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Nordic enforcement project on PFOS and PFOA in chemical products and articles

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Content

Glossary

Article: An object which during production is given a specific shape, surface or design which determines its function to a greater degree than does its chemical composition

Candidate list: List of substances of very high concern (SVHC) from which the substances to be included in Annex XIV of the REACH Regulation (list of substances subject to authorisation) are selected. The candidate list is established in accordance with Article 59 of the REACH Regulation.

CAS number: Unique substance identification number, assigned and registered by the Chemical Abstract Services (CAS)

CEN: The European Committee for Standardization

Derivatives: Chemical compound that can be derived from or prepared from another given chemical compound

EC number: Seven-digit EC number, an identifier for substances commercially available within the European Union

EOF: Extractable organic fluorine

ICSMS: Information and Communication System for Market Surveillance

PFAS: Per- and polyfluoroalkyl substances

PFCA: Perfluorinated carboxylic acid

PFHxA: Perfluorohexanoic acid

PFHxS: Perfluorohexanesulfonic acid

PFOA: Perfluorooctanoic acid

PFOA-related compounds: Substances that are considered to have a potential to degrade or be transformed to PFOA

PFOS: Perfluorooctane sulfonic acid

POPs Regulation: Regulation (EU) No 2019/1021 of the European Parliament and of the Council on persistent organic pollutants

REACH Regulation: Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorization and Restriction of Chemicals

Safety Gate (previously RAPEX): Rapid Alert System for dangerous non-food products / European platform to inform consumers and market surveillance authorities about dangerous products found on the EU/EEA market.

Substances of Very High Concern (SVHC): Substances that meet the criteria in Article 57 in the REACH Regulation. These are carcinogenic, mutagenic, toxic to reproduction, dangerous for the environment (persistent, bioaccumulative, toxic or very persistent and very bioaccumulative) or which have other serious properties, for example endocrine disrupting properties.

Summary

PFAS (Per- and polyfluoroalkyl substances) is a large group of synthetically produced organic compounds with many uses in both industry and consumer products. They occur as, for example, surfactants in metal plating, in cosmetics, in firefighting foams, and in textiles and paper packaging to make them repellent to water, grease and dirt. PFAS are considered particularly dangerous because they are extremely difficult to degrade in the environment and therefore stay in the environment for a long time.

This report describes the Nordic Enforcement Group's joint enforcement project on PFOA (perfluorooctanoic acid) and PFOS (perfluorooctane sulfonic acid) in chemical products and articles. The objectives of the project were to inspect the compliance of chemical products and articles placed on the Nordic market with the restrictions in the POPs Regulation[1] on PFOA[2] and PFOS[3], to raise awareness of the restrictions in the POPs Regulation and to learn together how the enforcement of the new restrictions can be carried out. In addition, the presence of PFAS that are not yet restricted in any chemical legislation and extractable organic fluorine (EOF) were analyzed to improve the knowledge of the authorities on the use of PFASs in various products and articles.

In total, 158 products were inspected, 95 chemical products and 63 articles. 40 % of the chemical products and 65 % of the articles contained PFAS. 15 products of the 158 controlled products were non-compliant with the inspected POPs restrictions for PFOA and PFOS (ski waxes and a jacket). Many products and articles contained PFOA and PFOS in levels below the limit values, and many contained PFAS that are not yet restricted in chemical legislation.

The PFOA and PFOS restrictions include hundreds of substances. For many of them there is still a lack of available information on occurrence and use, identification numbers (CAS or EC numbers) and external reference standards for quantitative analyses. There is a great need to develop analytical methods for targeted analyses of PFAS to make a more effective enforcement possible. Extractable organic fluorine (EOF) provided information on the presence of PFAS that was not included in the targeted PFAS analyses. The analyses showed high levels of organic fluorine in for example ski waxes, textiles, and firefighting foams. Some of it could originate from restricted PFAS. Many of the unrestricted substances found in products and articles may be a subject to restrictions in the future. Companies that place chemical products and articles on the market should start working to phase out these substances from production.

[1] Regulation (EU) No 2019/1021

[2] The POPs Regulation restricts PFOA, its salts and PFOA-related compounds. To avoid repetition, "PFOA" is used in this report.

[3] The POPs Regulation restricts PFOS and its derivatives. To avoid repetition, "PFOS" is used in this report.

1. Background

1.1. The Nordic Enforcement Group

The chemical authorities in the Nordic countries have for many years worked together in various enforcement projects, to ensure greater impact and to obtain better understanding of common EU legislation in the field of chemicals. The agencies also learn to carry out more effective market surveillance as a result from the exchange of information and experiences during these projects.

Joint enforcement projects, like this one on PFOA and PFOS in chemical products and articles, are driven by the Nordic Enforcement Group, a subgroup in the Nordic Chemicals Group under the Nordic Council of Ministers. The main purpose of the Nordic Enforcement Group is to exchange experience on control and enforcement of the European chemicals legislation and to prepare and carry out common enforcement projects.

We launched this joint enforcement project in early 2020 and it was finalized in December 2021. The main responsibility for the project was allocated to the Finnish Safety and Chemicals Agency (Tukes) and the project leader was Petteri Talasniemi. Participants from the other Nordic countries in the project group were Sara Björkqvist, Hanne Thygesen, Kenneth Ebert and Ida Lundstein Scharff from the Danish Environmental Protection Agency, Maryam Ashja and Amanda Rosen from the Swedish Chemicals Agency, Markus Koponen and Markus Mäki from the Finnish Safety and Chemicals Agency, Christian Iversen from the Norwegian Environment Agency and Bergdís Björk Bæringsdóttir and Björn Gunnlaugsson from the Environment Agency of Iceland.

1.2. Why are PFAS of very high concern?

PFAS (short for per- and polyfluoroalkyl substances) is a class of thousands of man-made substances that are widely used in consumer products and industrial applications. In 2018, the OECD found almost 5000 different CAS numbers for PFAS. The carbon-fluorine bond of PFAS is extremely strong and stable. PFAS have very low surface tension, are resistant to heat and chemical degradation as well as being water and oil repelling. Due to their unique physical and chemical properties, PFAS are used in a wide variety of articles and chemical products, such as water- and stain repellent textiles, firefighting foams, food contact materials, and cosmetics. Uses of PFAS have been examined in many studies, also in other projects funded by Nordic Council of Ministers (1, 2, 3, 4, 5). PFAS have been analysed in different products e.g., food contact materials, impregnation sprays, polishes, waxes, shoes and clothes (2, 3, 4). Despite an enormous number of studies on PFAS, it is still challenging to identify all their uses in various products and articles.

The common concern for all PFAS is a high persistence since PFAS (or their degradation products) are resistant to degradation and remain for a long time in environment. Some PFAS are known to accumulate in the bodies of humans and animals and in the environment, be highly mobile in the environment and pose a risk to human health and environment. At present, there is a lack of knowledge for the most PFAS about their toxicity or bioaccumulation properties. PFAS are ubiquitously found globally in the environment and in the bodies of almost all humans today. Several PFAS contamination cases of water (including drinking water) and soil have been discovered in the EU and globally.

The potential sources of PFAS exposure to humans and the environment are the production and use in industry, release from consumer products during use and the product end of life, including recycling and disposal of products. Humans can be exposed to PFAS via several exposure pathways, including contaminated food, water, air, consumer products, and household dust. The Nordic studies have highlighted the widespread presence of PFAS in the Nordic environment (6). In the most recent study from 2019, PFAS have been measured in bird eggs, fish, marine mammals, terrestrial mammals, surface water, wastewater treatment plants' effluents and sludge, and air. It has been suggested that PFAS concentrations are increasing in the environment due to their extreme persistency, and there is a potential for unexpected (eco)toxicological effects. The studies also show that it is very costly to manage the negative impacts on the environment and human health caused by PFAS contamination (7).

Global environmental contamination and risks have led to regulation of PFAS in chemical legislation. The EU has taken action to restrict PFOA and PFOS, consistent with the Stockholm Convention. The manufacturing, placing on the market and use of PFOA, PFOA salts and PFOA-related compounds as such, in mixtures or in articles are restricted under the POPs Regulation. The PFOA restriction started to apply from 4th of July 2020 while PFOS and its derivatives have been restricted since 2009. Because of the implemented restrictions, there have been changes in the industry shifting towards replacement of long-chain PFAS to short chain PFAS. Further restrictions on other PFAS are underway at the EU and global level.

Due to concerns related to PFAS, discussions are ongoing under the European Green Deal, on how to minimise environmental and human exposure to PFAS through legislative actions and other initiatives. In the recently announced EU chemical strategy for sustainability, the European Commission proposed a series of actions aimed at phasing out all non-essential uses of PFAS as a class. Among other regulatory actions, the enforcement of chemical legislations is crucial to ensure a high level of human health- and environmental protection.

1.3. Objectives of the project

The main objectives of the enforcement project were:

- to inspect the compliance of chemical products and articles placed on the Nordic market with the restrictions in the POPs Regulation on PFOS and PFOA,
- to raise awareness of the new restriction of PFOA in the POPs Regulation,
- to follow up the restriction with enforcement action and to achieve a greater degree of compliance, and
- to learn together, how the enforcement of the new restriction can be carried out.

A supplementary objective of this project was to improve the knowledge of the authorities on the use of PFAS which are not yet restricted in the chemical legislation. The results will also contribute to the ongoing regulatory discussions on PFAS.

2. Methodology

2.1. Legislative scope

The scope of the project was to check compliance of the chemical mixtures and articles with the POPs Regulation (EU) No 2019/1021 of:

- PFOA, its salts and PFOA-related compounds
- PFOS and its derivatives

The POPs Regulation restricts several persistent organic pollutants (POPs). The substances that are regulated in this legislation comes from the international Stockholm Convention and the Convention on Long-range Transboundary Air Pollution (CLRTAP).

According to the Article 3 of the POPs Regulation, the manufacturing, placing on the market and use of substances listed in Annex I such as PFOA and PFOS, whether on their own, in mixtures or as a constituent in articles are prohibited. Article 4 of the Regulation contains general exemptions from control measures. The exemptions apply, among other things, to substances that occur as unintentional trace contaminants in substances, mixtures or in articles. Substances present in articles already in use before this Regulation entered into force are also exempted. Substance-specific limit values for unintentional trace contaminants and other exemptions are listed for each substance in Annex I (Table 1).

The limit values contained in the POPs Regulation only apply to unintentional trace contaminants that have arisen, for example, in the manufacturing process. It is forbidden to intentionally add PFOA and PFOS in products and articles.

Table 1. Limit values for unintentional trace contaminants in the Annex I of the POPs Regulation

Restricted substance	Limit value for unintentional trace contaminant
Perfluorooctane sulfonic acid and its derivatives (PFOS)	up to 10 mg/kg (0.001 % by weight) where it is present in substances or in mixtures. up to 0.1 % in articles or 1 µg/m^2 when present in textiles and other coated materials.
Perfluorooctanoic acid (PFOA) and its salts	up to 0.025 mg/kg (0.0000025 % by weight) where they are present in substances, mixtures or articles.
Any individual PFOA-related compound or a combination of PFOA-related compounds	up to 1 mg/kg (0.0001 % by weight) where they are present in substances, mixtures or articles.
PFOA and its salts equal to or below 1 mg/kg (0.0001 % by weight) where they are present in polytetrafluoroethylene (PTFE) micropowders produced by ionising irradiation of up to 400 kilograys or by thermal degradation as well as in mixtures and articles for industrial and professional uses containing PTFE micropowders.	up to 1 mg/kg (0.0001 % by weight)

In addition to the restrictions on PFOA and PFOS, work is currently underway to develop restrictions for other long-chain PFAS (C9-C14 perfluorinated carboxylic acids (PFCAs)) and some short-chain PFAS such as perfluorohexanesulfonic acid (PFHxS) and perfluorohexanoic acid (PFHxA) and their salts and related compounds. Many of these substances have been identified as substances of very high concern (SVHC) and included in the candidate list. Furthermore, several member states have begun work on developing a broad restriction proposal under the REACH Regulation, which would cover all PFAS in non-essential uses. This would in the long run mean phasing out of all PFAS in non-essential uses.

2.2. Laboratory survey

At the start of the project a survey was carried out to find available analytical laboratories in the Nordic countries. The aim was to survey the analytical test methods analyzing for the presence of PFOS and PFOA in different product types. The possibility to measure the presence of other PFAS (not restricted) and extractable organic fluorine (EOF) was also investigated.

According to the POPs Regulation, the analytical method standards for PFOS adopted by the European Committee for Standardization (CEN) shall be used as analytical method to demonstrate that substances, mixtures and articles comply with the requirements. It is also stated that as an alternative to the CEN standard, another method of analysis may be used if it gives similar results. The standardized analytical method (CEN/TS 15968:2010) is available for the determination of PFOS in coated and impregnated solid articles, liquids, and firefighting foams. In contrast to PFOS, at present there are no standardized analytical methods available to measure PFOA in chemical products or articles. Standardized methods exist only for the analysis of PFOA in environmental samples. A development of a CEN standard for measuring PFAS in textiles is ongoing.

Based on the results of our survey, only a few commercial laboratories contacted in the Nordic countries used the CEN/TS 15968: 2010 method for PFOS or an equivalent method for their analyses. If available, the method has been modified to cover also other PFAS including PFOA. Most of the laboratories in the Nordic countries are focused primarily on providing environmental monitoring of PFAS and therefore use standard test methods to detect PFAS in various environmental samples (e.g., water, drinking water and soil). In some cases, these methods have been modified to cover also other matrices such as chemical products or textiles.

For most of the laboratories, there were difficulties to achieve the low limits of quantification needed in measurements of PFOA (0.025 mg/kg). This was mostly due to matrix effects of the chemical mixtures and articles (e.g., limitations of extraction methods). In addition, detection limits varied between the laboratories and different test methods.

Based on results of our survey most of the commercial laboratories had analysis packages with quantitative analyses of about 30-50 individual PFAS, so-called targeted PFAS analyses. However, the challenge was that the available methods cover only a limited number of PFOA-related compounds. PFOA-related compounds are restricted substances that are considered to have a potential to degrade or be transformed to PFOA (e.g., polymers with ≥ C8 based perfluoroalkyl side chains; 8:2 fluorotelomer compounds and 10:2 fluorotelomer compounds). Hundreds of PFAS are estimated to be degradable into PFOA, but many of them still lack available information on use and identification number (so-called CAS or EC number) and reference materials for quantitative analyses. The substances are largely unknown.

To sum up our laboratory survey, the PFOA restriction is challenging to enforce, this due to the absence of a standardized test methods measuring PFOA, its salts and PFOA-related substances in different matrices with adequately low limits of quantification. Another shortcoming that was noted regarding current analytical methods is that there is a lack of reference material for most PFAS. To ensure a proper enforcement of the restriction, there is a need for EU-standardized test methods in various matrices at sufficiently high sensitivity and reference materials for analyses. Due to the large number of PFAS on the market, there is a need to compile a regularly updated list of substances which are covered by restriction of PFOA, its salts and PFOA-related compounds.

Laboratory testing was found to be a limiting factor in the project, and it affected the selection of the inspected product types.

It should also be mentioned that the analytical methods used in this project differ between the countries, the tested products, and articles, which means that the results are not necessarily quantitatively comparable. The number of different PFAS analyzed for also differ between the countries, which means that the results are not directly comparable between the countries. More information on analytical methods and lists of measured substances are included in the Appendix 2.

Extractable organic fluorine (EOF)

In this project, analyses of extractable organic fluorine (EOF) were performed for some of the inspected chemical products and articles. The EOF analyses were performed at Örebro University. The EOF content provides information about the presence of unknown organofluorine compounds that could not be identified in the targeted PFAS analyses. An EOF analysis does not provide any information on which individual PFAS that are included but can in combination with quantitative analyses give an indication on whether there is a large proportion of unidentified PFAS in a sample. It is possible that the EOF measurements for certain products and chemical products may be affected by inorganic fluorine.

2.3. Market surveillance

The restrictions of PFOS and PFOA apply to both products for professional use and consumer products. Within the framework of this project, we chose to focus on both product types.

The preliminary selection of product samples was made based on experience from previous enforcement projects and research in the area on the presence of PFAS in products (i.e., a risk-based approach). In addition, information on the content of PFAS in chemical products that companies had reported to the national product registers was used in the selection. The final selection of product types was made based on the laboratory capabilities to analyse PFOS, PFOA and related substances in different product types. The results presented in this report are not based on random sampling, and therefore should not be considered as applicable to the entire market.

The controlled products were bought or acquired directly from importers/distributors, retailers, or from online stores. The types of products inspected included products for consumers (e.g., outdoor clothes, shoes, bicycle care products, ski waxes) and for the professional market (e.g., textile floorings, fire-fighting foam products). See Table 2 for more information on selected product types.

All companies whose products were inspected were informed about the results of the analyses and non-compliant products were followed up with enforcement actions.

The following chapters contain information about the amounts and types of products tested, the results found, and the enforcement actions taken against the non-compliances.

3. The results of the joint enforcement

3.1. General overview of the results

In this enforcement project, we tried to cover a wide range of chemical products and articles. All in all, 158 products were purchased/acquired, 95 of the products were chemical products and 63 were articles, as shown in Figure 1.

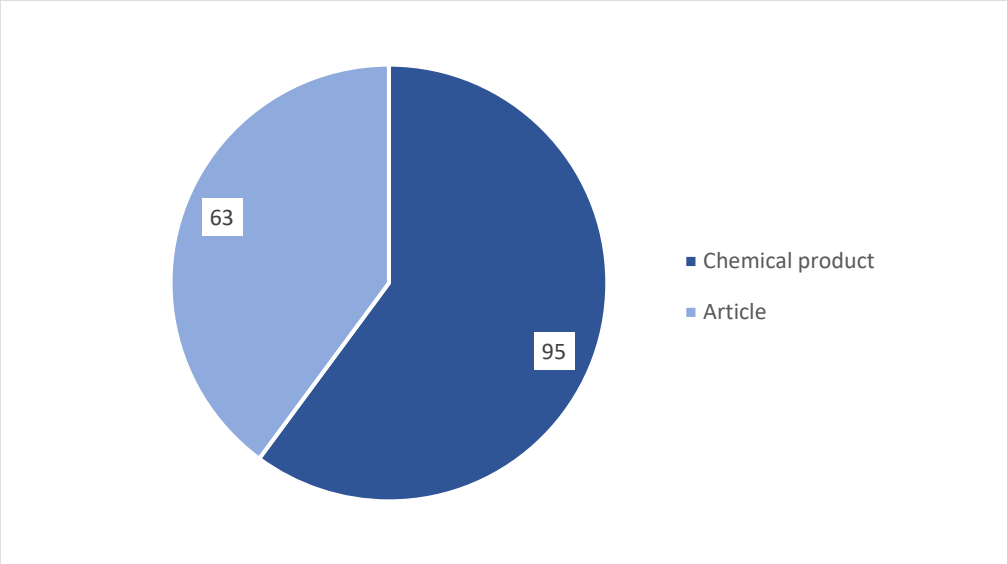


Figure 1 Overview of product categories controlled

The inspected products were purchased/acquired from 89 different companies. The EU regulation differentiates between different roles, which companies (economic operators) may have. Companies can be manufacturers, importers, or distributors, but they can also have several roles at the same time. The majority of the companies, 91 %, were distributors, while 18 % were importers and 35 % were manufacturers.

All the controlled product types, number of products and a summary of the analysis results are listed in Table 2.

Table 2 Summary of product types, number of products and the results of the PFAS-analysis

Product type	Number of products	PFAS not detected	PFAS detected	Restricted PFAS over the limit values	Restricted PFAS below the limit values
Textile flooring (carpet)	10	8	2	0	1
Snow suit	3	2	1	1*	0
Jacket (shell, softshell, rain, biking, hunting)	28	8	20	1	17
Pants (shell)	2	0	2	0	2
Gloves	4	1	3	0	3
Shoes (outdoor, hiking)	9	3	6	0	4
Backpack	4	0	4	0	3
Tent/accessories	3	0	3	0	2
Firefighting foam concentrate	10	6	4	0	0
Fire extinguisher	5	0	5	0	0
Textile impregnation product	7	5	2	0	0
Shoe care product	12	10	2	0	0
Bicycle care product (lubricant)	9	7	2	0	0
Floor care product (polish, lacquer)	7	4	3	0	0
Car care product (wax, oil)	4	4	0	0	0
Boat care product (polish, wax)	2	2	0	0	0
Ski wax/ski care product	39	19	20	14	2
In total	**158**	**79**	**79**	**16**	**34**

***** No enforcement measures were imposed, see section "3.2 Compliance with POPs Regulation"

Among the chemical products and articles that were inspected, the following can be summarized based on the analysis results:

- 40 % of the chemical products and 65 % of the articles contained PFAS substances,
- 9 % of the chemical products/articles were non-compliant,
- 2 % of the inspected chemical products and 50 % of the articles contained PFOA and/or PFOS substances in levels below the limit values set in the POPs regulation,
- Many products contained PFAS that are not restricted in the POPs regulation.

In Figure 2 an overview of the results per product type is shown.

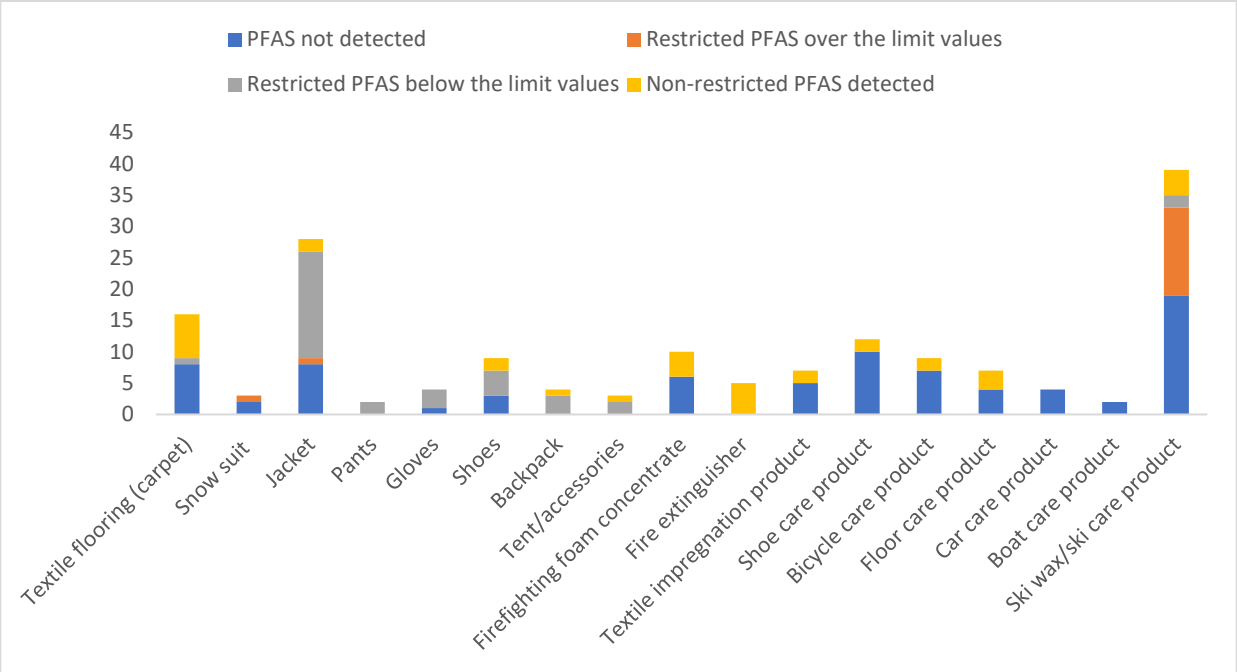


Figure 2 Summary of the results

PFAS substances were often present in products and articles in combinations of PFAS with different carbon chain lengths, for example in textiles and ski waxes C6 (PFHxA and related substances), C8 (PFOA), and C9-14 PFCAs (e.g., perfluorononanoic acid (PFNA) and perfluorodecanoic acid (PFDA)).

3.2. Compliance with the POPs regulation

The overall picture of the inspections is that 15 products and articles (9 %) of the 158 controlled products and articles were non-compliant with the POPs regulation. 14 ski waxes contained a PFOA while one jacket contained PFOS + derivatives above or around the limit values.

A snow suit contained a PFOA-related substance just above the limit value. It showed that the snow suit was produced before the PFOA restriction entered into force, which led to no enforcement measures being imposed with respect to this product. This due to uncertainties regarding which exemption that should apply, in combination with a minimal exceedance of the limit value.

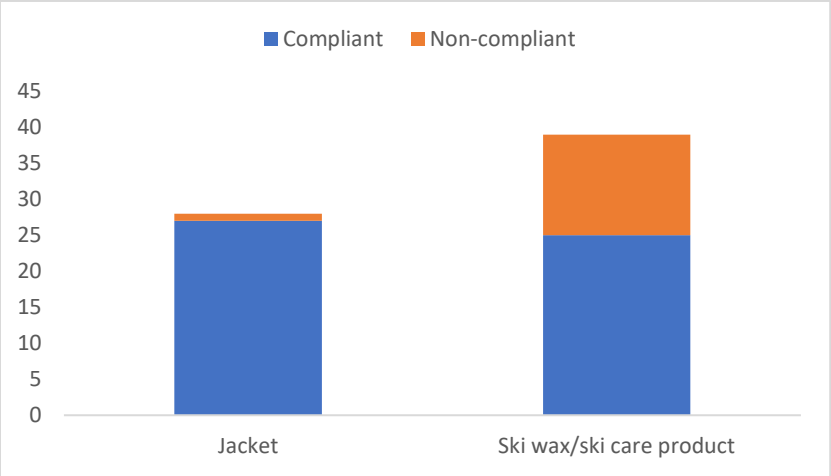


Figure 3 Product categories with non-compliances (number of compliant and non-compliant chemical products or articles)

Several products (textiles and ski wax) contained PFOA and/or PFOS substances in levels below the limit values regarding unintentional trace contaminants, as shown in Table 3 and Figure 2. It was very difficult or impossible to assess whether the PFOA and PFOS substances detected in the products below limit values were unintentional trace contaminants or whether they were intentionally added. The source of the substances as not known. In most cases, the concentrations were far below the limit values. It cannot be ruled out that a combination of PFAS with different chain lengths, in low concentrations may have been used to provide, for example, a dirt and water-repellent function in a product.

Table 3 Summary of number of product types containing PFOA and/or PFOS below the limit values

Product type	Number of products	PFOA and/or related substance	PFOS and/or its derivatives
Textile flooring (carpet)	1	1	0
Jacket	17	14	3
Pants	2	2	1
Gloves	3	3	0
Shoes	4	2	2
Backpack	3	3	1
Tent/accessories	2	2	0
Ski wax/ski care product	2	2	1
In total	**34**	**29**	**8**

3.3. Enforcement measures

Upon observation of a non-compliance, the companies which were controlled were made aware of the non-compliances with the law and requested to give their opinion on the issue. The most common enforcement measure was written advice.

In most of the cases the companies took the necessary corrective actions on a voluntary basis. For ski waxes, the most common follow-up action was the discontinuation of the sale of a product (sales ban). This was also the action taken regarding the jacket, which was also withdrawn from the market and recalled from the consumers.

Public announcement of non-compliant products was made by the enforcement authorities. Information on non-compliant products was forwarded to other Member States through ICSMS and/or Safety Gate systems, if needed. In some of the cases, follow-up activities are still ongoing.

In the cases with products containing restricted PFAS below the limit value regarding unintentional trace contaminants, it was assumed that the detected concentrations were due to unintentional trace contamination. The companies were therefore just informed about the findings.

All companies whose products were inspected were informed about the results of the analyses as well as provided with information about the POPs Regulation and other relevant legislation.

3.4. Non-restricted PFAS

The results showed that many products contained PFAS that are not restricted in the POPs regulation, as shown in Figure 2. PFAS substances were very often present in products in combinations of PFAS with different carbon chain lengths for example in textiles and ski waxes C6 (PFHxA and related substances), C8 (PFOA), and C9-14 PFCAs (e.g., perfluorononanoic acid (PFNA) and perfluorodecanoic acid (PFDA)). Ski waxes followed by textiles and bicycle care products were the product types that contained the largest amount of non-restricted PFAS. However, this is only an indication, as not all product types were analyzed for non-restricted substances (e.g., snow suits, car and boat care products were only analyzed for restricted PFAS). Table 4 presents an overview of the PFAS detected per product type.

Table 4 Overview of the PFAS substances detected in different product types (bold text indicates PFAS restricted in the POPs regulation)

Product type	Substances detected
Textile flooring (carpet)	**PFOA**, PFHxA, PFHpA, PFPeA
Snow suit	**8:2 FTOH**
Jacket	**PFOA, PFOS + salts, N-Et-FOSA**, **8:2 FTOH,** PFHxA, 6:2 FTS, 6:2 FTOH, PFBS, PFHpA, PFNA, PFDA, PFUnDA, , PFDoDA, PFTeDA
Pants	**PFOA**, **PFOS**, PFHxA, PFHxS, PFHpA, PFNA, PFDA, PFUnA, PFDoA
Gloves	**PFOA**, PFHxA, PFBS, PFNA, PFDA
Shoes	**PFOA**, **PFOS**, PFHxA, 6:2 FTOH, PFHpA, PFUnDA
Backpack	**PFOA**, **PFOS**, PFHxA, 6:2 FTOH, PFBS, PFHxS, PFDA, PFUnDA, PFHpS, PFHpA
Tent/accessories	**PFOA**, PFHxA, 6:2 FTS, 6:2 FTOH, PFNA, PFDA, PFHpA
Firefighting foam concentrate	PFBA, 4:2-FTS, PFHxA, 6:2-FTS
Fire extinguisher	4:2 FTS, PFHxA, 6:2 FTS
Textile impregnation product	PFHxA, 6:2 FTOH

Shoe care product	4:2 FTOH, 6:2 FTOH
Bicycle care product	PFHxA, PFNA, PFDA, PFUnDA, PFDoDA, PFTeDA
Floor care product	6:2 FTOH
Car care product	-
Boat care product	-
Ski wax	**PFOA**, **PFOS**, **8:2 FTOH, 10:2 FTOH,** PFBS, PFPS, PFHxA, 6:2 FTOH, PFHxS, PFHpS, PFHpA, PFDA, PFNA, PFUnDA, PFDoDA, PFTrDA, PFTeDA, PFHxDA, PFODcA

3.5. Extractable organic fluorine (EOF)

The extractable organic fluorine (EOF) analyses were performed for some of the inspected chemical products and articles. The EOF analyses showed high fluorine levels in, for example, ski waxes, textiles and fire extinguisher foams. The EOF analyses gave a clear indication that there are unknown organofluorine compounds in the products that could not be identified in the targeted PFAS analyses in this project. This could originate from both restricted and unrestricted PFAS. Overview of products analyzed for EOF, and results are shown in Table 5.

Table 5 The products tested for extractable organic fluorine (EOF) and the results of EOF-analyses

Product type	Number of products tested	Number of products in which EOF indicate unknown fluorine compounds
Jacket	12	11
Gloves	3	3
Shoes	9	7
Backpack	4	4
Tent/accessories	3	2
Fire extinguisher	5	5
Textile impregnation product	2	1
Shoe care product	4	2
Bicycle care product	7	-
Floor care product	5	-
Ski wax/ski care product	13	10
In total	**67**	**45**

4. Discussion

It is difficult to conclude whether a specific product type contain more PFAS than another. This as the number of PFAS included in the analyses and as the analysis methods differ between the countries, and as the number of products inspected among the different product types varies. The analysis results are however indicating that ski wax is the type of product with the largest presence of non-compliances, and that a major part of all the non-restricted PFAS are found among ski waxes, textiles and bicycle care products.

Regarding the PFOA restriction, it is challenging to enforce due to the absence of a standardized analytical method, which measures PFOA, its salts and PFOA-related substances in different matrices with sufficiently low limits of quantification. Standardized analytical methods are also a prerequisite for test results from different laboratories to be comparable. Another challenge is that the available methods cover only a limited number of PFOA-related compounds. PFOA-related compounds are restricted substances that are considered to have a potential to degrade or be transformed to PFOA (e.g., polymers with ≥ C8 based perfluoroalkyl side chains; 8:2 fluorotelomer compounds and 10:2 fluorotelomer compounds). It has been estimated that there are hundreds of PFOA-related substances on the market, but the substances are largely unknown. Due to the large number of PFAS on the market, there is a need to compile a regularly updated list of substances which are covered by the restriction of PFOA, its salts and PFOA-related substances.

Another shortcoming that was noted regarding current analytical methods is that there is a lack of reference material for most PFAS. The choice of reference material and test method affects the result. Reference materials are used, among other things, for method validation, calibration of analytical instruments or measurement uncertainty calculations. Reference materials can also be used to compare with samples and identify unknown substances. At present, the supply of reference materials for PFAS is limited. The few reference materials available on the market are expensive. The instruments needed to analyse PFAS are also very expensive, which means that the total cost of PFAS analyses is currently very high.

To ensure a proper enforcement of the restrictions, there is a need to develop EU-standardized analytical methods for PFOA, PFOA-related substances and for PFAS in general. In addition, it must be ensured that reference materials are available for PFAS analyses.

As several of the PFAS detected in the inspected products are planned to be restricted or there are ongoing discussions about restrictions (e.g., PFHxA, PFHxS and C9-14 PFCAs), there will be a need to inspect products for compliance regarding these substances once any new restrictions enter into force.

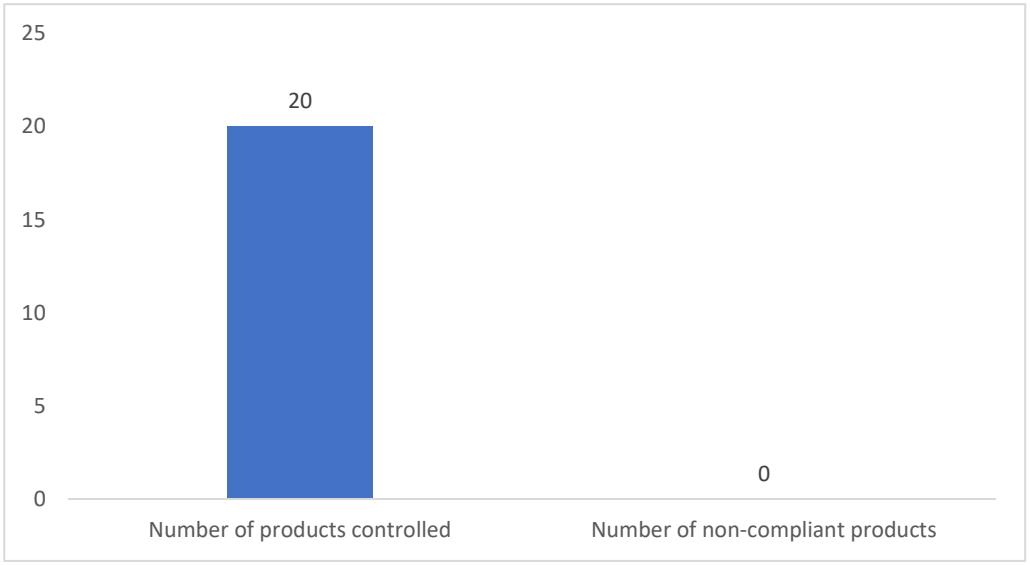
5. References

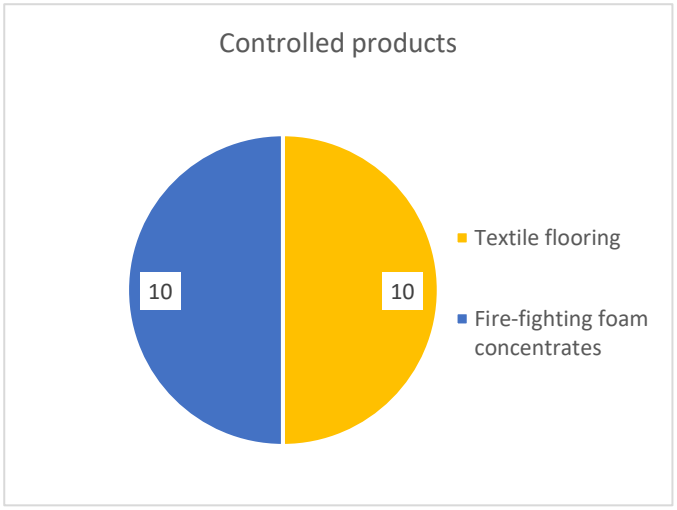
1. Juliane Glüge, Martin Scheringer, Ian T. Cousins, Jamie C. DeWitt, Gretta Goldenman, Dorte Herzke, Rainer Lohmann, Carla A. Ng, Xenia Trier, Zhanyun Wang: An overview of the uses of per- and polyfluoroalkyl substances (PFAS). Environmental Science: Processes & Impacts, 2020, 22, 2345–2373, https://doi.org/10.1039/D0EM00291G.
2. Per- and polyfluorinated substances in the Nordic Countries. Use, occurence and toxicology. TemaNord 2013:542. http://norden.diva-portal.org/smash/get/diva2:701876/FULLTEXT01.pdf
3. Analysis of per- and polyfluorinated substances in articles. Nordic working papers. 2015. http://norden.diva-portal.org/smash/get/diva2:808634/FULLTEXT04.pdf
4. Analysis of PFASs and TOF in products. TemaNord 2017:543. http://norden.diva-portal.org/smash/get/diva2:1118439/FULLTEXT01.pdf
5. PFAS in paper and board for food contact. Options for risk management of poly- and perfluorinated substances. TemaNord 2017:573. http://norden.diva-portal.org/smash/get/diva2:1201324/FULLTEXT01.pdf
6. PFASs in the Nordic environment. Screening of Poly- and Perfluoroalkyl Substances (PFASs) and Extractable Organic Fluorine (EOF) in the Nordic Environment. TemaNord 2019:515. http://www.diva-portal.org/smash/get/diva2:1296387/FULLTEXT01.pdf
7. The cost of inaction. A socioeconomic analysis of environmental and health impacts linked to exposure to PFAS. TemaNord 2019:516. http://norden.diva-portal.org/smash/get/diva2:1295959/FULLTEXT01.pdf

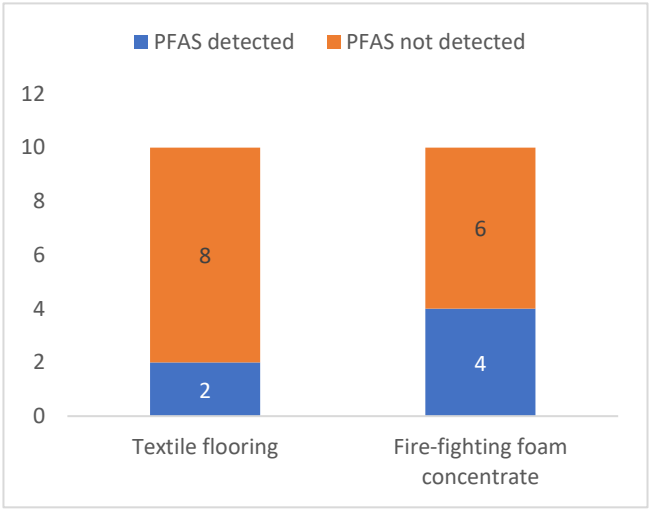
6. Appendices

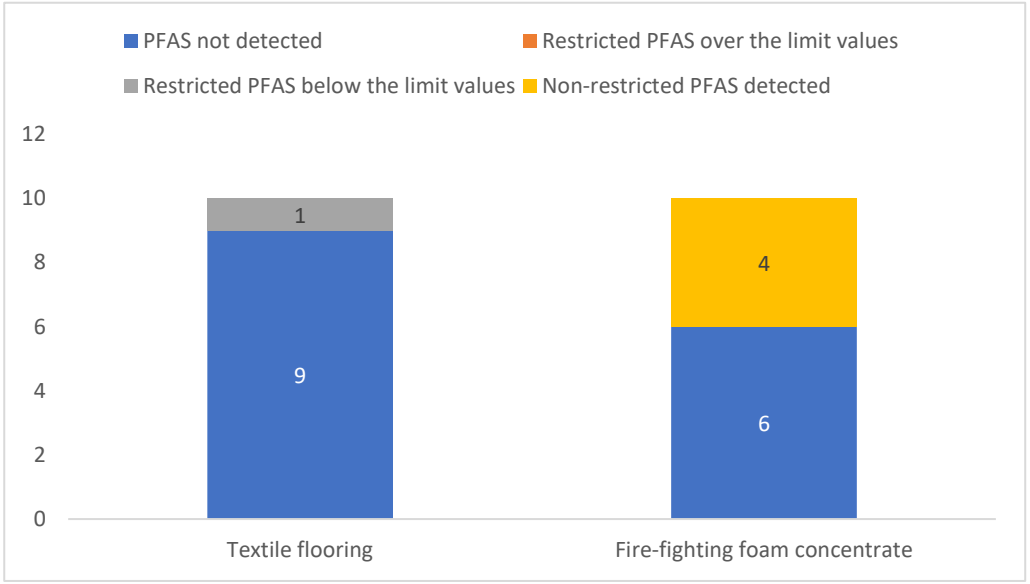
Appendix 1 Results by country

FINLAND

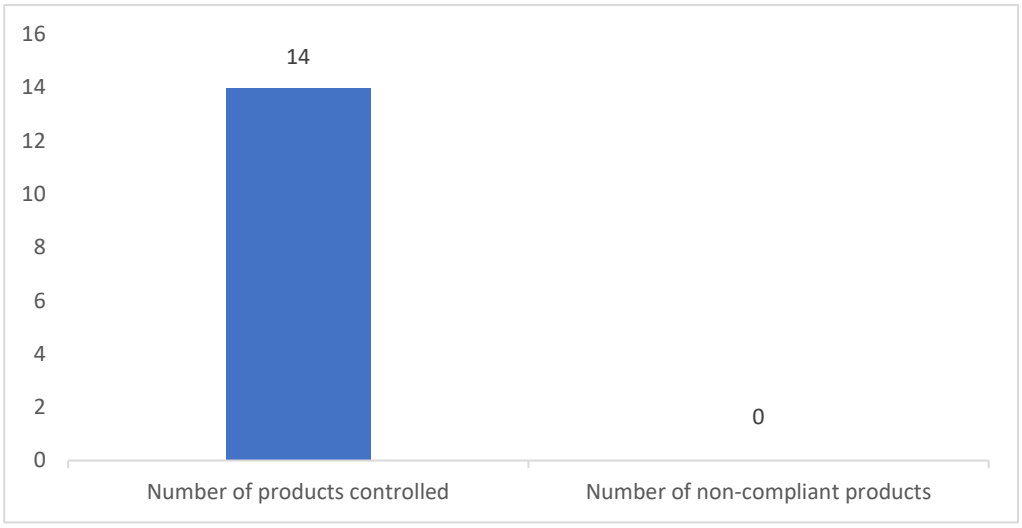


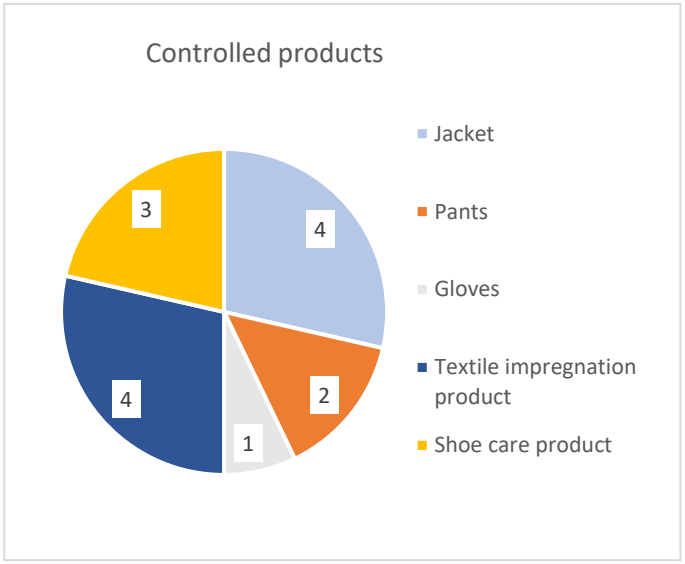


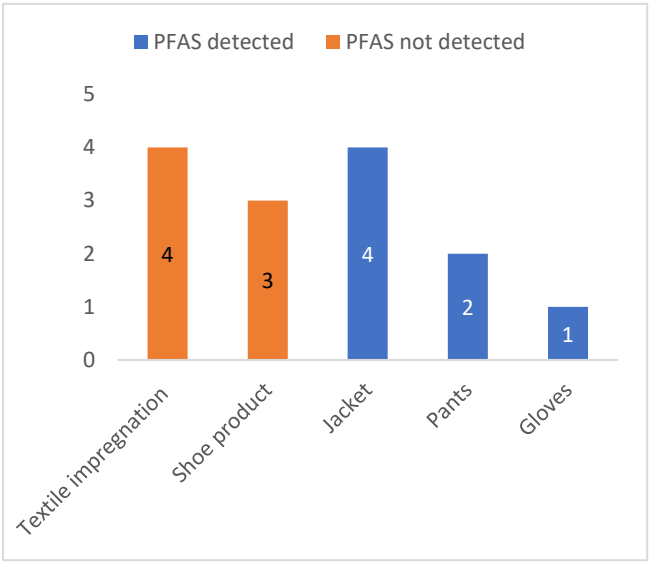


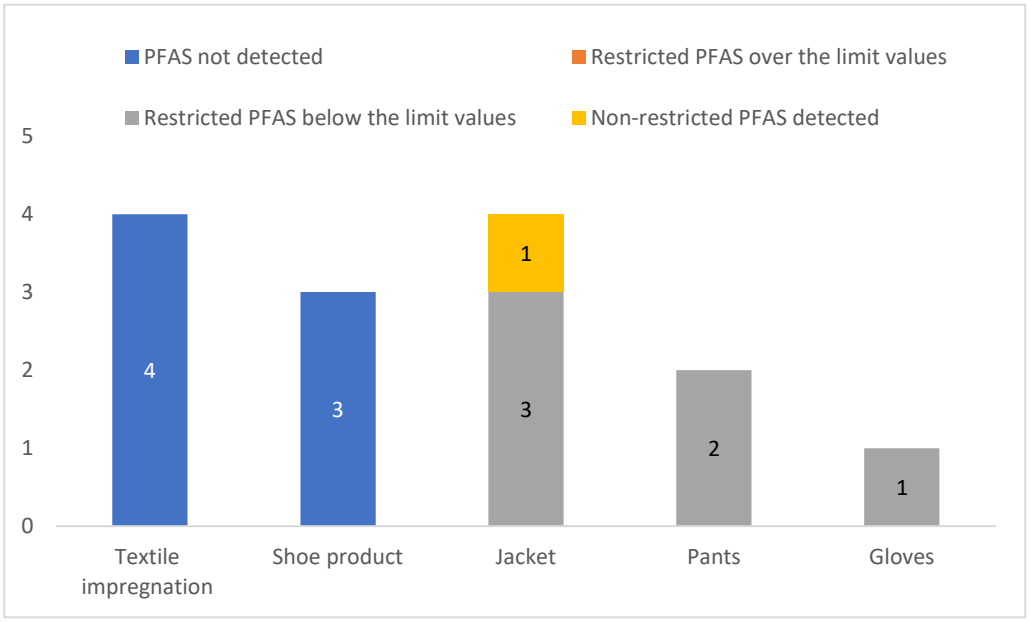


Pilot study

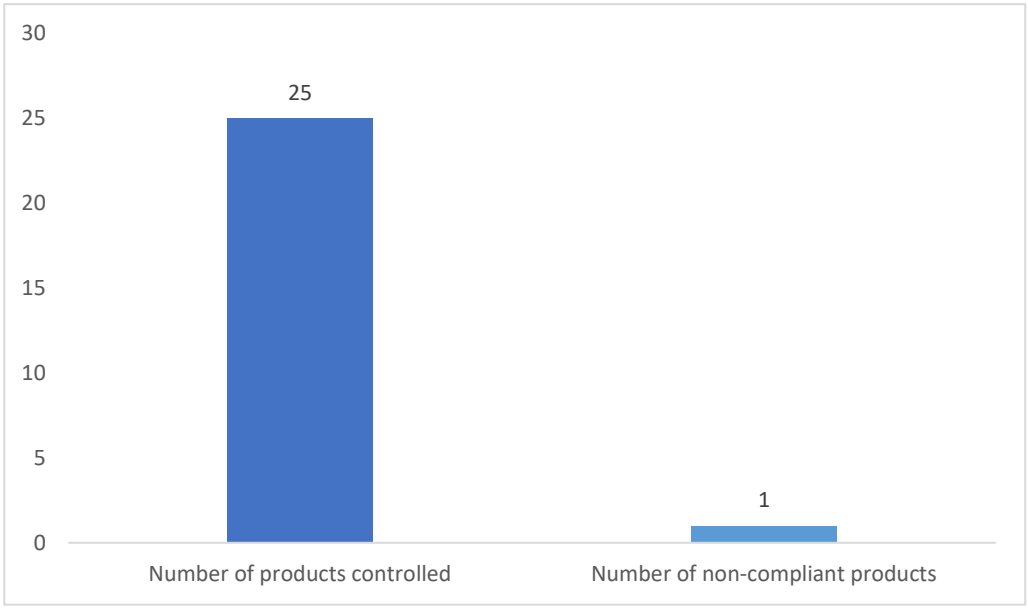

16
14
12
10
8
6
4
2
0
14
0
Number of products controlled
Number of non-compliant products


Controlled products
Jacket
Pants
Gloves
Textile impregnation product
Shoe care product
4
2
1
4
3


PFAS detected
PFAS not detected
5
4
3
2
1
0
4
3
4
2
1
Textile impregnation
Shoe product
Jacket
Pants
Gloves


PFAS not detected
Restricted PFAS over the limit values
Restricted PFAS below the limit values
Non-restricted PFAS detected
5
4
3
2
1
0
4
3
1
3
2
1
Textile impregnation
Shoe product
Jacket
Pants
Gloves

DENMARK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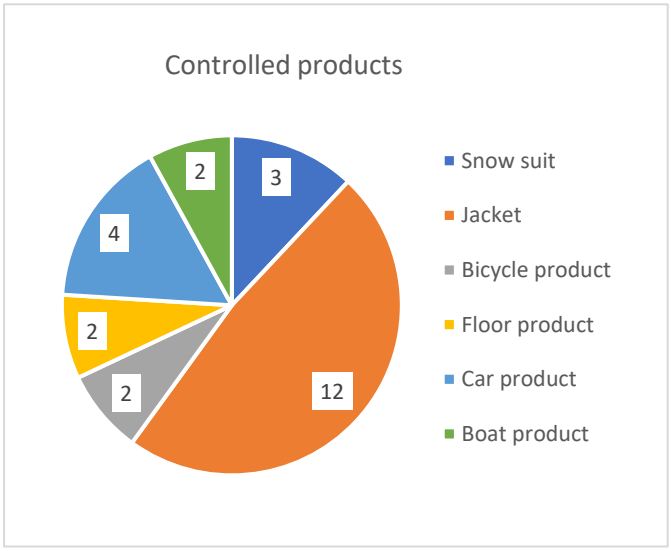
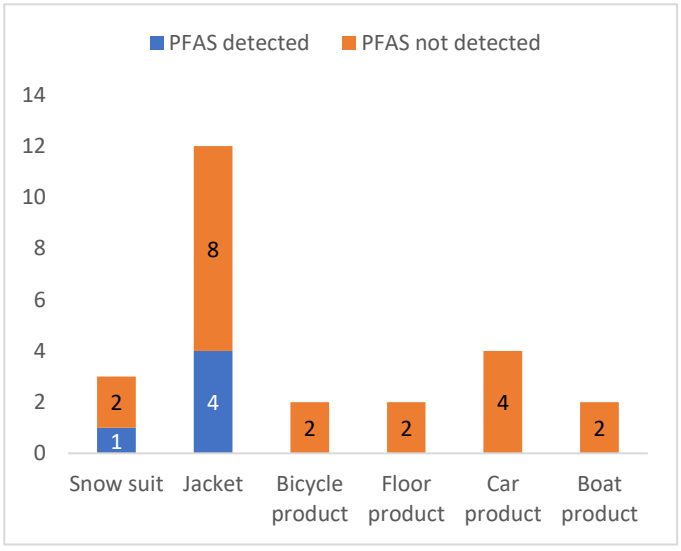
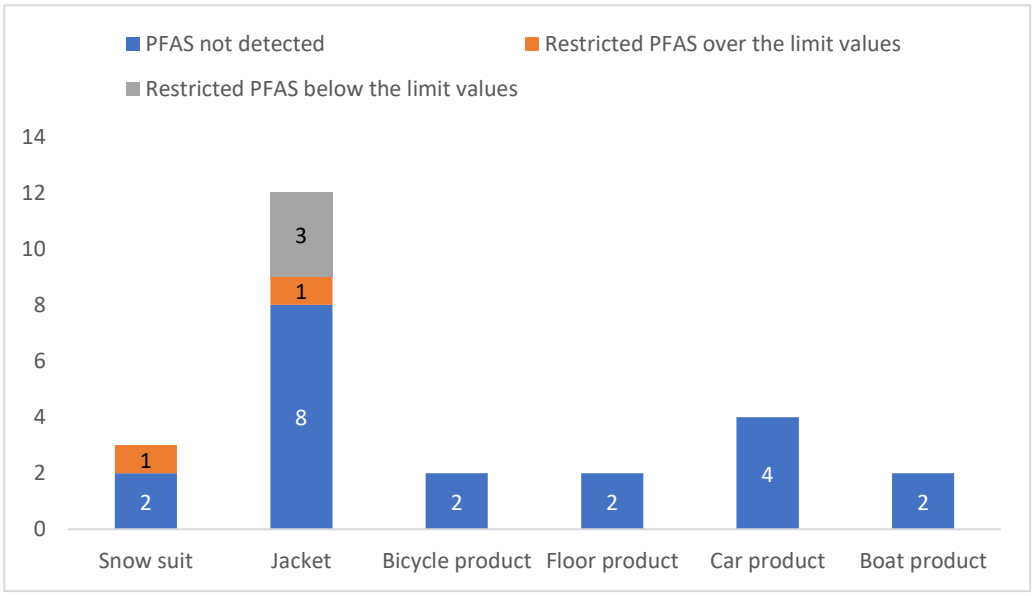
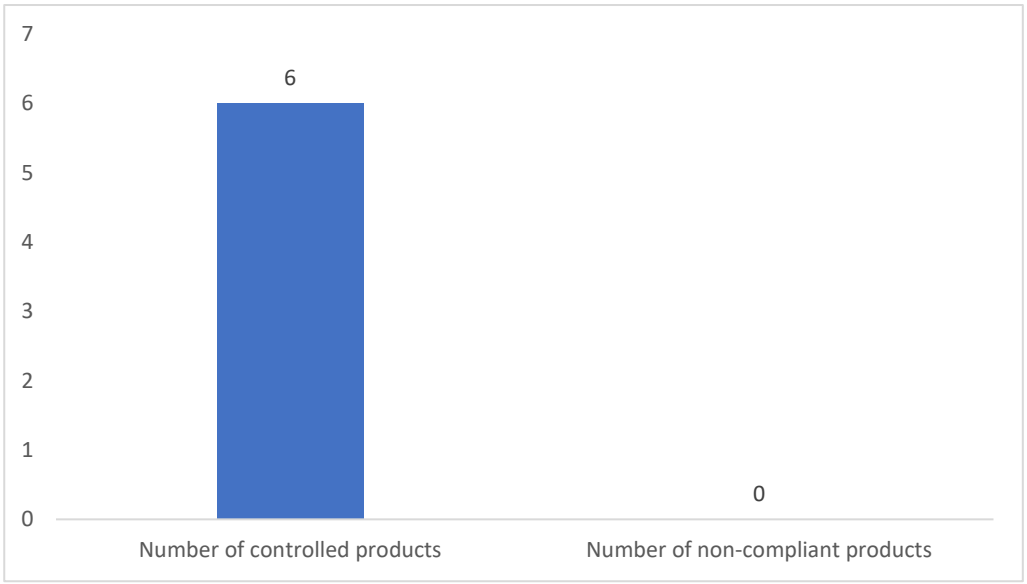
25
20
15
10
5
0
25
1
Number of products controlled
Number of non-compliant products


Controlled products
3
12
2
2
4
2
Snow suit
Jacket
Bicycle product
Floor product
Car product
Boat product


PFAS detected
PFAS not detected
14
12
10
8
6
4
2
0
2
1
8
4
2
2
4
2
Snow suit
Jacket
Bicycle product
Floor product
Car product
Boat product


PFAS not detected
Restricted PFAS over the limit values
Restricted PFAS below the limit values
14
12
10
8
6
4
2
0
1
2
3
1
8
2
2
4
2
Snow suit
Jacket
Bicycle product
Floor product
Car product
Boat product

ICELAND


7
6
5
4
3
2
1
0
6
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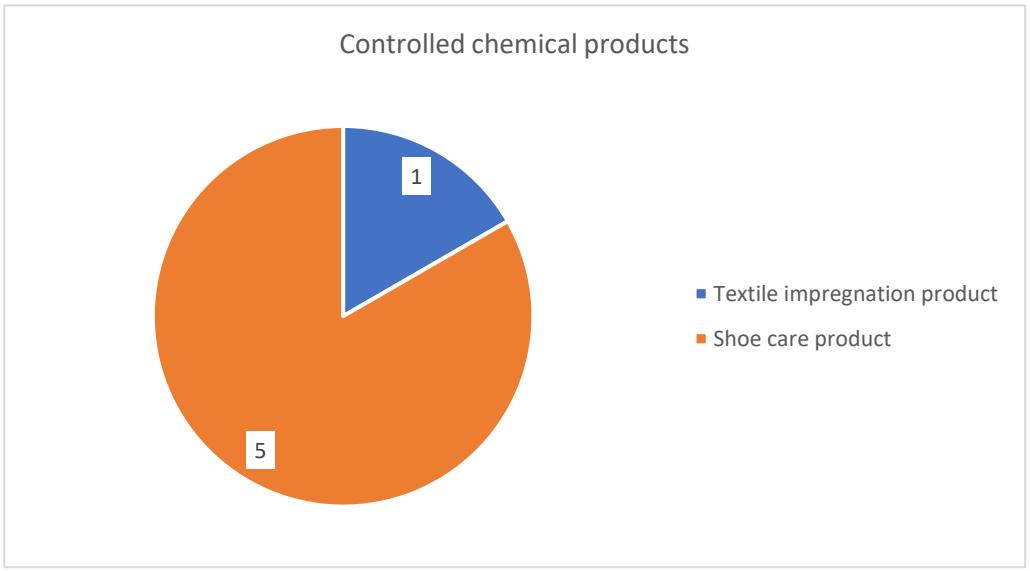
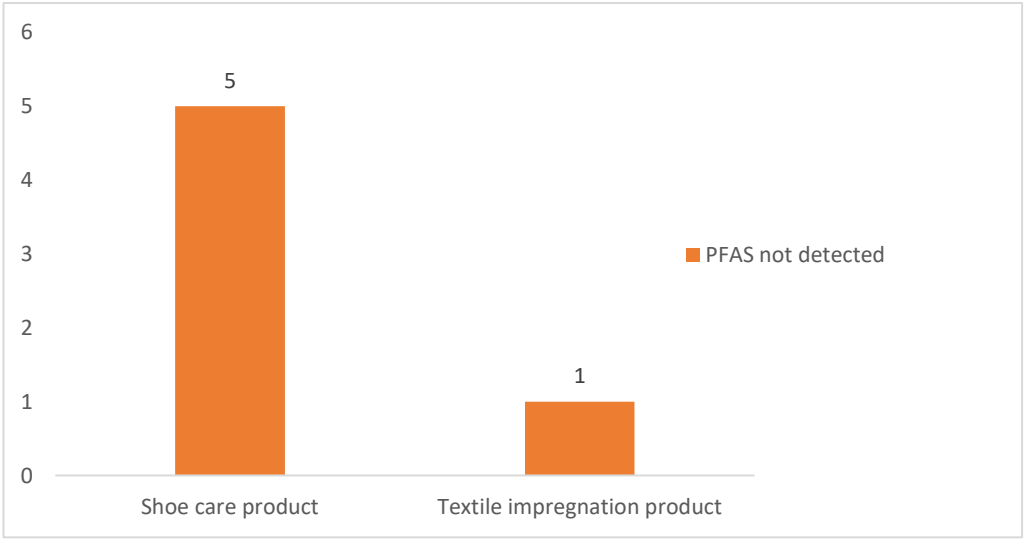
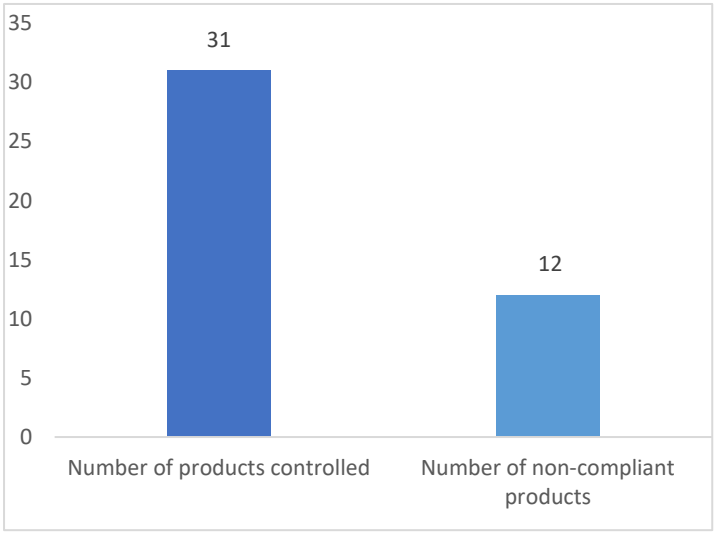
Number of controlled products
Number of non-compliant products


Controlled chemical products
1
5
Textile impregnation product
Shoe care product


6
5
4
3
2
1
0
5
1
PFAS not detected
Shoe care product
Textile impregnation product

NORWAY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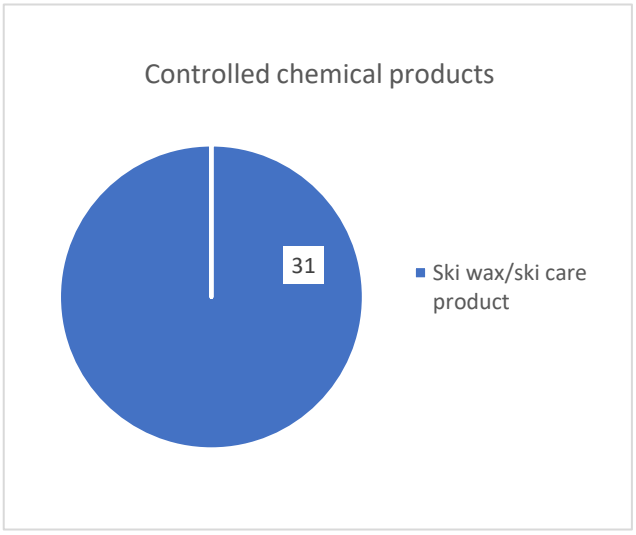
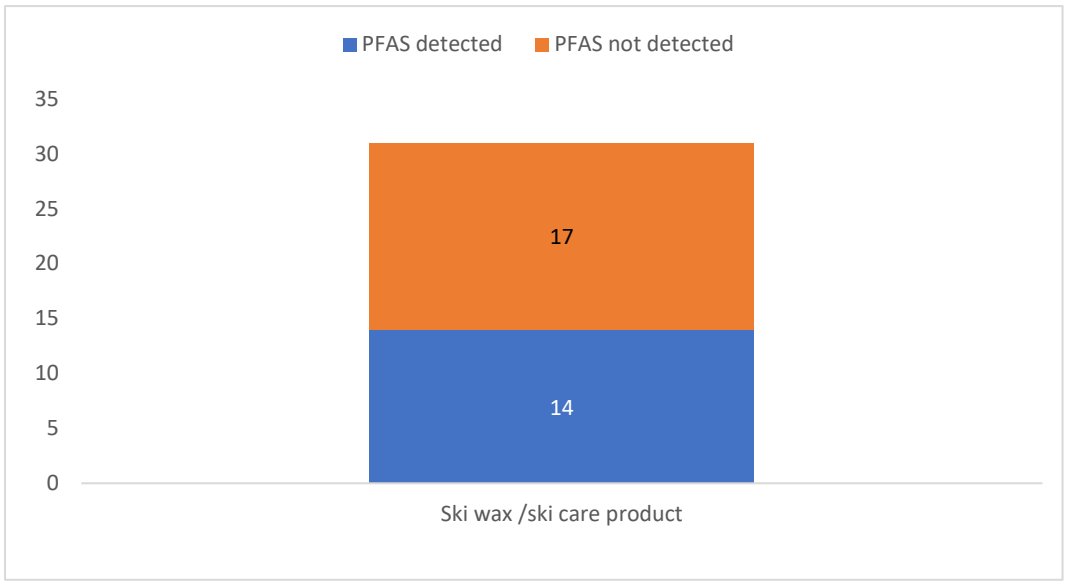
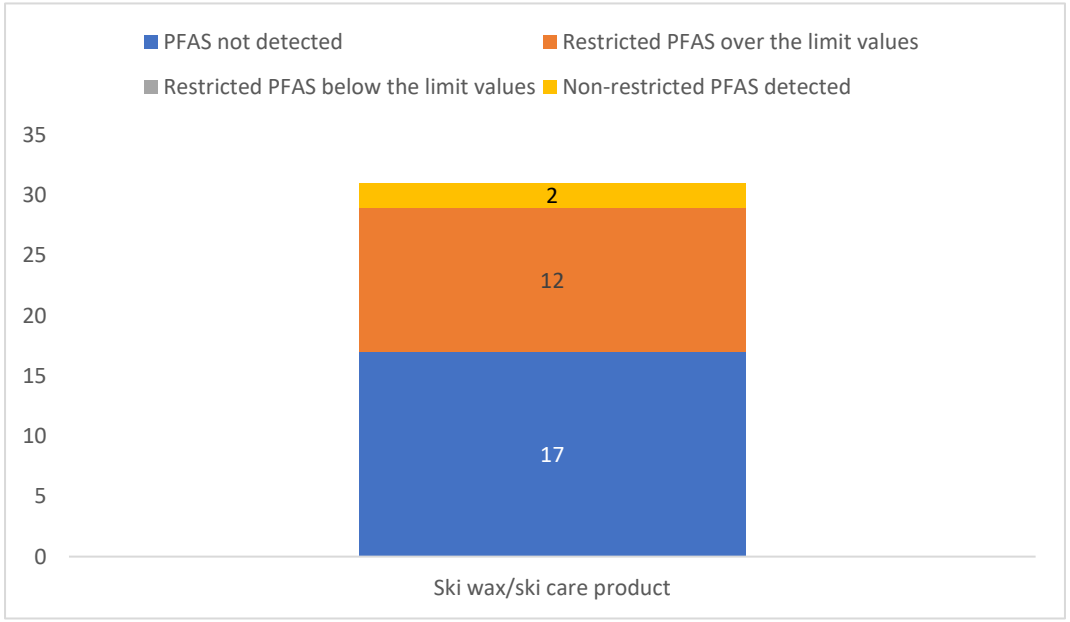
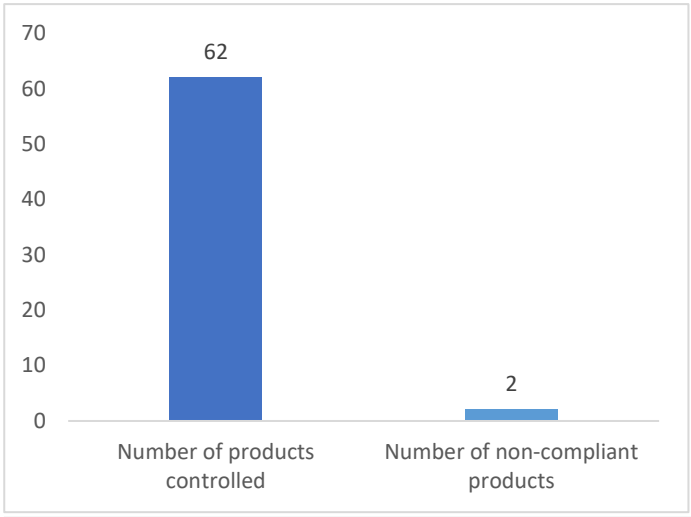
30
25
20
15
10
5
0
31
12
Number of products controlled
Number of non-compliant products


Controlled chemical products
31
Ski wax/ski care product


PFAS detected
PFAS not detected
35
30
25
20
15
10
5
0
17
14
Ski wax /ski care product


PFAS not detected
Restricted PFAS over the limit values
Restricted PFAS below the limit values
Non-restricted PFAS detected
35
30
25
20
15
10
5
0
2
12
17
Ski wax/ski care product

SWEDEN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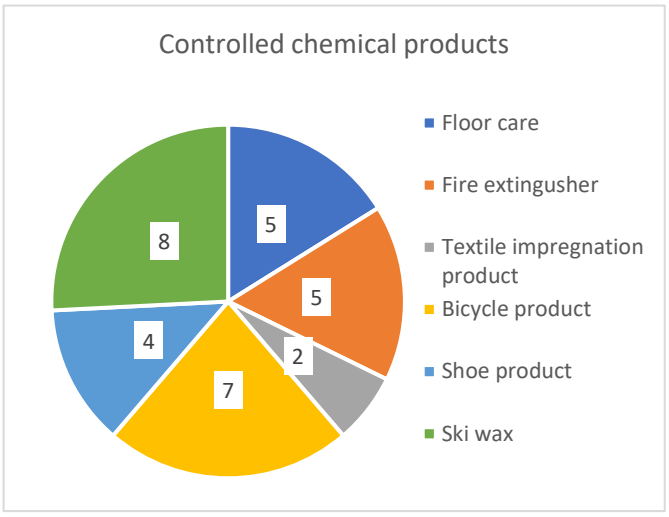
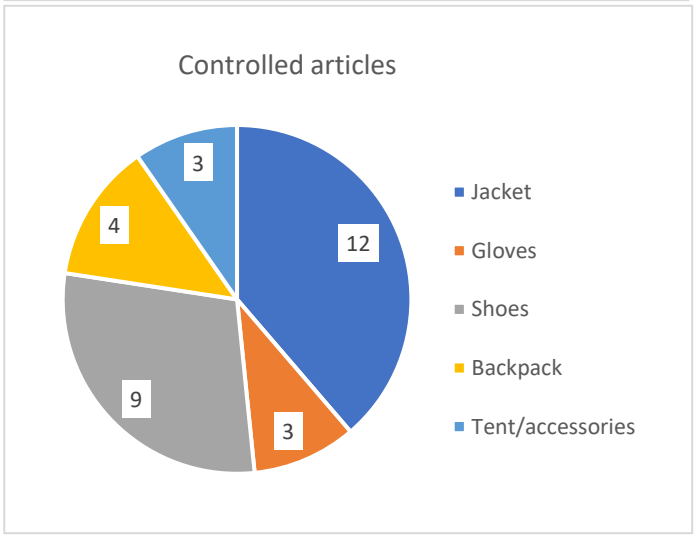
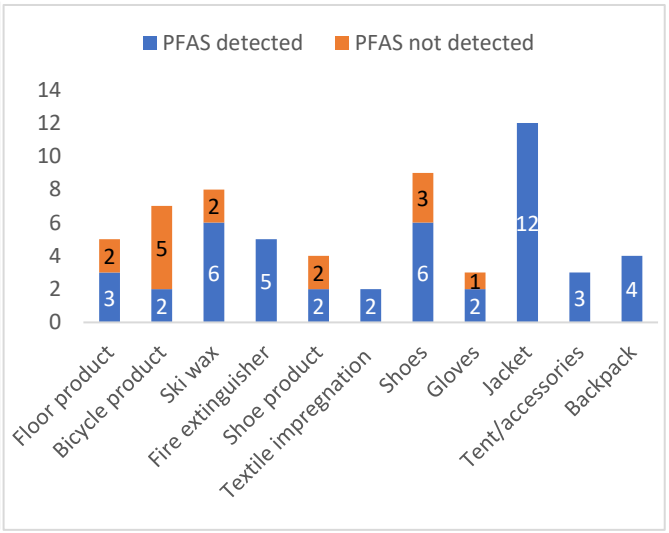
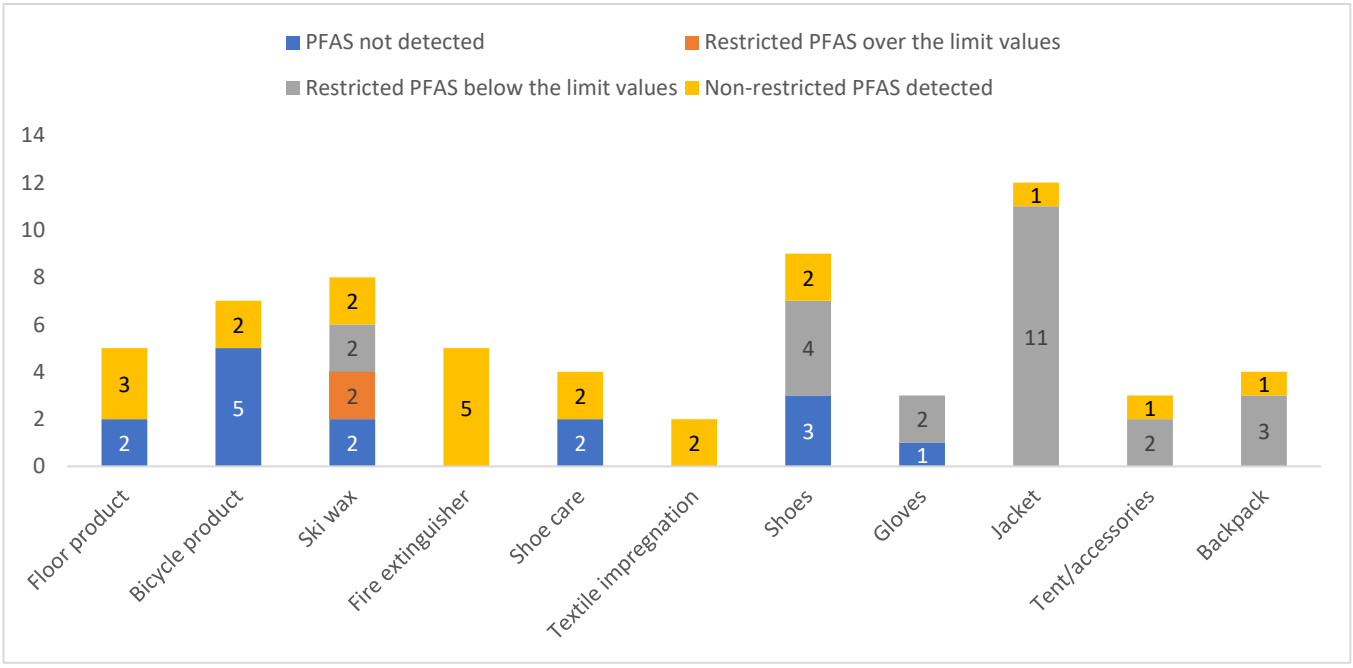
60
50
40
30
20
10
0
62
2
Number of products controlled
Number of non-compliant products


Controlled chemical products
5
5
2
7
4
8
Floor care
Fire extingusher
Textile impregnation product
Bicycle product
Shoe product
Ski wax


Controlled articles
12
3
9
4
3
Jacket
Gloves
Shoes
Backpack
Tent/accessories


PFAS detected
PFAS not detected
14
12
10
8
6
4
2
0
Floor product
Bicycle product
Ski wax
Fire extinguisher
Shoe product
Textile impregnation
Shoes
Gloves
Jacket
Tent/accessories
Backpack


PFAS not detected
Restricted PFAS over the limit values
Restricted PFAS below the limit values
Non-restricted PFAS detected
14
12
10
8
6
4
2
0
Floor product
Bicycle product
Ski wax
Fire extinguisher
Shoe care
Textile impregnation
Shoes
Gloves
Jacket
Tent/accessories
Backpack

Appendix 2 Analytical methods and substances

FINLAND

Fire-fighting foam

Analytical method: EPA 533 (drinking water); ISO 25101 Mod (water quality, LC-MS/MS)

Measured substances:

PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnA, PFDoA, PFTrDA, PFTA, PFHxDA, PFODA, PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS, PFDoS, 4:2 FTS, 6:2 FTS, 8:2 FTS, FhxSA, PFBSA, PFOSA, (HFPO-DA) (GenX), 11Cl-PF3OUdS, ADONA, 5HPFPeA, 9HPFNA, 9Cl-PF3ONS, 8:2 DiPaP, 6:2 DiPaP, EtFOSAA, MeFOSAA, PFEESA, FOSAA, HFPOTeA, HFPO-TA, NFDHA/3,6-OPFHpA, P37DMOA, PFMPA/PF4OPeA, PFECHS, PFMBA/PF5OHxA

Textile flooring

Analytical method: DIN 38414-14 mod., ASTM D7968-17a mod., ISO 25101 mod., CEN/TS 15968 mod (LC-MS/MS)

Measured substances:

PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnA, PFDoA, PFBS, PFHxS, PFHpS, PFOS, PFDS, 4:2 FTS, 6:2 FTS, 8:2 FTS

Outdoor clothes and impregnation sprays (pilot study)

Analytical method: comparable to CEN/TS 15968:2010 (LC-MS/MS)

Measured substances:

PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnA, PFDoA, PFTrA, PFTeA, PFHxS, PFHpS, PFOS, PFDS

DENMARK

Analytical method:

DS/CEN/TS 15968 “Determination of extractable perfluorooctane sulfonate (PFOS) in coated and impregnated solid objects, liquids and fire-fighting foams - Methods for sampling, extraction and analysis by LC-qMS or LC-tandem / MS”. Minor modifications of the reference method were made.

The analysis is done by LC-qMS. However, a few compounds were analyzed by GC-MS (8:2 FTOH, 8:2-FTAC, 8:2-FTMAC).

Measured substances:

PFOS, PFOA, 8:2-FTOH, N-Me-FOSA, N-Et-FOSA, N-ME-FOSE, N-Et-FOSE, 8:2-FTS, 8:2- FTAC, 8:2-FTMAC, 8:2 PAP, 8:2 diPAP

ICELAND

Analytical method: GLS OC 400:2019-01-18 similar to DIN 38414-14

Measured substances: PFOS, PFOA, PFBS, PFBA, PFPeA, PFHxS, PFHxA, PFHpS, PFHpA, PFOSA, PFNA, PFDS, PFDeA, PFUnDA, PFDoDA, PFTrDA, PF-3,7-DMOA, HPFHpA, 6:2 FTS, 4:2 FTS, 8:2 FTS

NORWAY

Analytical method:

Ionic and volatile PFAS were extracted and analysed according to the method described in Blom and Hanssen (2015). Short, a known amount of sample was measured and weighted before extraction with methanol on ultrasonic bath. The extract was up-concentrated and split in two for further analysis of PFAS and EOF. In total 20 different 13C-labelled internal standards were added to the extract followed by analysis on LC-MS/MS and GC-MS.

Quality assurance: A blank sample was processed with every batch of sample. Three parallels of a textile sample were spiked with native PFAS for control of repeatability and recovery.

For PFOS, the method is equivalent to the standardised method DS/CEN/TS 15968:2010,” Determination of extractable perfluorooctanesulphonate (PFOS) in coated and impregnated solid articles, liquids and firefighting foams - Method for sampling, extraction and analysis by LC-qMS or LC-tandem/MS”.

Measured substances:

4:2 FTS, 6:2 FTS, 8:2 FTS, 10:2 FTS, PFBS, PFPS, PFHpS, PFHxS, PFOS, PFNS, PFDS, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoDA, PFTrDA, PFTeDA, PFHxDA, PFOcDA, FOSA, N-Et-FOSE, N-Me-FOSAA, N-Et-FOSAA, 4:2 FTOH, 6:2 FTOH, 8:2 FTOH, 10:2 FTOH, N-Me-FOSA, N-Et-FOSA, N-Me-FOSE, N-Et-FOSE

SWEDEN

Analytical method:

Ionic and volatile PFAS were extracted and analysed according to the method described in Blom and Hanssen (2015). Short, a known amount of sample was measured and weighted before extraction with methanol on ultrasonic bath. The extract was up-concentrated and split in two for further analysis of PFAS and EOF. In total 20 different 13C-labelled internal standards were added to the extract followed by analysis on LC-MS/MS and GC-MS.

Quality assurance: A blank sample was processed with every batch of sample. Three parallels of a textile sample were spiked with native PFAS for control of repeatability and recovery.

For PFOS, the method is equivalent to the standardised method DS/CEN/TS 15968:2010," Determination of extractable perfluorooctanesulphonate (PFOS) in coated and impregnated solid articles, liquids and firefighting foams - Method for sampling, extraction and analysis by LC-qMS or LC-tandem/MS".

Measured substances:

4:2 FTS, 6:2 FTS, 8:2 FTS, 10:2 FTS, PFBS, PFPS, PFHxS, PFHpS, PFOS, PFNS, PFDS, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoDA, PFTrDA, PFTeDA, PFHxDA, PFODA, FOSA, N-MeFOSAA, N-Et-FOSAA, 4x3 PFECA, GenX, 4:2 FTOH, 6:2 FTOH, 8:2 FTOH, 10:2 FTOH, N-MeFOSA, N-EtFOSA, N-MeFOSE, N-EtFOSE