 analysed products, all within the leather or carpet product groups.

¹ Regulation EC No 552/2009 of 22 June 2009 amending Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) as regards Annex XVII , p.53

Introduction

1.2 Information on uses of PFCs in application areas

PFCs are used in several industrial branches, but they also occur in a large range of consumer products. Due to their extraordinary properties (chemically inert, non-wetting, very slippery, nontoxic, nonstick, highly fire resistant, very high temperature ratings, highly weather resistant) they are applied in fluoropolymer coated cookware, sports clothing, extreme weather military uniforms, food handling equipment, medical equipment, motor oil additives, fire fighting foams, paint and ink as well as water repellent products.

PFCs are a chemical family consisting of a carbon backbone fully surrounded by fluorine, which makes them impervious to heat, acid or other forces that typically break down chemical compounds.

Fluorotelomers are a range of chemicals with similar fluoride carbon backbones connected to a $-\text{CH}_2\text{-CH}_2-$ chain and different functional heads. They are industrially produced applying a telomerisation process, coupling tetrafluoroethene, which leads to straight-chained products with an even number of carbon atoms. Fluorotelomers are probably the most commonly used perfluorinated substances in products. The hydroxyl group as functional group will give fluorotelomer alcohols (FTOH). They are used to treat paper to improve its moisture and oil barrier properties. FTOHs are also used in waterproof outdoor clothing and in waterproofing agents for textiles. Fluorotelomeralcohols are manufactured as a raw material used in the synthesis of fluorotelomer-based surfactants and polymeric products. The manufacture of FTOHs usually result in a mixture containing six to twelve fluorinated carbon congeners, the 8:2 FTOH being the dominant one. Release of the volatile FTOHs may occur all along the supply chain from production, application into consumer use and disposal. They have the potential to form stable perfluorinated carboxylates such as PFOA and PFNA (PFCAs).

The general chemical structure of perfluorinated sulphonates (PFS) contains a perfluorinated carbon chain connected to a sulphonate group. In addition to this, fluorotelomer sulphonates (FTSs) contain two carbon atoms adjacent to the functional group that are not fluorinated. FTS are used among other fluorotelomers in fire fighting foam for their film forming properties and the ability to decrease fuel absorption. These foams are especially useful for major fires, e.g. chemical fires (Stockholm Convention on POPs Review committee, 2009). The quantities in the foams are low, but the foams are released directly into the environment. FTS is also used as a component in more complex structures (e.g. in water proofing agents) and as a substitute for perfluorooctane sulfonate (PFOS).

Fluorinated surfactants are used in very low levels in a large number of cleaning products, e.g. polish, waxes, all-purpose cleaners, window cleaners etc. Their use is widespread and directly released into wastewater.

Perfluorinated carboxylates (PFCA) is another important PFC group. The main use of perfluorooctanoate (PFOA) is as a process aid in the manufacture of various fluoropolymers, such as polytetrafluoroethylene (PTFE). These polymers

are among other things, used to coat cookware intended for stovetop cooking and baking.

The substances PFOS and PFOA are part of a group of old-generation PFCs which will be used to a lesser extent in the future due to their potential hazards. These hazards have resulted and will result in a number of international legislative bans worldwide. New generations of PFCs are developed continuously and applied in industrial amounts already.

Polyfluorinated sulphonamides are considered the most important PFCs because of their intentional industrial production and global distribution. PFOS and related substances are well known degradation products from substituted sulphonamides that are used commercially for numerous applications. However, the potential toxicity, extreme persistence and accumulation potential of their degradation product PFOS has resulted in prohibition for new uses or import by chemical regulatory authorities worldwide based on international restrictions by the United Nations Environmental Programme (UNEP) Stockholm convention, where PFOS is going to be classified as a POP (Persistent Organic Pollutant)

Other polyfluorinated chemicals, such as perfluoroalkylsulfonic acid derivatives (e.g. PFOSF), are probably used as paper additives/coatings to prevent oil from soaking through or staining the paper.

The total global production volume today is not known but was estimated to 5,000 tonnes per year in 2000 with new production sites rising in Asia.

Ongoing follow up by the Organisation for Economic Cooperation and Development (OECD) has identified several hundreds of perfluorinated precursors in use on the international market (OECD, 2007). Little information about them is available. However, the OECD encourage the development of an internet platform on sustainable (“green”) chemistry, that will soon be posted along with harmonized bioaccumulation test methods for PFCs that will be hosted by OECD. The first results from these activities will be presented by Christmas 2009 in a public report issued by OECD.

1.3 PFC production processes

There are two main production processes for PFCs: electrochemical fluorination (ECF) and telomerisation. In the electrochemical fluorination process, a technical mixture of hydrocarbons (different carbon chain lengths including branched isomers) with a functional group is subjected to fluorination, leading to a mixture of perfluorinated products with the same homologue and isomer pattern. Telomerisation involves coupling tetrafluoroethene, which leads to straight-chained products with an even number of carbon atoms. Fluorotelomer products often possess two carbon atoms adjacent to the functional group which are not fluorinated that yields linear, even carbon number substances. Telomers are produced and used commercially as mixtures, in which the typical length of the chains is between four and eighteen carbon atoms. Polyfluorinated compounds in the form of fluorinated telomers and PFOS, for example, can be further reacted and will then occur in other chemical compounds, e.g. acrylate polymers. This means that perfluorinated compounds and fluorinated telomers may occur in a large number of different chemical compounds either added as final treatments,

impurities and unreacted monomers of the production process or chemically bound to the polymeric structure.

From 1947 until 2002, the ECF process was used to produce the majority of PFO (Prevedouros et al., 2006).

2. Selection of samples for screening

In this survey, sample candidates were identified in different ways; either by having or giving certain properties that are common for perfluorinated chemicals (e.g water repellent, stain resistant, anti-grease, non-stick, surfactant), by their previous known high concentration of PFCs (Teflon table cloth, AFFF, water proofing agents) or by information from literature that production of these articles may include perfluorinated chemicals (epoxy resin board, semiconductor fabrication etc.). A number of product types were identified as potential PFC containing groups and several samples were collected in each group distributed as shown in table 1.

Table 1. Number of samples analysed in each group

Sample group	Number of samples
Waterproofing agents	5
Paint and inks	5
Impregnated products: Paper, textiles, leather and carpets	2+2+2+2
Non stick ware	6
Electronics	5
Fire fighting agents	5

The product group “waterproofing agents” includes different brands of water- and dirt-proofing agents and a lubricant. The product group “paint and inks” includes paint and printer ink. The sample group “impregnated products” include, besides office furniture textiles and table cloth, carpets, food packaging and leather. The group “electronics” includes light diodes, a Li-battery and several printed circuit boards (PCBs). The group “fire fighting agents” includes two powder foams and three aqueous film forming foams (AFFF).

Consumer products were collected mainly from Norway and some from Sweden. The group of electronics and some non-stick products were collected in Sweden. The samples purchased in Sweden were bought from retailers existing both in Norway and Sweden (e.g. Elgiganten, Ikea). Samples collected in Sweden were of known brands which are likely to be found also in Norway.

Industrial samples like AFFF, textiles and paint were acquired in Norway as well. The AFFF samples are for industrial use and not consumers products. See Appendix I for detailed list of samples.

The limited funding governed the restriction in the sample numbers. In total 34 consumer products could be tested, giving a snapshot of the distribution of PFCs on the Norwegian market. The found PFC concentrations are therefore not representative for all products of one segment but are a selection with focus on

potentially highly contaminated/treated materials available to the common consumer in Norway.

2.2 Content information

In some of the purchased samples (listed in table 2.) there is information available, on the package or in a SDS, about the use of PFCs.

Table 2. Number of samples analysed in each group

Sample id	Description	PFC content information on product
I1	Kiwi All Protector	Fluoroethen plast and polytetrafluorinated masses
I2	TF2 lubricant	Spray containing Teflon
I3	Rainguard; Boston	PFOS/PFOA free
I4	Fiber Protector	Without PFOS, containing fluoropolymers
I5	Granger XT Spray	Fluorochemical agent
NS1	Privilege pan	Teflon Pro Platinum non-stick coating, professional use
NS2	Tefal pan ø 28	PFOS/PFOA free
NS3	Rast camping pan	Teflon coated
NS4	IKEA pan	Teflon classic convenience
NS5	Eva cake form	SLIP-LET®
T2	Table cloth, Princess	Teflon fabric protector
C2	Carpet brown; Element	Teflon treated
B3	AFFF Arctic Foam 3% PFC-content; Solberg Scandinavia	
B4	AFFF Light water 1%; Solberg Scandinavia	
B5	AFFF Shtamex P	Fluorinated compound <10%

	3%, PRESTO Norge	
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3. Selection of PFCs for screening

A total of 29 different PFCs were analysed in all collected samples, shown in table 3. The substances are selected due to their well known and established analytical methods and the fact that several compounds have been analysed in previous studies.

Table 3. PFCs analysed in the samples

Abbreviation	Full name	CAS #	Detection method
Fluortelomer alcohols			
4:2 FTOH	4:2 Fluorotelomer alcohol	2043-47-2	GC-MS
6:2 FTOH	6:2 Fluorotelomer alcohol	647-42-7	GC-MS
8:2 FTOH	8:2 Fluorotelomer alcohol	678-39-7	GC-MS
10:2 FTOH	10:2 Fluorotelomer alcohol	865-86-1	GC-MS
Fluortelomer sulfonates			
6:2 FTS	6:2 Fluorotelomer sulfonate	27619-97-2	HPLC-MS
8:2 FTS	8:2 Fluorotelomer sulfonate	39108-34-4	HPLC-MS
Fluortelomer carboxylates			
6:2 FTUCA	6:2 Fluorotelomer unsaturated carboxylate	C ₈ H ₂ F ₁₂ O ₂ *	HPLC-MS
8:2 FTUCA	8:2 Fluorotelomer unsaturated carboxylate	70887-84-2	HPLC-MS
Perfluoro sulfonates			
PFBS	Perfluorobutane sulfonate	375-73-5	HPLC-MS
PFHxS	Perfluorohexane sulfonate	432-50-7	HPLC-MS
PFOS	Perfluorooctane sulfonate	1763-23-1	HPLC-MS
PFDCs	Perfluorodecane sulfonate	335-77-3	HPLC-MS
Perfluoro carboxylates			
PFBA	Perfluorobutanoate	375-22-4	HPLC-MS

PFPA	Perfluoropentanoate	2706-90-3	HPLC-MS
PFHxA	Perfluorohexanoate	307-24-4	HPLC-MS
PFHpA	Perfluoroheptanoate	375-85-9	HPLC-MS
PFOA	Perfluorooctanoate	335-67-1	HPLC-MS
PFNA	Perfluorononanoate	375-95-1	HPLC-MS
PFDoC	Perfluorodecanoate	335-76-2	HPLC-MS
PFUnA	Perfluoroundecanoate	2058-94-8	HPLC-MS
PFDoA	Perfluorododecanoate	307-55-1	HPLC-MS
PFTrA	Perfluorotetradecanoate	376-06-7	HPLC-MS
Fluoroctane sulfonamides/ sulfonamidoethanols			
PFOSA	Perfluorooctane sulfonamide	754-91-6	HPLC-MS
N-Me-FOSA	N-Methyl-heptadecafluorooctane sulfonamide	31506-32-8	HPLC-MS
N-Et-FOSA	N-Ethyl-heptadecafluorooctane sulfonamide	4151-50-2	HPLC-MS
N-Me-FOSE	N-Methyl-heptadecafluorooctane sulfonamidoethanol	24448-09-7	HPLC-MS
N-Et-FOSE	N-Ethyl-heptadecafluorooctane sulfonamidoethanol	1691-99-2	HPLC-MS

*No CAS number available

4. Analyses of PFC in consumer products and industrial materials

Both volatile and non-volatile PFCs were analysed using well established analytical methods applying GC/MS and LC/MS techniques. Quality assurance measures were applied to ensure best possible quality of the data. Liquid samples were diluted and extracted for both volatile and ionic PFCs, followed by a cleaning step with activated carbon. Solid samples were homogenized prior to extraction and then treated similar to liquid samples. All calculated concentrations are given in µg/L for liquids, µg/kg for solid materials like PCBs, µg/m² for textiles, paper and leather.

4.2 Analytical methods

At least two different methods were employed for the chemical analysis of the compounds of interest (volatile and non-volatile PFC). Both methods use a similar sample pre-treatment and extraction step differing in choice of solvent, internal standard and analytical instrument. The analytical method applied is similar to the standard method proposed to CEN for the determination of PFOS

(*Fel! Hittar inte referenskälla.*; CEN/TS 15986.)

Both Swerea IVF and NILU are part of the working group developing the method.

The analysed PFCs represent the extractable amount PFC by the applied method. Method blanks were included with each product type.

For an overview list of routine analysed PFCs at NILU see table 3.

4.3 Sample extraction

Solid samples were dried, homogenized and spiked with suitable internal standards. Extraction was performed with ultrasonic extraction with a polar organic solvent.

Liquid samples, were after addition of suitable internal standards, homogenized with vortex and centrifuged. The homogenate were extracted by liquid/liquid extraction with a polar solvent.

4.4 Cleanup and quantification

4.4.1 Neutral volatile compounds

The sample extracts were prepared according to a procedure which NILU has established for the analysis of volatile PFCs, involving treatment with activated carbon. For heavily contaminated samples like waterproofing agents and AFFF, an additional dilution step was necessary. Separation and quantification were done by gas chromatography coupled to mass spectrometry (GC/MS). Quantification was based on the use of ^{13}C -labeled standards. Special care was taken to control thermal compound degradation in the GC injector and on the GC column.

4.4.2 Ionic non volatile compounds

The sample extracts containing ionic PFCs were cleaned up according to a procedure which NILU has established for the analysis of PFOS and PFOA and other ionic PFCs with varying chain length. This includes purification with activated carbon. Separation and quantification were done by liquid chromatography coupled to a time-of-flight mass spectrometry (LC/TOF-MS). The applied method is in accordance with a standardized analytical method for PFOS developed now for CEN /EU. Quantification was based on the use of ^{13}C -labeled standards.

5. Results

All concentrations are related to the most common unit given in the EU regulation. PFCs in textiles, and, papers are given in $\mu\text{g}/\text{m}^2$, liquids in $\mu\text{g}/\text{L}$, and solid materials, like non-stick and electronics, in $\mu\text{g}/\text{kg}$.

Several groups of PFCs were analysed. For the ionic PFCs the following compounds were analysed:

- PFCAs with a chain length of C4 to C14

- PFSs with a chain length of C4 to C10
- Other PFCs like 6:2 and 8:2 FTS, PFOSA and some FOSE/As.

For the volatile PFCs FTOHs with C4, C6, C8 and C10 chains as well as FOSE/As were analysed.

PFBA, PFOS and PFHxS belonged to the most detected ionic PFCs (Figure 1). Even numbered PFCAs were detected more often than odd numbered PFCAs, indicating the dominance of the telomerisation production process. The detection of shorter chain PFS and PFCA indicates a substitution process within the PFC application in order to avoid PFOS use.

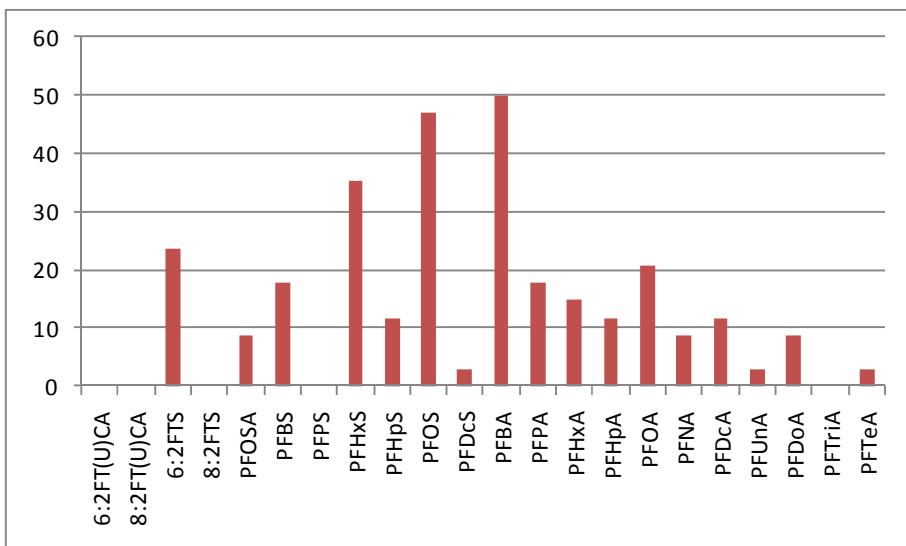


Figure 1: Percentage of detected ionic PFCs in the 34 analysed products

From the analysed volatile PFCs 6:2, 8:2 and 10:2 FTOH could be detected in more than 1/3 of all products.

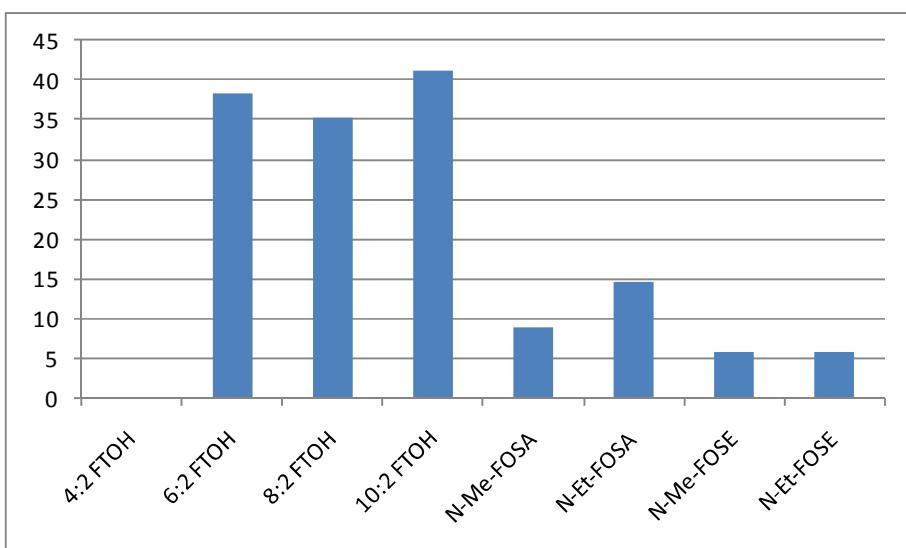


Figure 2: Percentage of detected volatile PFCs in the 34 analysed products

Analysed PFCs, either ionic or volatile, were found in all but 5 samples.

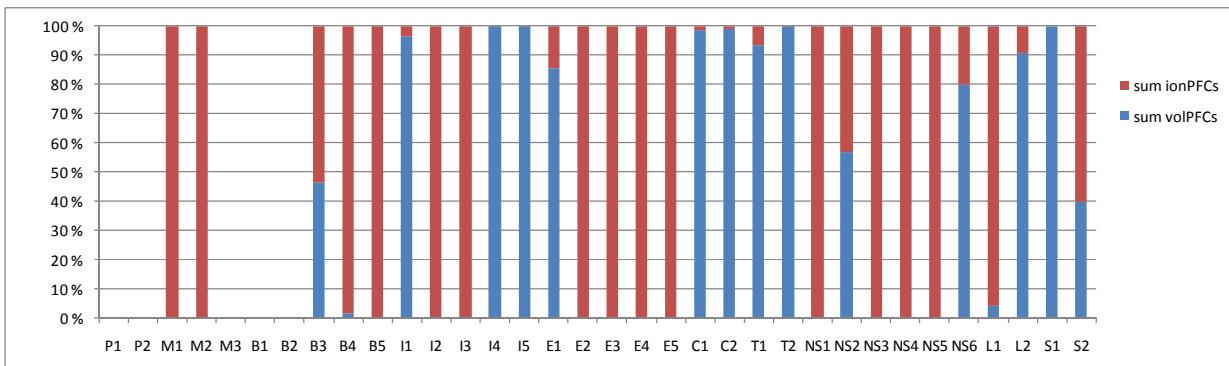


Figure 3: Overview about contribution of volatile and ionic PFCs in consumer products (product codes are listed in tables below).

5.2.1 Waterproofing agents and lubricants

Sample id	Description
I1	Kiwi All Protector
I2	TF2 lubricant
I3	Rainguard; Boston
I4	Fiber Protector
I5	Granger XT Spray

No PFOS was detected in any of the items of this product group. However, no investigated waterproofing agent/lubricant was free from PFCs. Two products, TF2 lubricant and Rainguard; Boston, only contained minor amounts of short chain PFBA and/or PFBS. Fiber Protector contained little ionic PFCs as well (PFBA and PFHpA) but high amounts of FTOHs were identified (sum allPFC 94 mg/L). The product with highest content of extractable PFC was Granger XT Spray followed by Kiwi All Protector. In both products the FTOHs were the major PFC group detected (Granger XT 465 mg/L and Kiwi All Protector 78 mg/L). PFOA and other PFCAAs detected in Granger XT Spray are of minor concentration and could be due to degradation or impurities of the FTOHs found. In Kiwi All Protector 4% of the sum all PFC consisted of ionic PFCs. A broad variety of PFCA was found, mainly PFDoA and PFNA.

No analysed product exceeded the EU PFOS regulation.

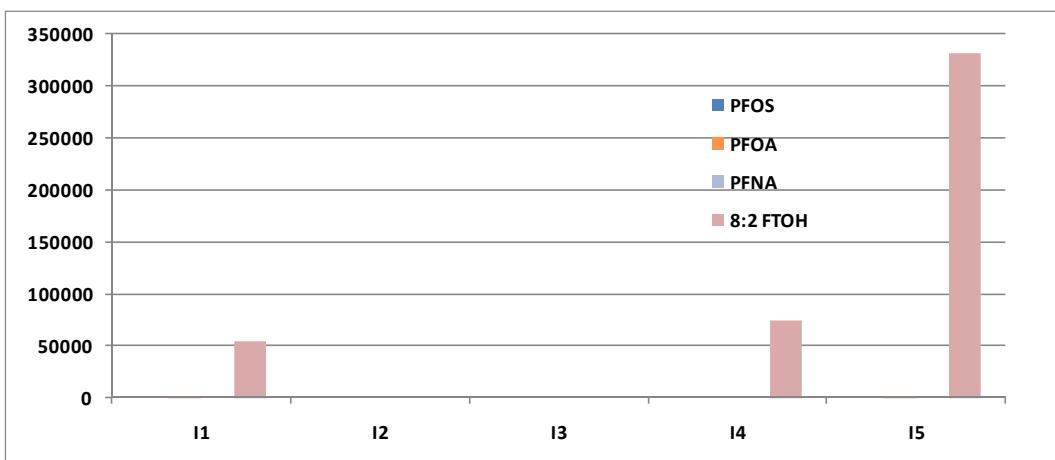


Figure 4: PFCs under legislation or planned legislation in waterproofing agents and lubricants (I) in µg/L

5.2.2 Paints and inks

Sample id	Description
S1	HP Photoprint ink yellow 363
S2	HP Laser ink pink C9723A
M1	Jotun wet room sealing compound
M2	HydroBan
M3	PCI Lastogum

No PFCs were detected in PCI Lastogum. Only ionic PFCs could be found in Jotun wet room sealing compound and HydroBan. PFOS was the main PFC with 5.8 and 4.8 µg/kg material. Small amounts of PFHxS could be found in both products as well and some PFBA in Jotun wet room sealing compound. It is unknown if the found PFC were added intentionally to the products or are impurities caused by production, transport or storage.

The two printer inks contained 8:2 and 10:2 FTOHs and HP Laser ink pink C9723A contained PFHxS, PFHpS and PFOS as well.

No analysed product exceeded the EU PFOS regulation.

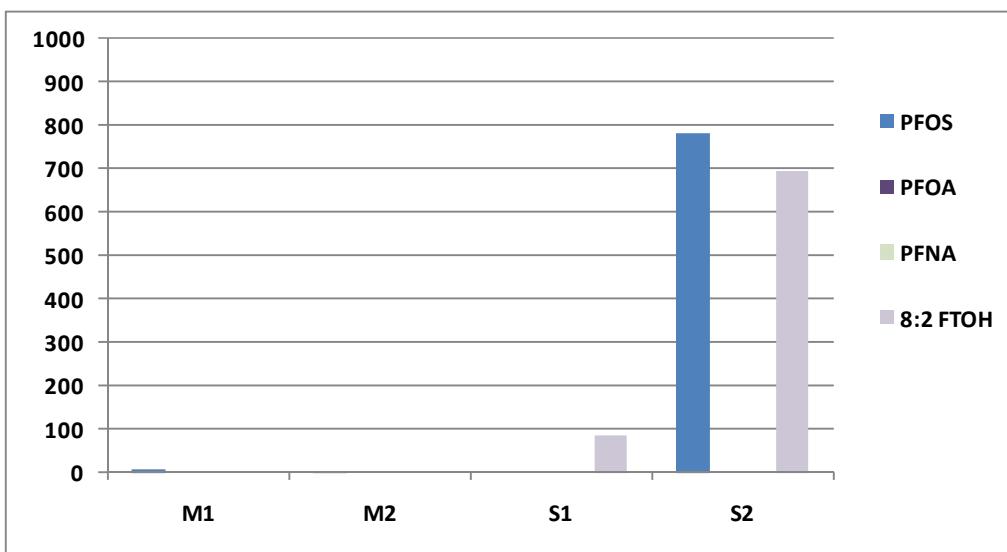


Figure 5: PFCs under legislation or planned legislation in paint (M) and printer ink (S) in µg/kg; for S1 in µg/L

5.2.3 Paper, textiles, carpets

Sample id	Description
P1	Mr. Lee, paper cup
P2	Mia Pai paper;
T1	Office furniture textile; (pool of 8)
T2	Teflon table cloth; Princess
L1	Office furniture leather; (pool of 3)
L2	Black shoe, leather
C1	Carpet grey; Monte Carlo 3000
C2	Carpet brown; Element

Both investigated paper samples were free for PFOS. The Mr. Lee paper cup and the Mia Pai paper contained no PFCs.

The office furniture textile, (pool of 8) and Teflon table cloth; Princess were PFOS free as well. Several PFCAs could be detected in the table cloth possibly due to the Teflon-treated characteristics of the textile.

6:2, 8:2 and 10:2 FTOH were detected in both textile samples comprising more than 90% of the overall PFC concentrations.

The two leather samples had the highest concentrations of PFCs. Office furniture leather; (pool of 3) and black shoe, leather, showed PFOS levels of 38 and 21 µg/m², exceeding the EU regulation of 1 µg/m². In the shoe material PFBS and PFHxS could be found as well. The office furniture leather showed PFBS

concentrations as well. Stain and water proofing treatment could be the cause of the elevated levels, but natural PFOS content of the leather is a possible source as well adding to the found levels. Only small amounts of 8:2 and 10:2 FTOH were found in the office leather, but higher concentrations were found in the shoe, again indicating stain and water proofing.

The two analysed carpets were Carpet brown; Element, Teflon treated and Carpet grey; Monte Carlo 3000 no information about any treatment. PFOS was found in both of them, with levels slightly exceeding the EU threshold of 1 µg/m² for the Teflon treated carpet (1.04 µg/m²). The same carpet contained 6:2 FTS, PFHxS, and PFHxA, PFHpA and PFOA caused probably by the Teflon treatment as well. Both carpets contain 6:2, 8:2 and 10:2 FTOH, with Carpet brown; Element, containing 10 times higher levels than Carpet grey; Monte Carlo 3000. The FTOHs stand for more than 90% of the overall PFC concentration. The detected levels of PFC in Carpet grey; Monte Carlo 3000 are maybe due to transfer during transport and storage but additional stain proofing cannot be excluded either.

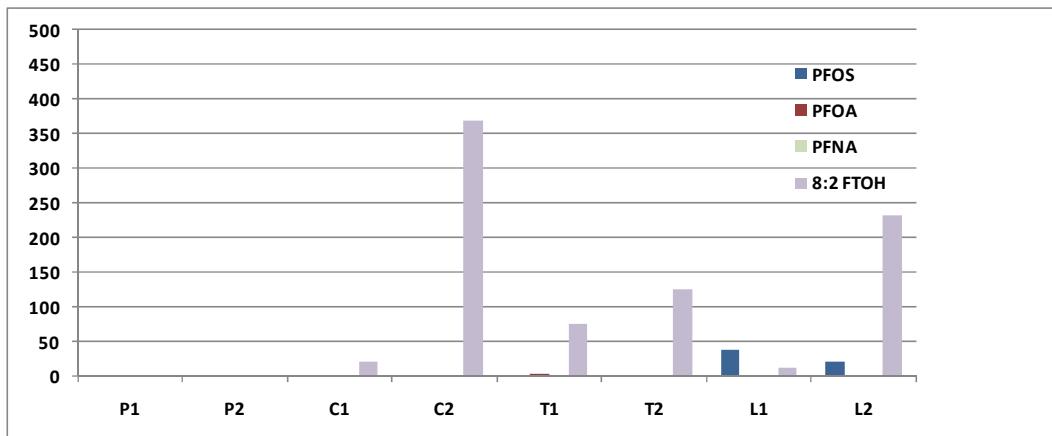


Figure 6: PFCs under legislation or planned legislation in paper (P), carpets (C), textiles (T) and leather (L) in µg/m²

5.2.4 Non-stick ware

Sample id	Description
NS1	Privilege pan
NS2	Tefal pan 28 cm
NS3	Rast camping pan
NS4	IKEA pan, Kavalkad 28 cm
NS5	Eva cake form
NS6	Tefal iron

The non-stick layer of the investigated products was scraped off prior to analyses. The given concentrations relate only to the scraped off material.

Of the six investigated products only the Tefal pan 28cm was free for PFOS (as indicated on the package). PFOA was only detected in one product, the Rast camping pan. Rast camping pan was also the nonstick product containing the highest PFC concentrations. Besides PFOA, PFHxS, PFOS and PFBA were found. PFBA was detected in all products, with highest concentrations in Eva cake form and Rast camping pan. Both products indicate a Teflon coating on the packaging. Besides PFOS, PFHxS was detected in 4 products as well. Traces of 10:2 FTOH were found in Tefal pan 28 cm and the Tefal iron. The Tefal iron showed some elevated levels for 6:2 FTOH as well.

None of the detected PFCs exceeded EU thresholds.

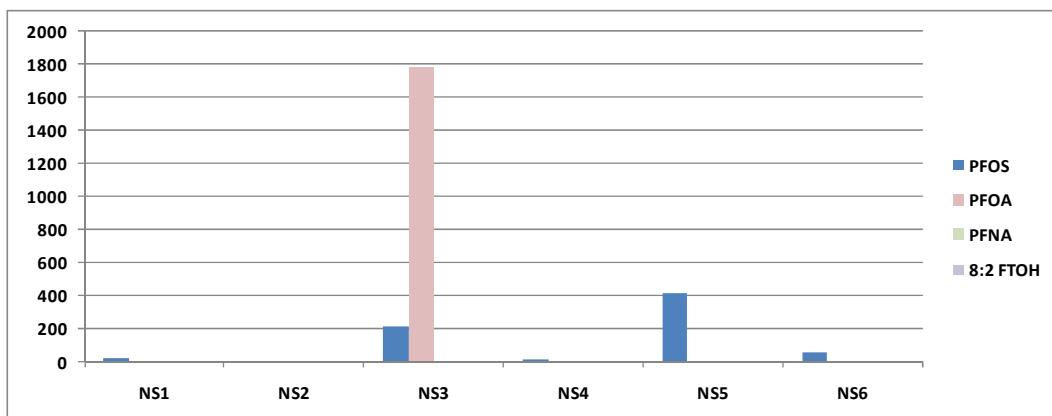


Figure 7: PFCs under legislation or planned legislation found in non-stick household ware in $\mu\text{g}/\text{kg}$

5.2.5 Electronics

Sample id	Description
E1	Li-battery, cell phone
E2	PCB, cell phone
E3	PCB, washing machine
E4	Light diodes, PD202B
E5	PCB, Disney electronic toy

The electronic articles where characterized by very low PFC levels. All three investigated power circuit boards (PCB) contained small amounts of PFOS (PCB from Sony Ericsson cell phone, the PCB from Whirlpool washing machine and PCB from Disney electronic toy). The other articles were free from PFOS. That

indicates a PFOS related process/treatment under the production of PCBs. PFBA and 6:2 FTS was found in two of the three PCBs as well. The Li-battery from a Sony Ericsson cell phone, contained PFNA and the light diode showed small concentrations of 6:2 FTS. Of the volatile PFCs only the Li-battery from a Sony Ericsson cell phone contained 6:2 FTOH and N-Me- and N-Et-FOSE.

None of the detected PFCs exceeded EU thresholds.

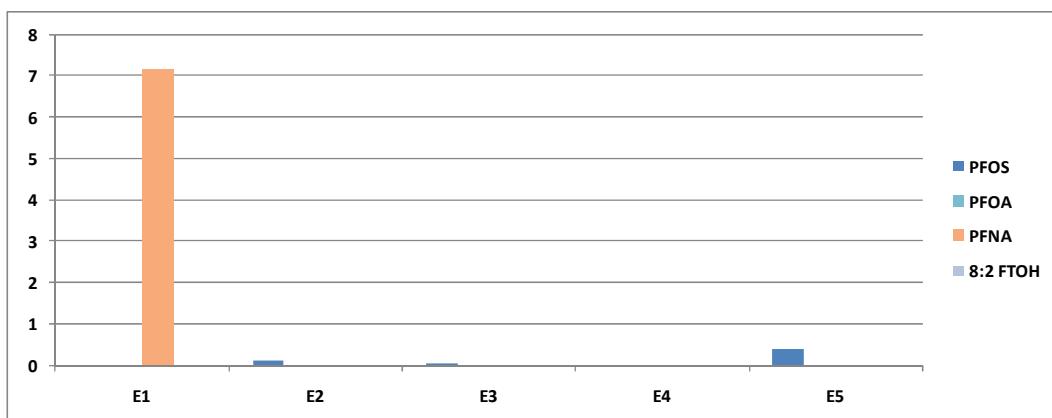


Figure 8: PFCs under legislation or planned legislation in electronic parts (E) in $\mu\text{g}/\text{kg}$

5.2.6 Fire fighting agents

Sample id	Description
B1	Powder; CHUBBFIRE GPX-ABE, PRESTO Norge
B2	Powder; Tempus multimax; PRESTO Norge
B3	ARFF Arctic Foam 3%; Solberg Scandinavia
B4	ARFF Light Water 1%; Solberg Scandinavia
B5	ARFF Shtamex P 3 %, PRESTO Norge

Of the five firefighting agents, two were powder foams and three ARFF. The two powder foams Tempus multimax; PRESTO Norge and CHUBBFIRE GPX-ABE, PRESTO Norge were free for all PFCs analysed. However, the three ARFF contained various PFCs. Arctic Foam 3%; Solberg Scandinavia and Shtamex P 3%, PRESTO Norge contained mainly 6:2 FTS, but smaller amounts of several PFCAs. No PFOS was found in these two foams. The Light Water 1% is an ARFF based on PFOS and is besides PFHpS, PFHxS and PFDoS its main PFC compound. Arctic Foam 3%, contained high amounts of 6:2, 8:2 and 10:2 FTOHs as well in similar amounts as the ionic PFCs. FOSAs and FOSEs were found in the Light Water but no FTOHs. Shtamex P 3% contained some minor amounts of 6:2 and 10:2 FTOHs as well.

The AFFF sample “Light Water” is a PFOS based fire fighting agent that is prohibited in Norway. The producer (3M) of “Light Water” is phasing out this PFOS based product and it is not sold in Norway anymore. It was received from Solberg Scandinavia in 2006 related to a research project. It was analysed during this screening as well because old amounts of it might still be in storage.

None of the legal fire fighting agents contained PFOS concentrations exceeding EU regulations.

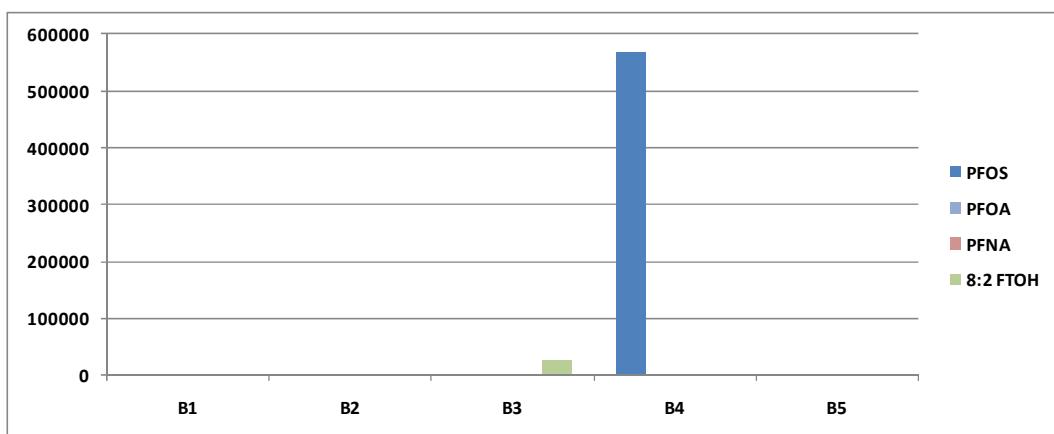


Figure 9: PFCs under legislation or planned legislation in fire fighting foam (B) in µg/L for AFFF and µg/kg in powders.

5.2.7 Summarised results with regard to present regulation

Presently, PFOS and its precursors is the only EU regulated PFC substance. A summary of PFOS content close to or exceeding the legal amounts in the analysed samples is shown below.

Table 4: Overview of analysed samples close to or exceeding the regulation conditions.

Impregnating agents
None of the samples exceed the regulatory conditions of 0.005% by weight (50 mg/kg)
Paints and inks
None of the samples exceed the regulatory conditions of 0.1% of weight (1 g/kg)
Nonstick ware
None of the samples exceed the regulatory conditions of 0.1% by weight (1 g/kg)
Paper, textiles, leather, carpets
The paper samples did not exceed the regulatory conditions of 1 µg/m ²

The textile samples did not exceed the regulatory conditions of 1 µg/m ²			
L1	Office furniture leather; (pool of 3)	38.0 µg/m²	1 µg/m ²
L2	Black shoe, leather	21.2 µg/m²	1 µg/m ²
C1	Carpet grey; Monte Carlo 3000	0.71 µg/m ²	1 µg/m ²
C2	Carpet brown; Element	1.04 µg/m²	1 µg/m ²
Electronics			
None of the samples exceed the regulatory conditions of 0.1% by weight (1 g/kg)			
Fire fighting agents			
None of the samples exceed the regulatory conditions of 0.005% by weight (50 mg/kg)			

5.3 Unknown PFCs

Additionally, all samples were screened for unknown PFCs according to the PFOS replacements identified by the Stockholm Convention on Persistent Organic Pollutants POPs review committee (POPRC). Due to the nature of the used analytical instrumentation, additional ionic PFCs could be screened for. However, not many unknown PFC could be extracted from the fullscan chromatograms. In sample B3 signals agreeing with the C₆-C₁₂-fluorinated amphoteric telomeres based on a perfluorinated sulphonamide could be found. In addition, C₈-C₁₂ fluorotelomersulfonates could be found. For sample B5, 4-fluorobenzaldehyde is listed in the safety datasheets as a major component. Polyfluoroalkyl phosphonic acids and phosphoric acids and their diesters (PAPs and diPAPs), used mainly in packaging, could not be found by the applied means in the paper samples. However, a lot of signals in the analysed samples remain unknown and their chemical structure could not be solved during the project.

6. Discussion

6.2 National and international regulations

There is international ongoing research to learn more about sources, fate and pathways of exposure to PFCs, but so far the only legally regulated group of PFC substances includes PFOS and its salts, as well as known precursors like PFOSA, N-Me-/ -Et-FOSE and FOSA and several identified precursors and intermediates. Some of these substances are listed in the OECD list from 2007 (OECD, 2007). Full international agreement on which chemicals belong to the regulated group of

chemicals as well as trace analytical methodology are not available today for all compounds hampering the regulation and control of PFOS.

In Norway the legislation already ban the use of PFOS and PFOS related substances in textiles, impregnating agents and fire fighting foam. PFOS in fire fighting foam is found to be the main source of PFOS emission in Norway².

From April 2007, it is forbidden to produce, import, export or sell impregnating agents or fire fighting foams with a content of 0,005 weight % or more of PFOS or PFOS related substances. Additionally, firefighting foams with content of PFOS or PFOS related substances shall not be used and shall be sent for destruction in an environmentally safe way.

From July 2007 it is also forbidden to produce, import, export and sell textiles or other coated materials if the amount of PFOS is equal or greater than 1 µg/m² of the coated material.

The REACH regulation also regulates PFOS with similar thresholds, but is not as stringent as the Norwegian legislation when it comes to fire-fighting foams. PFOS containing foams that were placed on the market before 27 December 2006 may be used until 27 June 2011.

Additionally the REACH regulation, annex XVII, states that

- PFOS may not be placed on the market or used as a substance in preparations in a concentration equal to or higher than 0.005% of mass.
- PFOS may not be placed on the market in semi-finished goods or in parts of such goods if the concentration of PFOS is equal to or greater than 0.1% of mass.
- For textiles or other coated materials, the amount of PFOS must be less than 1 ug/m² of the coated material.
- Photoresists or anti-reflective coatings for photolithography processes, photographic coatings applied to films, papers, or printing plates and mist suppressants for non-decorative hard chromium (VI) plating and wetting agents for use in controlled electroplating systems (where the amount of PFOS released into the environment is minimised) may contain PFOS in larger quantities. The exception granted for chromium plating processes does not apply to decorative chromium plating. The term Perfluorooctane sulfonates (PFOS), means any substance containing the PFOS moiety (C₈F₁₇SO₂) with the potential to degrade to the anionic form C₈F₁₇SO₃⁻ in the environment. These substances include the acid form of PFOS, the metal salts and the halides of PFOS and also the amides. Polymers including the PFOS moiety are also within the scope of REACH annex XVII.

United States Environmental Protection Agency (US EPA) has a voluntary agreement with the fluoropolymer industry. In this global stewardship programme on perfluorooctanoic acid (PFOA) and related chemicals, the industry commit to

² SFT (TA-2395/2008)

reducing PFOA and related chemicals by 95% no later than 2010, and to work toward total eliminating PFOA from emissions and in products no later than 2015.

Australia is developing definitions of and limit uses to non-dispersive applications, similar to restrictions in EPA consent orders with companies.

Japan has conducted extensive environmental monitoring, published an environmental risk assessment in March 2008, and added PFOS to its Pollutant Release and Transfer Register (PRTR) for annual reporting.

China has implemented EU requirements for PFOS and PFOS precursors in certain applications.

7. Conclusions and recommendations

Even with a restricted sample number, this screening of polyfluorinated products shows the extreme versatility and application in a variety of industrial materials and consumer applications and products. It is strongly recommended to have a continuous follow-up on product groups where the knowledge and known analyses of PFC content are limited. This data achieved, with respect to all identified data gaps and parts of limited information in this study can be supportive and a guideline for the future work of assessments of important perfluorinated substances in Norway. However, the EU legislation not only focuses on PFOS but on any substance containing the PFOS moiety ($C_8F_{17}SO_2$) with the potential to degrade to the anionic form $C_8F_{17}SO_3^-$ in the environment. These substances include the acid form of PFOS, the metal salts and the halides of PFOS and also the amides. Polymers including the PFOS moiety are also within the scope of Directive 2006/122/EC. A complete list of all respective compounds does not exist as well as trace analytical methods capable for all compounds mentioned. More work is needed in order to be able to enforce the new regulation. The source of some of the lower PFC concentrations detected is not always clear, since no Teflon® material was involved. Impurities, transfer during production, storage, transport or other sources could contribute to the overall PFC load. However, PFOS levels close to the regulated value in both carpets analysed, might pose an important exposure path for humans especially children. More screening is suggested for the carpet, leather and textile group in order to assess the possible exposure. Even if levels might seem low in some products, the amount of these products used and deposited could lead to a considerable source of emission into the Norwegian ecosystem especially as waste. New PFCs will be introduced into the European and global market, eventually turning up in Norwegian households

8. Reference list

CEN/TS 15968 "Determination of extractable perfluorooctanesulfonate (PFOS) in coated and impregnated solid articles, liquids and fire fighting foams – Method for sampling, extraction and analysis by LC-MS/MS or LC-MS"

European Community Regulation 1907/2006, REACH - Registration, Evaluation, Authorisation and Restriction of Chemicals

Preliminary List of PFOS, PFAS, PFOA and Related Compounds and Chemicals from 17th of August, 2007, issued by the Organization for Economic Co-operation and Development (OECD).

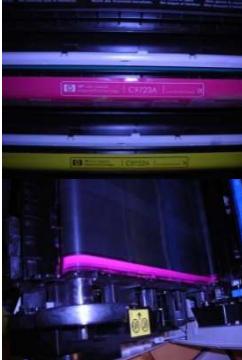
Prevedouros, K., Cousins, I.T., Buck, R.C. & Korzeniowski, S.H. Sources, fate and transport of perfluorocarboxylates, 2006.

Draft annotated outline for a study on alternatives to perfluorooctane sulfonate (PFOS), POPRC, June 2009

Appendix I. List of analysed samples

Id	Selected samples	Commercial name	Origin
	Waterproofing agents		
I1		Kiwi All Protector	Norway
I2		TF2 lubricant	Norway
I3		Rainguard; Boston	Norway
I4		Fiber Protector	Norway

I5		Granger XT Spray	Norway
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	Paints and inks		
S1		HP Photoprint ink yellow 363	Norway
S2		HP Laser ink pink C9723A	Norway
M1		Jotun våtroms sparkel	Norway
M2		HydroBan	
M3		PCI Lastogum	

	Non-stick ware		
NS1		Privilege pan	Norway
NS2		Tefal pan Ø 28	Norway
NS3		Rast camping pan	Norway
NS 4		IKEA pan, 28 cm, Kavalkad	Bought at IKEA Sweden
NS5		Eva cake form	Norway

NS6		Tefal iron	Bought at Elgiganten, Sweden
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	Paper, textiles, leather, carpets		
P1		Mr. Lee, paper cup	Norway
P2		Mia Pai paper;	Norway
T1		Office furniture textile; (pool of 8)	Norway
T2		Teflon table cloth; Princess	Norway

L1		Office furniture leather; (pool of 3)	Norway
L2		Black shoe, leather	Sample received from manufacturer, Sweden
C1		Carpet grey; Monte Carlo 3000	Norway
C2		Carpet brown; Element	Norway

	Electronics		
E1		Li-battery, cell phone	Sample received from manufacturer, Sweden
E2		PCB, cell phone	Sample received from manufacturer, Sweden
E3		PCB, washing machine	Sample received from manufacturer, Sweden
E4		Light diodes, PD202B	Bought from ELFA, Sweden
E5		PCB, Disney electronic toy	Bought at BR leksaker, Sweden



	Fire fighting agents		
B1		Powder; CHUBBFIRE GPX-ABE, PRESTO Norge	Norway
B2		Powder; Tempus multimax; PRESTO Norge	Norway
B3		AFFF Arctic Foam 3%; Solberg Scandinavia	Norway
B4		AFFF Light water 1%; Solberg Scandinavia	Norway
B5	Sthamex-AFFF	AFFF Shtamex P 3 %, PRESTO Norge	Norway

Appendix II Data tables

Waterproofing agents and lubricant

µg/L	L	L	L	L	L
	I1	I2	I3	I4	I5
6:2FT(U)CA	<LOD	<LOD	<LOD	<LOD	<LOD
8:2FT(U)CA	<LOD	<LOD	<LOD	<LOD	<LOD
6:2FTS	<LOD	<LOD	<LOD	<LOD	<LOD
8:2FTS	<LOD	<LOD	<LOD	<LOD	<LOD
PFOSA	<LOD	<LOD	<LOD	<LOD	<LOD
PFBS	<LOD	<LOD	38.65	<LOD	<LOD
PFPS	<LOD	<LOD	<LOD	<LOD	<LOD
PFHxS	<LOD	<LOD	<LOD	<LOD	<LOD
PFHpS	<LOD	<LOD	<LOD	<LOD	<LOD
PFOS	<LOD	<LOD	<LOD	<LOD	<LOD
PFDCS	<LOD	<LOD	<LOD	<LOD	<LOD
PFBA	142	81	118	95	75.9
PFPA	<LOD	<LOD	<LOD	<LOD	<LOD
PFHxA	25.6	<LOD	<LOD	<LOD	23.0
PFHpA	53.6	<LOD	<LOD	6.2	6.43
PFOA	208	<LOD	<LOD	<LOD	26.2
PFNA	593	<LOD	<LOD	<LOD	<LOD
PFDCa	168	<LOD	<LOD	<LOD	<LOD
PFUnA	198	<LOD	<LOD	<LOD	<LOD
PFDoA	1200	<LOD	<LOD	<LOD	<LOD
PTFTriA	<LOD	<LOD	<LOD	<LOD	<LOD
PFTeA	401	<LOD	<LOD	<LOD	<LOD
4:2 FTOH	<LOD	<LOD	<LOD	<LOD	<LOD
6:2 FTOH	535	<LOD	<LOD	1750	13250
8:2 FTOH	54780	<LOD	<LOD	74250	330800
10:2 FTOH	22425	<LOD	<LOD	17800	120721
N-Me-FOSA	<LOD	<LOD	<LOD	3.95	<LOD
N-Et-FOSA	18.8	<LOD	<LOD	67.9	<LOD
N-Me-FOSE	<LOD	<LOD	<LOD	<LOD	<LOD
N-Et-FOSE	<LOD	<LOD	<LOD	<LOD	<LOD

Paint and inks

$\mu\text{g}/$	L	kg	kg	kg	kg
	S1	S2	M1	M2	M3
6:2FT(U)CA	<LOD	<LOD	<LOD	<LOD	<LOD
8:2FT(U)CA	<LOD	<LOD	<LOD	<LOD	<LOD
6:2FTS	<LOD	<LOD	<LOD	<LOD	<LOD
8:2FTS	<LOD	<LOD	<LOD	<LOD	<LOD
PFOSA	<LOD	<LOD	<LOD	<LOD	<LOD
PFBS	<LOD	<LOD	<LOD	<LOD	<LOD
PFPS	<LOD	<LOD	<LOD	<LOD	<LOD
PFHxS	<LOD	400	0.53	0.31	<LOD
PFHpS	<LOD	97.6	0.10	<LOD	<LOD
PFOS	<LOD	782	5.80	4.76	<LOD
PFDCS	<LOD	<LOD	<LOD	<LOD	<LOD
PFBA	<LOD	<LOD	2.93	<LOD	<LOD
PFPA	<LOD	<LOD	<LOD	<LOD	<LOD
PFHxA	<LOD	<LOD	<LOD	<LOD	<LOD
PFHpA	<LOD	<LOD	<LOD	<LOD	<LOD
PFOA	<LOD	<LOD	<LOD	<LOD	<LOD
PFNA	<LOD	<LOD	<LOD	<LOD	<LOD
PFDCa	<LOD	<LOD	<LOD	<LOD	<LOD
PFUnA	<LOD	<LOD	<LOD	<LOD	<LOD
PFDoA	<LOD	<LOD	<LOD	<LOD	<LOD
PFTriA	<LOD	<LOD	<LOD	<LOD	<LOD
PFTeA	<LOD	<LOD	<LOD	<LOD	<LOD
4:2 FTOH	<LOD	<LOD	<LOD	<LOD	<LOD
6:2 FTOH	<LOD	<LOD	<LOD	<LOD	<LOD
8:2 FTOH	85.0	696	<LOD	<LOD	<LOD
10:2 FTOH	25.1	153	<LOD	<LOD	<LOD
N-Me-FOSA	<LOD	<LOD	<LOD	<LOD	<LOD
N-Et-FOSA	<LOD	<LOD	<LOD	<LOD	<LOD
N-Me-FOSE	<LOD	<LOD	<LOD	<LOD	<LOD
N-Et-FOSE	<LOD	<LOD	<LOD	<LOD	<LOD

Textiles, leather, carpet and paper

$\mu\text{g}/$	m2	m2	m2	m2	m2	m2	L	kg
	C1	C2	T1	T2	L1	L2	S1	S2
6:2FT(U)CA	<LOD							
8:2FT(U)CA	<LOD							
6:2FTS	<LOD	1.35	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
8:2FTS	<LOD							
PFOSA	<LOD							
PFBS	<LOD	<LOD	<LOD	<LOD	308	1.36	<LOD	<LOD
PFPS	<LOD							

PFHxS	<LOD	0.08	<LOD	<LOD	<LOD	4.81	<LOD	400
PFHpS	<LOD	97.6						
PFOS	0.71	1.04	<LOD	<LOD	38.0	21.2	<LOD	782
PFDcS	<LOD							
PFBA	<LOD							
PFPA	<LOD	<LOD	3.34	<LOD	<LOD	<LOD	<LOD	<LOD
PFHxA	<LOD	1.11	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFHpA	<LOD	0.51	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFOA	<LOD	1.67	3.74	0.40	<LOD	<LOD	<LOD	<LOD
PFNA	<LOD	<LOD	0.42	<LOD	<LOD	<LOD	<LOD	<LOD
PFDcA	<LOD	<LOD	1.29	<LOD	<LOD	<LOD	<LOD	<LOD
PFUnA	<LOD							
PFDoA	<LOD	<LOD	0.38	<LOD	<LOD	<LOD	<LOD	<LOD
PFTrIA	<LOD							
PFTeA	<LOD							
4:2 FTOH	<LOD							
6:2 FTOH	17.0	220	19.1	5.40	<LOD	<LOD	<LOD	<LOD
8:2 FTOH	22.0	368	76.4	126	13.3	231	85.0	696
10:2 FTOH	13.7	169	38.6	56.0	2.61	43.4	25.1	153
N-Me-FOSA	<LOD							
N-Et-FOSA	<LOD							
N-Me-FOSE	<LOD							
N-Et-FOSE	<LOD							

Non-stick products

μg/	kg	kg	kg	kg	kg	kg
	NS1	NS2	NS3	NS4	NS5	NS6
6:2FT(U)CA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
8:2FT(U)CA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
6:2FTS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
8:2FTS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFOSA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFBS	<LOD	<LOD	<LOD	<LOD	2.84	<LOD
PFPS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFHxS	0.80	<LOD	14.08	1.86	11.89	<LOD
PFHpS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFOS	24.9	<LOD	213.0	14.20	414.7	60.01
PFDcS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFBA	16.3	4.68	605	17.5	805	333
PFPA	88.8	<LOD	<LOD	<LOD	<LOD	<LOD
PFHxA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFHpA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

PFOA	<LOD	<LOD	1779	<LOD	<LOD	<LOD
PFNA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFDoA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFUnA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFTriA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
PFTeA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
4:2 FTOH	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
6:2 FTOH	<LOD	<LOD	<LOD	<LOD	<LOD	5.90
8:2 FTOH	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
10:2 FTOH	<LOD	3.03	<LOD	<LOD	<LOD	4.65
N-Me-FOSA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
N-Et-FOSA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
N-Me-FOSE	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
N-Et-FOSE	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

Electronic parts

µg/	kg	kg	kg	kg	kg
	E1	E2	E3	E4	E5
6:2FT(U)CA	<LOD	<LOD	<LOD	<LOD	<LOD
8:2FT(U)CA	<LOD	<LOD	<LOD	<LOD	<LOD
6:2FTS	<LOD	<LOD	0.12	1.18	0.57
8:2FTS	<LOD	<LOD	<LOD	<LOD	<LOD
PFOSA	<LOD	<LOD	<LOD	<LOD	0.65
PFBS	<LOD	<LOD	<LOD	<LOD	<LOD
PFPS	<LOD	<LOD	<LOD	<LOD	<LOD
PFHxS	<LOD	<LOD	<LOD	<LOD	0.06
PFHpS	<LOD	<LOD	0.02	<LOD	<LOD
PFOS	<LOD	0.11	0.03	<LOD	0.40
PFDoS	<LOD	<LOD	<LOD	<LOD	<LOD
PFBA	<LOD	<LOD	0.81	<LOD	24.4
PFPA	<LOD	<LOD	<LOD	<LOD	<LOD
PFHxA	<LOD	<LOD	<LOD	<LOD	<LOD
PFHpA	<LOD	<LOD	<LOD	<LOD	<LOD
PFOA	<LOD	<LOD	<LOD	<LOD	<LOD
PFNA	7.13	<LOD	<LOD	<LOD	<LOD
PFDoA	<LOD	<LOD	<LOD	<LOD	<LOD
PFUnA	<LOD	<LOD	<LOD	<LOD	<LOD
PFTriA	<LOD	<LOD	<LOD	<LOD	<LOD
PFTeA	<LOD	<LOD	<LOD	<LOD	<LOD
4:2 FTOH	<LOD	<LOD	<LOD	<LOD	<LOD
6:2 FTOH	3.49	<LOD	<LOD	<LOD	<LOD
8:2 FTOH	<LOD	<LOD	<LOD	<LOD	<LOD

10:2 FTOH	<LOD	<LOD	<LOD	<LOD	<LOD
N-Me-FOSA	<LOD	<LOD	<LOD	<LOD	<LOD
N-Et-FOSA	<LOD	<LOD	<LOD	<LOD	<LOD
N-Me-FOSE	35.9	<LOD	<LOD	<LOD	<LOD
N-Et-FOSE	2.83	<LOD	<LOD	<LOD	<LOD

Fire fighting powders and foams

µg/	kg	kg	L	L	L
	B1	B2	B3	B4	B5
6:2FT(U)CA	<LOD	<LOD	<LOD	<LOD	<LOD
8:2FT(U)CA	<LOD	<LOD	<LOD	<LOD	<LOD
6:2FTS	<LOD	<LOD	37700	8400	776600
8:2FTS	<LOD	<LOD	<LOD	<LOD	<LOD
PFOSA	<LOD	<LOD	199	12300	<LOD
PFBS	<LOD	<LOD	<LOD	253700	<LOD
PFPS	<LOD	<LOD	<LOD	<LOD	<LOD
PFHxS	<LOD	<LOD	<LOD	370000	<LOD
PFHpS	<LOD	<LOD	<LOD	901300	<LOD
PFOS	<LOD	<LOD	<LOD	568000	<LOD
PFDCS	<LOD	<LOD	<LOD	114400	<LOD
PFBA	<LOD	<LOD	404	27647	960
PFPA	<LOD	<LOD	966	125000	1825
PFHxA	<LOD	<LOD	<LOD	<LOD	3810
PFHpA	<LOD	<LOD	<LOD	<LOD	<LOD
PFOA	<LOD	<LOD	1880	<LOD	<LOD
PFNA	<LOD	<LOD	<LOD	<LOD	<LOD
PFDCa	<LOD	<LOD	447	<LOD	<LOD
PFUnA	<LOD	<LOD	<LOD	<LOD	<LOD
PFDoA	<LOD	<LOD	<LOD	<LOD	143
PTFTriA	<LOD	<LOD	<LOD	<LOD	<LOD
PFTeA	<LOD	<LOD	<LOD	<LOD	<LOD
4:2 FTOH	<LOD	<LOD	<LOD	<LOD	<LOD
6:2 FTOH	<LOD	<LOD	1610	<LOD	848
8:2 FTOH	<LOD	<LOD	26500	<LOD	<LOD
10:2 FTOH	<LOD	<LOD	5800	<LOD	110
N-Me-FOSA	<LOD	<LOD	110	24922	<LOD
N-Et-FOSA	<LOD	<LOD	2207	9210	767
N-Me-FOSE	<LOD	<LOD	<LOD	7515	<LOD
N-Et-FOSE	<LOD	<LOD	<LOD	472	<LOD