KEMI
Swedish Chemicals Agency

Chemical Analysis of Selected Fire-fighting Foams on the Swedish Market 2014

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KEMI
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.

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Foreword

The Swedish Chemicals Agency (KEMI) has been assigned by the Swedish Government to produce a national action plan for a toxic-free everyday environment: *Action plan for a toxic-free everyday environment 2011 – 2014 – protect the children better*. The action plan has been extended to 2020. Efforts are going on in several areas, both in Sweden, within the EU and internationally and often in cooperation with other authorities.

Reducing chemical risks in the everyday environment is one step towards attaining the Swedish Parliament´s environment quality objective A Non-Toxic Environment, which is the objective that the Swedish Chemicals Agency is responsible for. Within the framework of the action plan, we compile knowledge in the Swedish Chemicals Agency's report and PM series elaborated by experienced colleagues, researchers or consultants. In this way, we present new and essential knowledge in publications which can be downloaded from the website www.kemikalieinspektionen.se.

This report is an account of a study on fire-fighting foams, conducted by Örebro University on behalf of the Swedish Chemicals Agency and the Swedish Civil Contingencies Agency (MSB) in the autumn of 2014. Anna Kärrman (PhD) and her team at MTM Research Centre, School of Science and Technology, Örebro University, performed chemical analysis of selected fire-fighting foams on the Swedish market. The aim was to identify the presence of per- and polyfluorinated alkyl substances (PFASs) including precursor compounds. This report describes analysis of eight selected products for target analysis of known PFASs and six products for non-target analysis to elucidate the main components and if they contain organofluorine or not.

Contacts at the Swedish Chemicals Agency were Jenny Ivarsson and Bert-Ove Lund; Head of Unit Agneta Falk Filipsson was responsible for the assignment at the Swedish Chemicals Agency.

The opinions and recommendations presented in the report are entirely those of the author and do not necessarily reflect the Swedish Chemicals Agency's point of view.

Stockholm, May 2015

Innehåll

Summary **5**

Sammanfattning **6**

1 Materials and Methods **7**

2 Results **8**

2.1 Presence and levels of known fluorinated substances 8

2.1.1 Telomer substances 8

2.1.2 Perfluorinated carboxylic acids (PFCAs) 9

2.1.3 Perfluorinated sulfonic acids (PFSAs) 9

2.1.4 Sulfonamides 9

2.2 Non-target analysis 9

3 Discussion **10**

4 References **11**

Appendix 1 Results of target analysis **12**

Appendix 2 Name and abbreviations of substances analysed **14**

Appendix 3 Target PFAS-profile (µg/kg) and the structure(s) of tentatively identified compounds **15**

Summary

At the request of the Swedish Chemicals Agency and the Swedish Civil Contingencies Agency (MSB), Örebro University performed chemical analysis of selected fire-fighting foams on the Swedish market in the autumn of 2014. The aim was to identify the presence of per- and polyfluorinated alkyl substances (PFASs) including precursor compounds.

Producers consider the ingredients in their products as trade secrets thus fire-fighting foams can contain a variety of additives and the presence of fluorinated substances needs to be elucidated due to environmental and health concerns. This report describes analysis of eight selected products for target analysis of known PFASs and six products for non-target analysis to elucidate the main components and if they contain organofluorine or not.

Selected products for flammable and combustible liquid fires (class B fires), were kindly donated from distributors and users. Known PFASs were found in all products analysed, most commonly detected were short chain perfluorinated carboxylates, from low ppb levels up to ppm levels. Perfluorohexanoic acid (PFHxA, C6) was found in highest concentrations, up to 14 000 µg/kg. Fluorotelomer sulfonic acid with six fluorinated carbons (6:2 FTS) was quantified in all studied products up to 10 000 µg/kg. Bioaccumulating perfluorinated acids ($C \geq 7$) and perfluorinated sulfonic acids were detected less frequent and in lower concentrations. Perfluorooctane sulfonic acid (PFOS) was found in two different products from users, but not in the corresponding samples from sealed containers or distributors indicating probable site contamination from previous fire-fighting products.

The six selected products for non-target analysis all contained fluorinated telomer products with 6:2 configuration including mercaptoalkylamido sulfonate, sulfonamide amine, and sulfonamide alkylbetaine. The connection between the PFAS-profile (target analysis) and the identified structures is clear, residues of C6 and shorter carboxylic acids and C6 fluorotelomer sulfonate in the mg/kg range are present in products that have the 6:2 telomer configuration as part of the precursor molecule. Other PFAS-profiles in products from users can be due to contamination from previously used products.

Sammanfattning

På uppdrag av Kemikalieinspektionen och Myndigheten för samhällsskydd och beredskap (MSB) utförde Örebro universitet kemiska analyser av utvalda brandskum på den svenska marknaden under hösten 2014. Syftet var att identifiera förekomsten av per- och polyfluorerade alkylsubstanser (PFAS), inkluderat olika prekursorer.

Brandskum kan innehålla olika tillsatsämnen och tillverkare betraktar ingredienserna i sina produkter som företagshemligheter. På grund av hälso- och miljöproblem behöver förekomsten av PFAS i brandskum klargöras. Denna rapport beskriver analyser av utvalda brandskum, såväl riktade analyser av kända PFAS av åtta produkter som screeninganalyser av sex produkter. Detta för att kartlägga huvudkomponenterna i brandskummen och om de innehåller organiskt fluor eller inte.

Utvalda produkter för brandfarliga och brännbara vätskebränder (klass B-bränder), donerades av distributörer och användare. Kända PFAS detekterades i alla analyserade produkter. Mest frekvent var kort-kedjiga perfluorerade karboxylsyror, från låga ppb-nivåer upp till ppm-nivåer. Högsta koncentrationer hittades av perfluorhexansyra (PFHxA, C6), upp till 14 000 µg/kg. Fluortelomersulfonat med sex perfluorerade kol (6:2 FTS) kvantifierades i alla studerade produkter, upp till 10 000 µg/kg. Bioackumulerande perfluorerade karboxylsyror (C $\geq$ 7) och perfluorerade sulfonsyror hittades i mindre utsträckning och i lägre koncentrationer. Perfluoroktansulfonsyra (PFOS) detekterades i två olika produkter från användare, men inte i motsvarande prover från slutna behållare eller distributörer. Detta indikerar att produkterna troligen kontaminerats av tidigare använda brandskumsprodukter innehållandes PFOS.

Alla de sex utvalda produkterna som screenades innehöll fluorerade telomerer med 6:2-karaktär (mercaptoalkylamido sulfonat, sulfonamid amin, och sulfonamid alkylbetain). Kopplingen mellan PFAS-profilen (riktad analys) och de identifierade strukturerna är tydlig. Rester av C6 och kortare karboxylsyror och C6-fluortelomersulfonat hittades i mg/kg-nivåer i produkter som har 6:2 telomerkonfiguration som del av prekursormolekylen. Andra PFAS-profiler i produkter från användare kan bero på föroreningar från tidigare använda produkter.

1 Materials and Methods

Fire-fighting products were provided from different sources as listed in Table 1 and were sampled by the provider, between 200 mL – 5 000 mL, in clean plastic containers (not containing Teflon® or other fluoropolymers) during autumn 2014. The products were thereafter sub-sampled and diluted (between 100 and 10 000 times) with laboratory produced water (MilliQ-quality) and methanol (LC-MS grade) and were filtered (GHP filters, 0.2 µm) before analysis.

Table 1 Fire-fighting foams included in this study and the analysis performed on each product

Name	**Manufacturer/ Distributer**	**Source**	**Non-target**	**Target**
ARC Miljö	Dafo Brand	Ringhals	X	X
		MSB		X
		Räddningstjänsten Storgöteborg *		X
Skum AFFF 3%	Dafo Brand	MSB	X	X
Alcoseal 3-6%	Angus/Kidde	Räddningstjänsten Storgöteborg	X	X
Sthamex AFFF-P 3%	Dr Sthamer/Presto	Växjö flygplats	X	X
		IVL/Arlanda		X
		Försvarets Material Verk *		X
Towalex AFFF 3% master	Tyco	Tyco *	X	X
Towalex AFFF-AR 3x3	Tyco	Tyco *		X
Towalex AFFF 3% super	Tyco	Tyco *		X
OneSeven B-AR	Nordic Fire & Rescue Service (NFRS)	NFRS *		X
		Växjö flygplats	X	X

* Samples taken from sealed containers *or* directly from distributors

Names and abbreviations of substances analysed are listed in Appendix 2. Target analysis of perfluorinated alkyl acids and sulfonates (PFCAs, PFSAs), fluorotelomersulfonates (FTS), sulfonamides including perfluorooctane sulfonamides (PFOSA, N-ethylFOSA, N-methylFOSA) and perfluorooctanesulfonamidoethanols (FOSEs), unsaturated and saturated telomer acids (FTUCAs and FTCAs), was performed by LC-MS/MS on an Acquity UPLC Xevo TQ-S tandem mass spectrometer (Waters Corporation, Milford, USA) with an atmospheric electrospray interface operating in negative ion mode. The analytes were

separated on an Acquity BEH C18 column (2.1 x 100 mm, 1.7 µm), with flow rate 300 µL/min using a gradient program delivering mobile phases consisted of 2 mM NH_4Ac in MeOH, and 2 mM NH_4Ac in H_2O. An extra guard column (PFC isolator, Waters Corporation, Milford, US) was inserted between the pump and injector to remove any PFAS originating from the LC system. Capillary voltage was set to 0.6 kV, source and desolvation gas (N_2, 950L/hr) temperatures were 150 and 450°C. Cone voltages and collision energies were optimized for each transition. Multiple reaction monitoring was used monitoring the product ions.

Target analysis of fluorotelomer alcohols (FTOHs) was performed by GC-MS on a 7890A GC system equipped with a 5975C mass selective detector (Agilent Technologies, Palo Alto, CA). Selected ion monitoring (SIM) operated in the positive chemical ionization (PCI) mode with methane as reagent gas was used to monitor protonated molecular ions $[M+H]^+$ and a fragment ion (loss of HF + H_2O) of 6:2, 8:2 and 10:2 FTOH. Compound separation and quantification were performed on a 60 m Supelcowax® 10, 250 µm x 0.25 µm, column (Supelco Inc., Bellefonte, PA).

Quantification of target compounds was performed by adding labeled internal standards to the diluted (approximately 10 mg to 1 mL) products. Interferences in the analysis from sample matrix were corrected for by the labeled standards. Labeled internal standards were added to clean methanol/water and were analysed as a blank control. PFBuS and 6:2 FTS were detected in blanks and reported values are at least three times higher than the blank concentration. Several blank injections were performed to monitor possible system carry-over or memory effects.

Non-target analysis was performed on an Acquity UPLC Xevo Synapt QTOF (Waters Corporation, Milford, USA) with atmospheric electrospray. The UPLC separation was identical as for the target analysis described above when in negative mode. In positive mode 0.1% formic acid was used instead of 2mM ammonium acetate. Non-target screening was done in both positive and negative mode and by the MS^E-technology that maximises the information obtained from a single analysis by switching low collision energy mode, to obtain precursor ion data, with a high collision energy ramp to obtain fragment ion data. Tentatively identification of unknown compounds was performed using software MassLynx and MarkerLynx, elemental composition and by matching structures with the database Chemspider. A diluted solution (10 000 times) of the products was injected.

2 Results

2.1 Presence and levels of known fluorinated substances

Concentrations of selected analysed target PFAS compounds are summarised in tables 2-5 in Appendix 1.

2.1.1 Telomer substances

Fluorotelomer alcohols (FTOHs) and fluorotelomer acids (FTUCAs but not FTCAs) were detected in Sthamex and ARC Miljö but only in low trace levels (low µg/kg). It should be noted that GC-MS analysis of FTOHs was hampered by the fact that no clean-up was used and should be considered as semi quantitative. Fluorotelomer sulfonate with six perfluorinated carbons (6:2 FTS) was present in all studied products. Presence of telomer acids and sulfonates can be indicative of degradation of larger fluorinated telomer substances.

2.1.2 Perfluorinated carboxylic acids (PFCAs)

Several carboxylates were quantified in all studied products. Persistent perfluorinated acids were detected in ppb to ppm levels. Bioaccumulating perfluorinated acids ($C \geq 7$, PFHpA and higher) were detected less frequent and in ppb levels typically 10-100 µg/kg. PFHxA (C6) was found in highest concentration (up to 14 000 µg/kg) and in all products. PFPeA (C5) and PFBA (C4) followed the same pattern as PFHxA but in decreasing concentrations with decreasing chain length. In some cases there are large differences in PFCA levels in the same product from different users.

2.1.3 Perfluorinated sulfonic acids (PFSAs)

Perfluorobutane sulfonate (PFBuS) and perfluorohexane sulfonate (PFHxS) were detected in OneSeven B-AR, ARC Miljö and Sthamex samples taken from users. PFOS was detected in two samples, ARC Miljö and Sthamex, both from users. Only Oneseven B-AR from the group of samples originating from sealed containers or distributors contained PFSA (PFHxS in low µg/kg).

2.1.4 Sulfonamides

No detectable concentrations of N-Ethyl FOSA/E and N-Methyl FOSA/E were found. PFOSA was detected in two Sthamex products from users at 700-5000 µg/kg.

2.2 Non-target analysis

An attempt to identify the major signals for both positive and negative ions present in ARC Miljö, Sthamex, Alcoseal 3-6%, Skum AFFF 3%, Towalex 3% master, and OneSeven B-AR was made. Foams are complex mixtures of additives such as surfactants, and a mixture of chemicals is present in the products. Tentative identification was made when the structure could be confirmed by the presence of the molecular ion and at least two product ions. The found structures containing organofluorine were compared to literature and could be confirmed to be present in fire-fighting foams included in other studies (1-4) for all cases except for ARC Miljö.

Fluorotelomermercaptoalkylamido sulfonate with six fluorinated carbons (6:2 FTSAS) was identified in Towalex master, Alcoseal 3-6%, Skum AFFF3% and OneSeven B-AR.

Fluorotelomer sulfonamide alkylbetaine (6:2 FTAB, CAS 34455-29-3) was identified in Sthamex, Alcoseal 3-6% and Towalex 3% master.

ARC Miljö also contains the 6:2 fluorotelomer chemistry which could be identified as 6:2 fluorotelomer sulfonamide amine (CAS 80475-32-7) however one of the main signals represented a structure in where the amine group connected to the sulfonyl group bears one oxygen.

The six products with their target PFAS-profile (µg/kg) and the structure(s) of tentatively identified compounds are presented in Appendix 3.

3 Discussion

All B-class foams included in the non-target analysis contain 6:2 fluorotelomer substances. 6:2 telomer substances are expected to degrade in the environment to persistent perfluorinated alkyl acids (2, 5). The presence of PFHxA and 6:2 FTS can be residues from the manufacturing process or due to storage degradation. The connection between the PFAS-profile (target analysis) and the identified structures is clear, traces of C6 carboxylic acid and 6:2 fluorotelomer sulfonate in the mg/kg range are present in products that have the 6:2 telomer configuration as part of one of the major organic molecules. 6:2 FTSAS has previously been reported to be present in AFFFs with product names F-500, Tridol S3%, Anslite 3% AFFF-DC-3, Niagara 1-3, and Ansul Ansulite ARC (3). 6:2 FTAB has been reported to be present in Forafac 1157, F-500, Niagara 1-3, and Tridol S (1, 2).

Other PFAS-profiles in samples collected from users can be due to contamination from previously used products. There is also a possibility that users keep an older product that has the same name as a current product but in where the ingredients have been replaced over time. Table 3 and Table 5 show the target analysis results for products that were taken directly from the distributors, however two products in this study, Skum AFFF 3% and Alcoseal 3-6%, were only obtained from end users. The results from this limited study indicate that contamination from previously used products might be widespread.

There is no indication that the C8 chemistry (including 8:2 FTS) resulting in PFOS and PFOA transformation products are present in the products included in this study that are from distributors or taken from sealed containers. If this is the case for all products in Sweden today is difficult to say and is outside the scope of this study. It must be noted that only organic compounds can be detected in this study. Moreover, any organic component that for some reason does not ionize under the used analytical conditions (positive and negative atmospheric electrospray ionization together with water/methanol/ammonium acetate or formic acid) cannot be detected. The signal intensity may not be representative of the actual concentration and the homogeneity of the foam samples has not been studied which may be a source of uncertainty both in the non-target and target analysis. For unequivocal identification of unknowns reference standards are required. No clean-up was employed but a reasonable separation was achieved with Ultra Performance Liquid Chromatography (UPLC) prior to mass detection. The appearance of the peaks corresponding to organofluorines is indicative of telomer products thus lacking the signature of isomer peaks commonly present in for example electrochemical fluorination (ECF) products. However this needs to be verified with authentic standards.

4 References

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2. Moe MK, Huber S, Svenson J, Hagenaars A, Pabon M, Trumper M, Berger U, Knapen D, Herzke D. Chemosphere 89 (2012) 869-875.
3. Weiner B, Yeung LWY, Marchington EB, D´Agostino LA, Mabury SA. Environmental Chemistry 10 (2013) 486-493.
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Appendix 1 Results of target analysis

Table 2 Target analysis of perfluorinated carboxylic acids (µg/kg) in foam samples taken from users.

Product	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFTrDA	PFTDA
OneSeven B-AR [1]	3482	6100	14217	554	285	<1	<1	<1	<1	<1	<1
ARC Miljö [1]	<1	212	1174	147	218	128	129	53	62	25	44
ARC Miljö [2]	472	264	1928	256	391	137	89	44	31	<1	<1
AFFF 3%	<1	22	76	<1	<1	<1	<1	<1	<1	<1	<1
Alcoseal 3-6%	<1	32	796	51	124	<1	17	<1	<1	<1	<1
Sthamex AFFF-P 3% [1]	2695	1986	9947	8378	64141	96	244	20	72	<1	<1
Sthamex AFFF-P 3% [2]	792	219	1617	50	343	22	156	11	72	<1	42

Table 3 Target analysis of perfluorinated carboxylic acids (µg/kg) in foam samples taken from distributors or intact containers.

Product	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFTrDA	PFTDA
OneSeven B-AR [2]	1485	1122	512	131	3	<1	<1	<1	<1	<1	<1
ARC Miljö [3]	546	108	1074	20	<1	<1	<1	<1	<1	<1	<1
Towalex plus	<1	78	1481	23	70	7	26	<1	<1	<1	<1
Towalex 3x3	1008	551	9770	134	239	20	63	<1	<1	<1	<1
Towalex 3% super	<1	<1	84	<1	<1	<1	<1	<1	<1	<1	<1
Towalex 3% master	1142	620	10352	149	344	21	79	8	26	<1	<1
Sthamex AFFF-P 3% [3]	83	17	83	<1	<5	<5	<1	<1	<1	<1	<1

Table 4 Target analysis of perfluorinated sulfonic acids, telomer sulfonic acids and sulfonamide (µg/kg) in foam samples taken from users.

Product	**PFBuS**	**PFHxS**	**PFOS**	**PFDS**	**6:2 FTS**	**PFOSA**
OneSeven B-AR [1]	294	74	<1	<1	4031	<1
ARC Miljö [1]	<1	<1	<1	<1	9726	<1
ARC Miljö [2]	383	1342	6792	<1	8548	<1
AFFF 3%	<1	<1	<1	<1	1653	<1
Alcoseal 3-6%	<1	<1	<1	<1	3765	<1
Sthamex AFFF-P 3% [1]	2878	12126	156581	614	3312	5805
Sthamex AFFF-P 3% [2]	51	<1	<1	<1	1838	728

Table 5 Target analysis of perfluorinated sulfonic acids, telomer sulfonic acids and sulfonamide (µg/kg) in foam samples taken from distributors or intact containers.

Product	**PFBuS**	**PFHxS**	**PFOS**	**PFDS**	**6:2 FTS**	**PFOSA**
OneSeven B-AR [2]	<1	52	<1	<1	2407	<1
ARC Miljö 3	<1	<1	<1	<1	4373	<1
Towalex plus	<1	<1	<1	<1	3449	<1
Towalex 3x3	<1	<1	<1	<1	8130	<1
Towalex 3% super	<1	<1	<1	<1	284	<1
Towalex 3% master	<1	<1	<1	<1	4109	<1
Sthamex AFFF-P 3% [3]	<1	<1	<1	<1	9498	<1

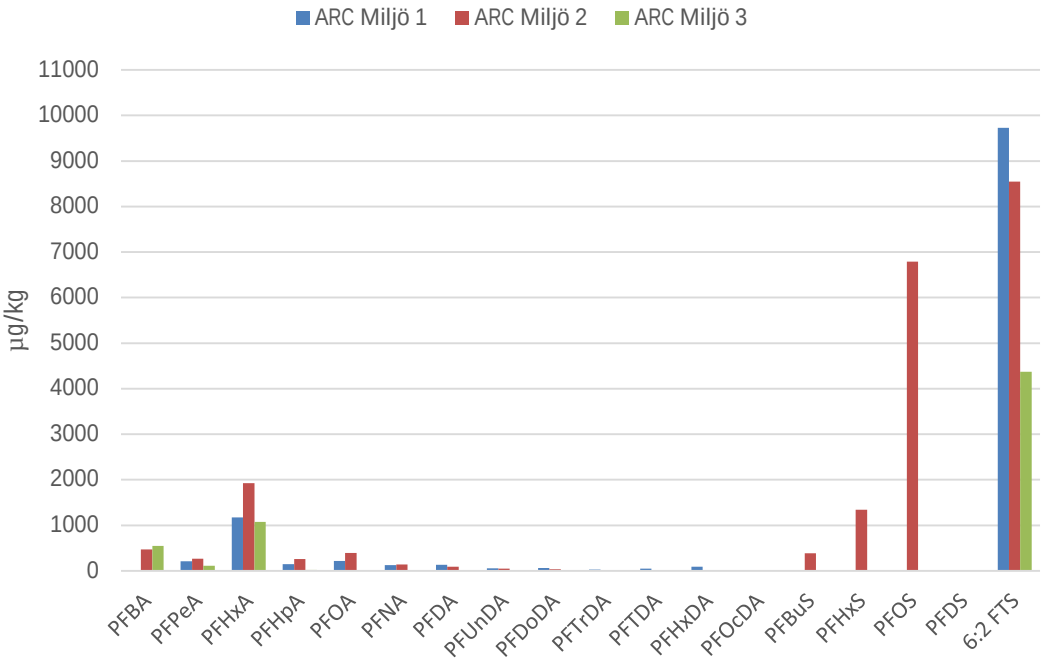
Appendix 2 Name and abbreviations of substances analysed

Per- and polyfluorinated alkyl substances	PFAS
Perfluorobutanoic acid	PFBA
Perfluoropentanoic acid	PFPeA
Perfluorohexanoic acid	PFHxA
Perfluoroheptanoic acid	PFHpA
Perfluorooctanoic acid	PFOA
Perfluorononanoic acid	PFNA
Perfluorodecanoic acid	PFDA
Perfluoroundecanoic acid	PFUnDA
Perfluorododecanoic acid	PFDoDA
Perfluorotridecanoic acid	PFTrDA
Perfluorotetradecanoic acid	PFTDA
Perfluorobutane sulfonic acid	PFBuS
Perfluorohexane sulfonic acid	PFHxS
Perfluorooctane sulfonic acid	PFOS
Perfluorodecane sulfonic acid	PFDS
6:2 Fluorotelomer Sulfonate	6:2 FTS
Perfluorooctane sulfonamide	PFOSA
N-Methylheptadecafluorooctane sulfonamide	N-MeFOSA
N-Ethylheptadecafluorooctane sulfonamide	N-EtFOSA
N-Methylheptadecafluorooctane sulfonamidoethanol	N-MeFOSE
N-Ethylheptadecafluorooctane sulfonamidoethanol	N-EtFOSE
Fluorotelomer alcohol	FTOH
1H,1H,2H,2H-perfluoro-1-octanol	6:2 FTOH
1H,1H,2H,2H-perfluoro-1-decanol	8:2 FTOH
1H,1H,2H,2H-perfluoro-1-dodecanol	10:2 FTOH
Fluorotelomer unsaturated carboxylic acid	FTUCA
Fluorotelomer carboxylic acid	FTCA
Fluorotelomermercaptoalkylamido sulfonate	6:2 FTSAS
Fluorotelomer sulfonamide alkylbetaine	6:2 FTAB

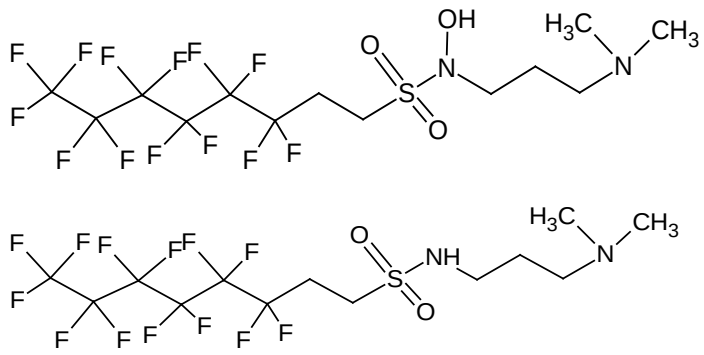
Appendix 3 Target PFAS-profile (µg/kg) and the structure(s) of tentatively identified compounds

ARC MILJÖ

Levels of selected target PFAS (µg/kg) in ARC Miljö. [1] and [2] are samples from users and [3] is from a sealed container

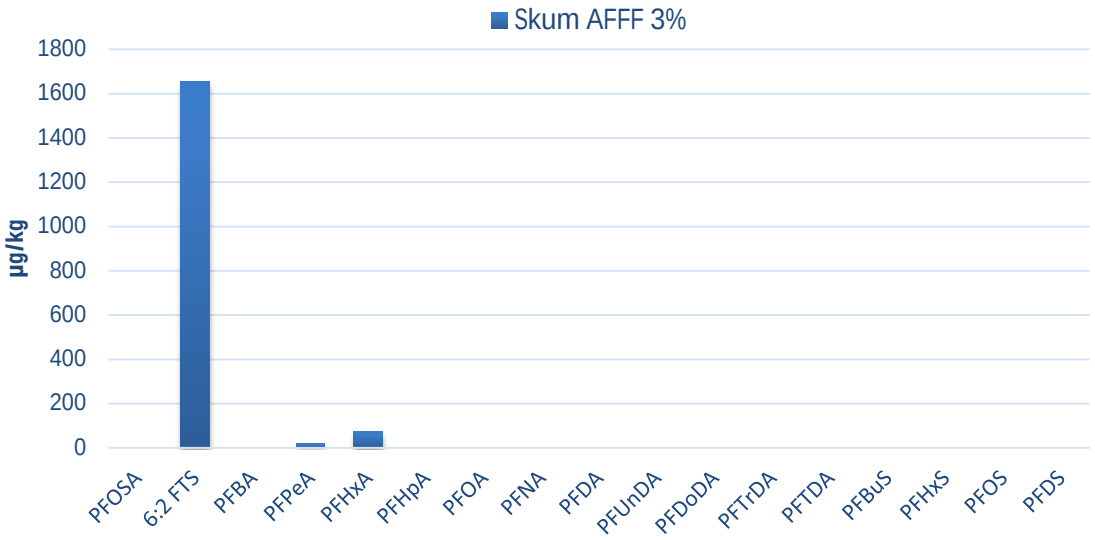


Tentatively identified PFAS as a main ingredient is 6:2 fluorotelomer sulfonamide amine (CAS: 80475-32-7).

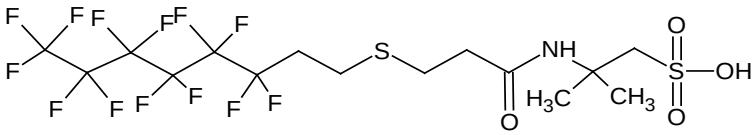


SKUM AFFF 3%

Levels of selected target PFAS (µg/kg) in Skum AFFF 3%. The sample origins from a user.

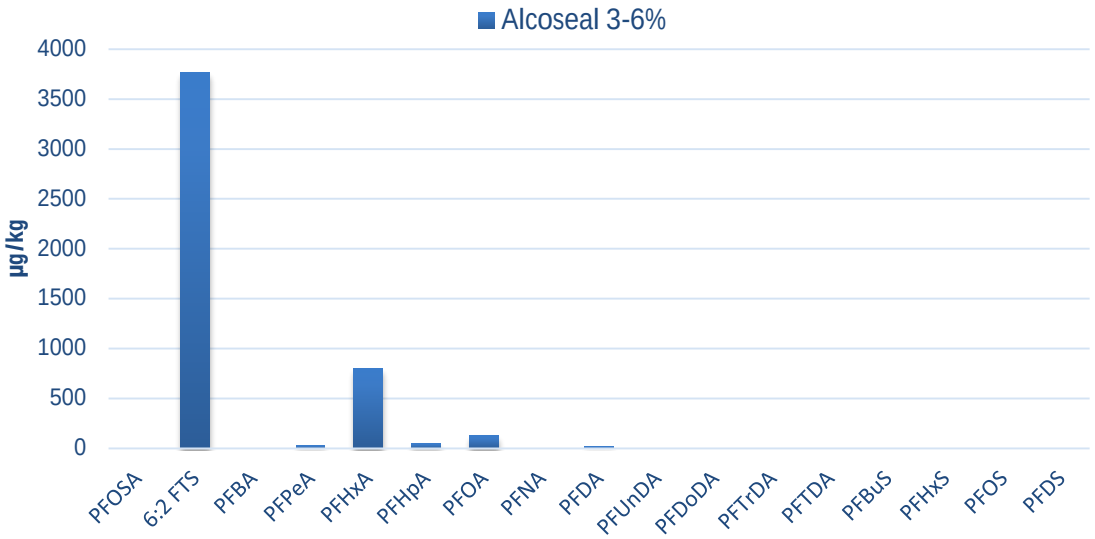


Tentatively identified PFAS as a main ingredient is 6:2 FTSAS (fluorotelomermercaptoalkylamido sulfonate).

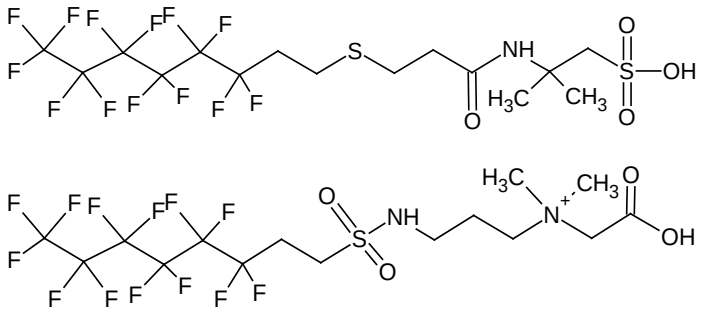


ALCOSEAL 3-6%

Levels of selected target PFAS (µg/kg) in Alcoseal 3-6%. The sample origins from a user.

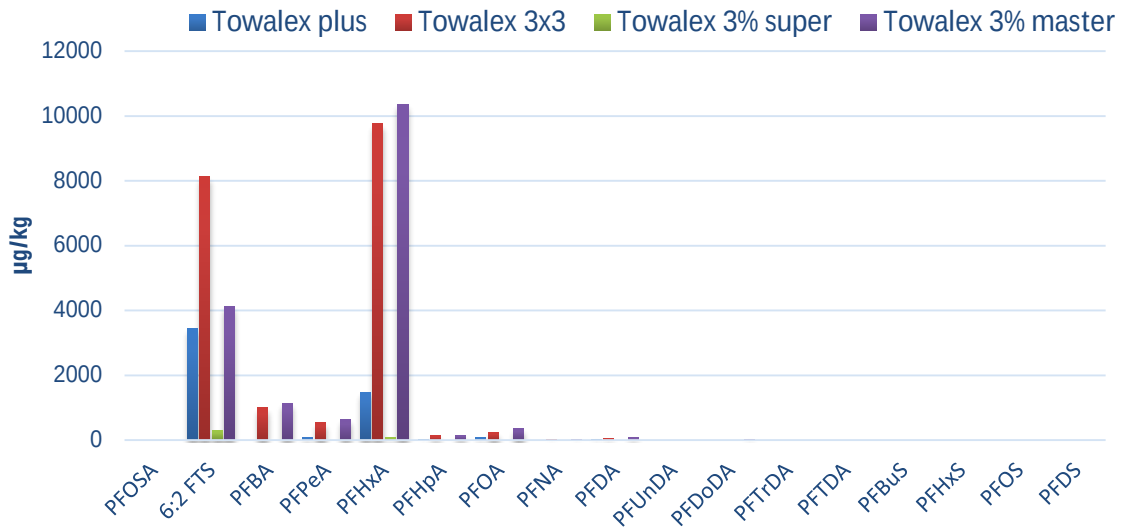


Tentatively identified PFASs as main ingredients are 6:2 FTSAS (fluorotelomermercaptoalkylamido sulfonate) and 6:2 FTAB (fluorotelomer sulfonamide alkylbetaine, CAS: 34455-29-3).

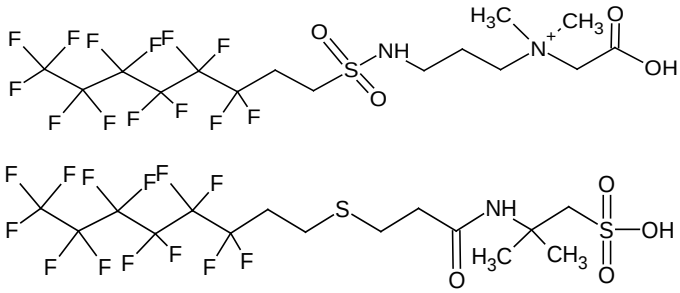


TOWALEX

Levels of selected target PFAS (µg/kg) in different Towalex products. The samples origin from sealed containers or directly from distributors.

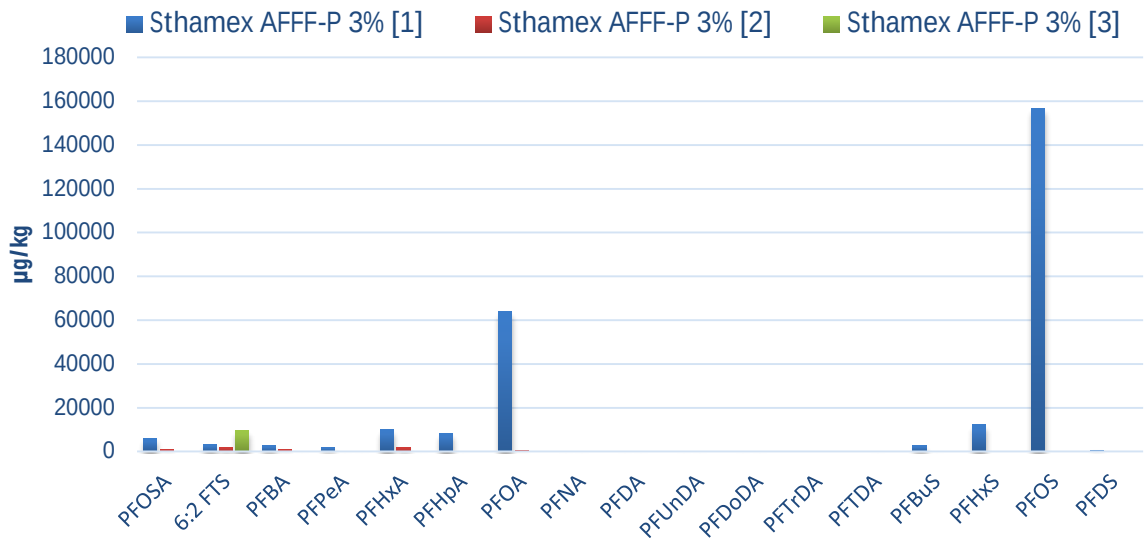


Tentatively identified PFASs as main ingredients in Towalex 3% master are 6:2 FTAB (fluorotelomer sulfonamide alkylbetaine, CAS: 34455-29-3) and 6:2 FTSAS (fluorotelomermercaptoalkylamido sulfonate).

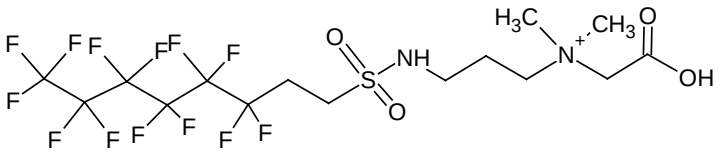


STHAMEX AFFF

Levels of selected target PFAS (µg/kg) in Sthamex AFFF. [1] and [2] are samples from users and [3] is from a distributor.

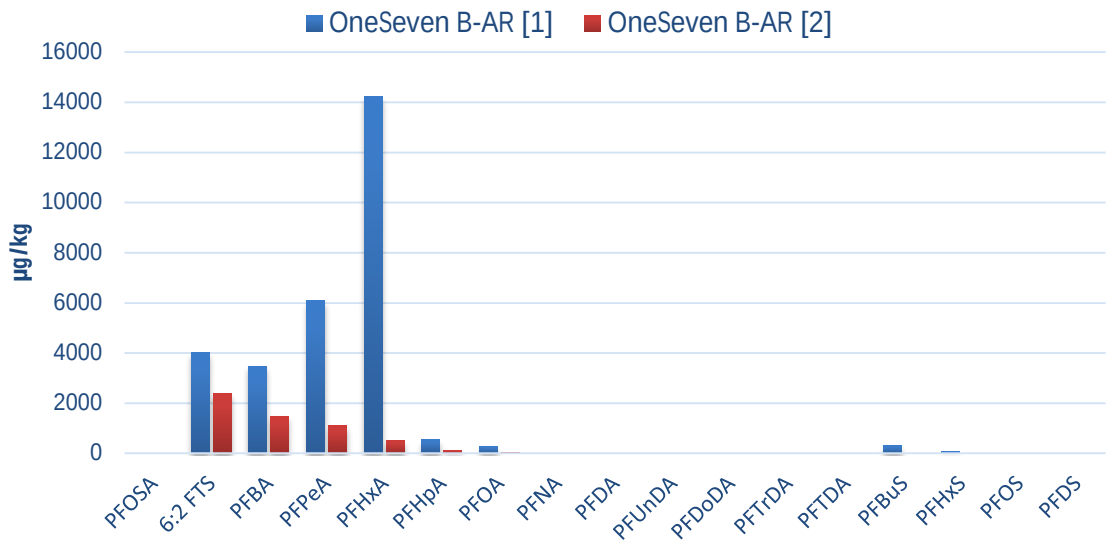


Tentatively identified PFAS as a main ingredient is 6:2 FTAB (fluorotelomer sulfonamide alkylbetaine) CAS: 34455-29-3.

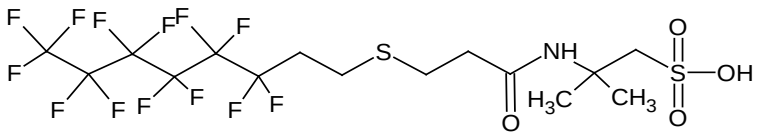


ONESEVEN B-AR

Levels of selected target PFAS (µg/kg) in OneSeven B-AR. [1] is a sample from a user and [2] is from a distributor.



Tentatively identified PFAS as a main ingredient is 6:2 FTSAS (fluorotelomermercaptoalkylamido sulfonate).



KEMI
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