Mattilsynet

DTU

PFAS in paper and board food contact materials

SELECTED SAMPLES FROM THE NORWEGIAN MARKET 2024

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PFAS in paper and board food contact materials

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Innholdsfortegnelse

Sammendrag

Per- og polyfluorerte alkylstoffer (PFAS) er menneskeskapte kjemikalier som har attraktive industrielle egenskaper, som at de er olje- og vannavstøtende. De kan derfor brukes som belegg på papir og papp som kommer i kontakt med mat (FCM) for å forbedre funksjonaliteten. PFAS' høye kjemiske stabilitet gjør dem motstandsdyktige mot biologisk nedbrytning, noe som utgjør en risiko for menneskers helse og miljøet. Negative helseeffekter knyttet til PFAS er blant annet hormonforstyrrende effekter på fertilitet, graviditet, spedbarnsutvikling og skjoldbruskkjertelfunksjon, økt kolesterol og reduserte antistoffresponser på barnevaksiner. I 2020 publiserte Den europeiske myndighet for mattrygghet (EFSA) en risikovurdering for mattrygghet som etablerte et tolerabelt ukentlig inntak per kg kroppsvekt (TWI) på 4,4 ng/kg kroppsvekt for Σ4PFAS (PFOA, PFNA, PFHxS, PFOS).

Det finnes foreløpig ingen spesifikk EU-forordning som dekker matkontaktmaterialer av papir- og papp. Men i rammeforordning 1935/2004 om materialer beregnet på å komme i kontakt med mat, står det at materialer må produseres slik at de ikke overfører kjemikalier til mat i mengder som kan være helsefarlige. Mattilsynet gjennomførte i 2024 en undersøkelse av PFAS i matkontaktmaterialer i samarbeid med Danmarks Tekniske Universitet, Danmarks Matinstitutt (DTU Food), på grunn av risikoen for migrering av PFAS fra matkontaktmaterialer til mat. Mattilsynet tok ut 30 matkontaktmaterialer av papir- og papp fra det norske markedet i perioden mars-juni 2024, og sendte prøvene til DTU Food. Ved DTU Food ble det utført migrasjonsstudier ved hjelp av en akkreditert LC-MS/MS-metode. De ekstraherbare mengdene PFAS ble undersøkt, og migrasjonstester ble utført i elleve prøver med kvantifiserbart PFAS-innhold. Deretter ble det utført ikke-målrettet screening av næringsmiddelsimulanter.

PFAS ble funnet i ekstrakter av 11 av 30 analyserte matkontaktmaterialer, nærmere bestemt i tre muffinsformer, tre papirsugerør, tre pizzaesker, et popcornbeger og en papptallerken.

Summen av konsentrasjonene av 11 analyserte perfluorkarboksylsyrer (PFCA) og 4 polyfluorsulfonsyrer (PFSA) i ekstraktene varierte fra 0,006 til 912 µg/kg mat. Fluortelomeralkoholer ble påvist i fire prøveekstrakter med konsentrasjoner på 0,503–9,161 µg/kg. MonoPAP-er og diPAPS ble funnet i kvantifiserbare mengder kun i én pizzaeske (0,05 µg/kg). Alle de elleve prøvene der PFAS ble funnet i kvantifiserbare mengder, ble undersøkt med migrasjonstesting i triplikater (tre omganger). Den høyeste migrasjonen av summen av PFAS ble funnet i popcornbeger og muffinsformer (ΣPFAS: 9.028; 3.145; 2.333 µg/kg mat). PFAS ble påvist i lavere mengder, i motsetning til den forrige undersøkelsen som ble utført i 2017. Det ble observert en potensiell migrasjon av PFAS fra matkontaktmaterialer til matvarer. Dette indikerer at matkontaktmaterialer kan være en kilde til PFAS i mat. Det bør også gi en økt bevissthet om tilstedeværelsen av slike stoffer, og viktigheten av å begrense bruken.

En ikke målrettet screening av prøvene avdekket 5200 egenskaper. Av disse hadde 1159 tre eller flere fluoratomer, med en nøyaktighet under 2 ppm. Ingen av de målrettede forbindelsene i det interne biblioteket ble funnet i de analyserte matkontaktmaterialene.

Summary

Per- and polyfluorinated alkyl substances (PFAS) are anthropogenic chemicals possessing some attractive industrial properties such as oil and water repellent. Therefore, they can be used as coatings of paper and board food contact materials (FCM) improving their functionality. However, high chemical stability of PFAS makes them resistant to biological degradation, posing a risk to human health and the environment. Endocrine-disrupting effects on fertility, pregnancy, infant development, and thyroid hormone function, increased cholesterol and reduced antibody responses to childhood vaccines are examples of adverse health effects related to PFAS. Thus in 2020, the European Food Safety Authority published a food safety risk assessment established a tolerable weekly intake per kg body weight (TWI) of 4.4 ng/kg_{bw} for Σ4PFAS (PFOA, PFNA, PFHxS, PFOS).

So far, there is no specific EU regulation covering paper and board FCM, but in Regulation 1935/2004 on materials intended to come into contact with food, it is stated that materials must be manufactured in such a way that they do not transfer chemicals to food in quantities that could endanger human health. Due to the risk of migration of PFAS from FCM to food, a survey on PFAS in FCM was conducted by the Norwegian Food Safety Authority in collaboration with the Technical University of Denmark, the National Food Institute (DTU Food). During the period March-June 2024, 30 paper and board FCM from the Norwegian market were sampled by the Norwegian Food Safety Authority, covering five main regions of Norway, and collected samples were sent to DTU Food. At DTU Food migration studies using accredited LC-MS/MS method was performed. The extractable amounts of PFAS from the sampled FCM were investigated and migration tests were conducted in eleven samples with the quantifiable PFAS contents. Subsequently, complimentary non-targeted screening of food simulants was performed.

PFAS was found in extracts of 11 of 30 FCM analysed; in three muffin forms, three paper straws, three pizza boxes, a popcorn beaker and paper plate.

The sum of concentrations of 11 analysed perfluorcarboxylic acids (PFCA) and 4 polyfluorsulphonic acids (PFSA) in the extracts ranged from 0.006-912 µg/kg food. Fluortelomer alcohols were detected in four sample extracts at concentrations 0.503-9.161 µg/kg. MonoPAPs and diPAPS were found at quantifiable amount only in one pizza box (0.05 µg/kg). All eleven samples in which PFAS were found in quantifiable amounts were subjected to migration tested in triplicates. The highest migration of the sum of PFAS were found in popcorn beaker and muffin forms (ΣPFAS: 9.028; 3.145; 2.333 µg/kg food). In contrast to the previous investigation conducted in 2017, PFASs were detected at lower amounts. A potential migration of PFASs from FCM into foods was observed, indicating that FCM could be a source of PFASs in food, but also rising awareness of such phenomena and limited use.

Nontargeted screening reviled the presence of 5200 features were in the analysed samples, of which 1159 were suggested to contain three or more fluorine atoms with accuracy <2 ppm. However, none of the target compounds included in the in-house library was identified in any of the FCM present in the study.

1 Preface

This investigation was performed in cooperation between Project Leader Signe Sem from the Norwegian Food Safety Authority, Chemical Safety and EEA Section, Post-doctoral researcher Agnieszka A. Niklas, and senior adviser Tommy Licht Cederberg from the Technical University of Denmark, National Food Institute, Research Group for Analytical Food Chemistry.

The laboratory work on extraction and migration testing of food contact material (FCM) samples and chemical analyses of per- and polyfluorinated substances (PFAS) was performed by Laboratory Technician Lene Gram Hansen in collaboration with Post-doctoral researcher Agnieszka A. Niklas.

The non targeted screening was performed by research assistant Flavia Gravina under supervision of Post-doctoral researcher Agnieszka A. Niklas.

The report was prepared by Post-doctoral researcher Agnieszka A. Niklas, DTU in cooperation with research assistant Flavia Gravina, DTU, senior adviser Tommy Licht Cederberg DTU, and project Leader Signe Sem and Julie Tesdal Håland, The Norwegian Food Safety Authority. The DTU DOC-number was 23/1018042 and the Norwegian Food Authority' Elements number was 2023/244629.

2 Introduction

2.1 Background

Per- and polyfluorinated alkyl substances (PFAS) comprise a large group of chemicals, which can be used to coat food contact materials (FCM) made of paper and board, making them resistant to fat and water. These coated packaging materials are typically intended for greasy foods, high-temperature use, or long-term food storage. Examples of PFAS-coated packaging include cupcake liners, cups, plates, paper wrapping, and packaging for storing dry foods ((Begley et al., 2005); (Trier, 2011); (Yuan et al., 2016); (Schaider et al., 2017)).

PFAS are persistent in the environment, and some of these substances and their transformation products exhibit multiple adverse health effects, including endocrine disruption, carcinogenicity, suppression of immune response to vaccination, and harm to fetuses ((Olsen et al., 2007); (White et al., 2011); (Fei et al., 2007); (Schrenk et al., 2020)). The European Food Safety Authority ((Knutsen et al., 2018)) has evaluated perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS), with other PFAS currently under evaluation. They report estimated human half-lives for PFOS and PFOA of approximately 5 years and 2–4 years, respectively. EFSA has based health-based guidance values on epidemiological studies: For PFOS, the effects of increased cholesterol in adults and decreased antibody response to vaccination in children led to the establishment of a tolerable weekly intake (TWI) of 13 ng/kg body weight (bw). Increased cholesterol was the main critical effect for PFOA, leading to a TWI of 6 ng/kg bw, although reduced birth weight and elevated serum levels of the liver enzyme alanine aminotransferase (ALT) were also considered. EFSA concludes that PFAS exposure for a significant portion of the population exceeds the proposed TWIs; it should be noted that the contribution of PFAS from FCM migration is not included in the exposure calculations. In 2020, EFSA published a food safety risk assessment (Schrenk et al., 2020) and established a tolerable weekly intake per kg body weight (TWI) of 4.4 ng/kg bw for Σ4PFAS (PFOA, perfluorononanoic acid (PFNA), perfluorohexane sulfonic acid (PFHxS), PFOS).

Previous results from DTU have shown the presence of PFAS in various packaging materials, including popcorn and pancake bags, interlayer papers, dry food bags (such as breakfast products and ready-made mixes), pizza and fritters packaging ((Trier, Granby, et al., 2011)(Trier, Nielsen, et al., 2011)(Trier, 2011)). In 2009 and 2011, a screening project on the content of PFAS in paper and cardboard revealed PFAS in one-third of the samples. Migration testing to food simulants of selected samples demonstrated migration from microwave popcorn packaging, baking paper, and sandwich paper. Control projects in 2013, with official sampling by the Danish Veterinary and Food Administration (DVFA), showed a lower frequency (4%) and lower content of PFAS in 46 samples from packaging and food businesses. Conversely, samples taken by DTU at the retail level had PFAS content in 12 of 27 samples. In 2015, control of 26 samples of paper and board FCM by DVFA only showed PFAS content above the detection limit in a muffin form, corresponding to 0.78 PFOA equivalents/kg food.

In the 2015 project conducted by DTU, The National Food Institute, and the Norwegian Food Safety Authority, PFAS was not found in 53 Norwegian FCM samples (Trier et al., 2016). However, in the 2017 project, PFAS was detected in extracts from 13 of the 35 FCM analysed (Granby & Håland, 2018). This contrasts with the previous investigation conducted in 2015, where PFASs were not detected in several products.

A PhD study at DTU Food (2019-2021) demonstrated not only the migration of PFAS from paper and board to food simulants but also to real food after high-temperature applications ((Lerch, 2022; Lerch et al., 2022, 2023)). This includes migration from plates used in microwaves during food preparation and muffin baking in paper forms.

Nomenclature of PFAS

The names and chemical structures of PFAS classes relevent for this project are presented in Figure 1. The classes analysed for in the present study include the perfluorocarboxylic acids (PFCA, the subgroup which includes perfluoroctanoic acid; PFOA); the perfluorosulphonic acids (PFSA, the subgroup which includes perfluoroctane sulphonic acid; PFOS), perfluorosulphone amides (PFSAA), fluorotelomer alcohols (FTOH); mono-polyfluoroalkyl phosphate esters (monoPAPS); di-polyfluoroalkyl phosphate esters (diPAPS).

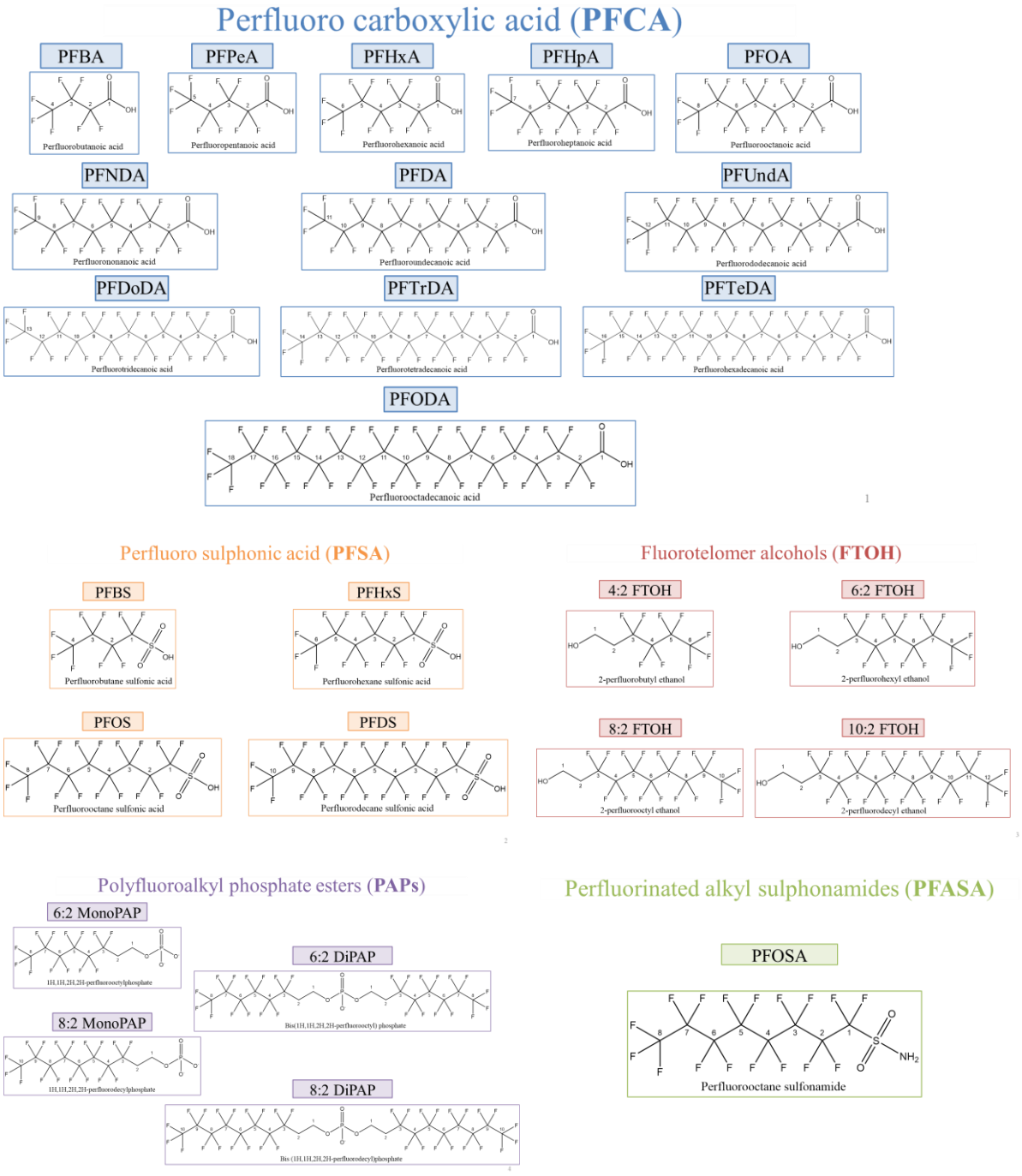
2.2 Regulations concerning PFAS in FCM

There is no specific EU legislation for food contact materials made of paper and board, but Regulation 1935/2004 of 27 October 2014 on materials and articles intended to come into contact with food covers all types of food contact materials (European Commission, 2004). Article 3 of this regulation states that materials and articles must be manufactured in compliance with good manufacturing practices so that, under normal and foreseeable conditions of use, they do not transfer their constituents to food in quantities that could endanger human health.

A global phase-out of several previously used perfluorinated substances is underway, and recently, at the European level through REACH, a regulation of PFOA and related substances has been adopted, effective from July 2020 (Regulation EU 2017/1000). In 2014, Germany and Norway jointly proposed to ECHA (the European Chemical Agency) a restriction on PFOA and PFOA precursors in consumer products, setting a limit of 2 µg/kg. They assessed that human exposure to PFOA is excessively high, necessitating a reduction in exposure to PFOA and related compounds. The Norwegian Environment Agency has prohibited the presence of PFOA in consumer products. In 2009, perfluorooctane sulfonic acids and their salts were added to Annex B: Persistent Organic Pollutants (POPs) (restriction) of the Stockholm Convention.

In Denmark, from July 2020, has prohibited placing on the market paper and board FCM in which PFAS has been intentionally added. The indicator value is 20 microgram of total organic fluorine (TOF) per gram of paper.

Figure 1 The names and chemical structures of PFAS classes relevant for this project.



2.3 Aim of the project

The aim of the present project is to analyse samples of relevant food contact materials made of paper and board for PFAS content and migration. The project surveys PFAS in paper and board food contact materials in the Norwegian market.

3 Materials and methods

3.1 Sampling

The FCM were sampled early in the first two quarters of 2024 and received at The Technical University of Denmark from March to May 2024.
Samples of paper and board FCM were collected from importers or retail shops in Norway. Relevant samples included paper and board FCM in direct contact with food, such as muffin cups, paper straws, fast food packaging, pizza trays, coffee/teacups, and bags for microwave popcorn—products with grease and water-repellent surfaces. Sampling was conducted in five Norwegian Food Safety Authority regions: "Greater-Oslo", "North", "Middle", "East," and "South and West." Food inspectors from these regions forwarded the samples to DTU along with the accompanying documentation. DTU received in total 30 samples divided in subgroups of paper and board FCM as shown in Table 3. Detailed sampling information is given in Annex B. Extracts from all samples were analysed for PFAS contents. Next, migration tests using simulants were performed for ten samples with quantifiable PFAS contents.

Table 1. Types of paper & board Food Contact Material samples analysed in the project with the region of origin.

Region	Sample
Nord	Airfryer paper, Food bags, Muffin cup
Sør og Vest	Popcorn bag x2, Muffin cup x2, Paper straws x2, Pizza boxes x2
stor Oslo	Paper straws x2, Popcorn bag x2, Pizza boxes x2, Muffin cup x2
Midt	Pizza boxes x2, Paper straws, Paper plates
Øst	Popcorn bag x2, Pizza box, Muffin cup x2, AirFryer paper

3.2 PFAS analysis

An accredited analytical method (FC430) developed at DTU, based on liquid chromatography tandem mass spectrometry (LC-MS/MS) were used for the PFAS analyses.

Extraction, migration and calibration standards

Perfluorocarboxylic acids (PFCA)/perfluoro sulphonic acids (PFSA):

Carved square pieces of 6 cm2 FCM were extracted with 1.00 mL ethanol by placing the FCM samples in microcentrifuge tubes in an ultrasonication bath at 60°C for 1 hour. Subsequently 210 µL extract was transferred to a 0.2 µm filter vial (Whatman Mini uniprep polypropylen 500 µL), together with 40 µL C13-labelled internal standard mixture (final internal standard concentrations 0.5 ng/mL).

For the migration testing 6 cm2 FCM sample was added 1.50 mL 50% ethanol:water (simulant) and heated for 2 hours at 70°C in a heating block, after which a quantity was transferred to a filter vial and added internal standard as described above.

Quantitation was performed using external calibration standards at 0; 0.1; 0.3; 0.5; 2; 5 ng/mL of perfluorocarboxylic acids (PFCA) / polyfluorosulphonic acids (PFSA) added several corresponding C13-labelled internal standards of 0.5 ng/mL.

Perfluoroalkyl phosphates (PAPs):

Carved square pieces of 6 cm2 of FCM were extracted with 1.00 mL 50% ethanol:water by placing the FCM samples in microcentrifuge tubes in an ultrasonication bath at 60°C for 1 hour. Subsequently 240 µL extract was transferred to a 0.2 µm filter vial (Whatman Mini uniprep polypropylen 500 µl), together with 10 µL C13-labelled internal standard mixture (final internal standard concentrations 5 ng/mL).

For the migration testing 6 cm2 FCM sample was added 1.50 mL 50% ethanol:water (simulant) and heated for 2 hours at 70°C in a heating block, after which a quantity was transferred to a filter vial and added internal standard as described above.

Quantitation was performed using external calibration standards of 0, 1, 2, 5 ng/mL of perfluoroalkyl phosphates (PAPs) added several corresponding C13-labelled internal standards of 5 ng/mL. Samples for which PFAS concentrations exceeded the calibration curves were diluted prior to addition of internal standard and repeated analysis by LC-MS.

Fluorotelomer alcohols (FTOH)

Carved square pieces of 6 cm2 of FCM were extracted with 1.00 mL 50% ethanol:water by placing the FCM samples in microcentrifuge tubes in an ultrasonication bath at 60°C for 1 hour. Subsequently 225 µL extract was transferred to a 0.2 µm filter vial (Whatman Mini uniprep polypropylen 500 µl), together with 25 µL C13-labelled internal standard mixture (final internal standard concentrations 20 ng/mL).

For the migration testing 6 cm2 FCM sample was added 1.50 mL 50% ethanol:water (simulant) and heated for 2 hours at 70°C in a heating block, after which a quantity was transferred to a filter vial and added internal standard as described above.

Quantitation was performed using external calibration standards of 0, 5, 10, 25, 50, 75 ng/mL of fluorotelomer alcohols (FTOH) added several corresponding C13-labelled internal standards of 20 ng/mL. Samples for which PFAS concentrations exceeded the calibration curves were diluted prior to addition of internal standard and repeated analysis by LC-MS.

LC-MS/MS detection

The LC-MS/MS detection of the PFAS compounds was performed on a Dionex Ultimate 3000 /Bruker EVOQ Elite UPLC-MS/MS. The LC-MS/MS *m/z>m/z* transitions of PFSA and the general settings of the EVOQ Elite Bruker triple quadrupole with electrospray ionisation interface appears from Annex A, including also the analytical detection limit (LOD) of the individual PFAS.

LC-conditions for perfluorocarboxylic acids (PFCA)/perfluoro sulphonic acids (PFSA):

Phenomenex Luna Omega Polar C18 (100 Å, 1.6 µm, 100 x 2.1 mm) LC column in series with a Waters ACQUITY UPLC CSH C18 (130 Å, 1.7 µm, 100 x 2.1 mm) column; injection volume 5 µL, column oven temp.50°C, autosampler temp.10°C. Eluent flow was initially

0.15mL/min.; eluent A: 2 mM ammonium acetate in 90 % Milli-Q water/10 % methanol, pH adjusted to 9.0, and B: methanol. Eluent programme; 0 min. 10% B; 0.5 min. 10% B, 5.5 min 60% B, 12 min. 95% B at 0.2 mL/min., 16min. 95% B at 0.2 mL/min., 16.1 min 10% B at 0.15 mL/min to end 23 min.

LC-conditions for perfluoroalkyl phosphates (PAPs):

Waters ACQUITY UPLC CSH C18 (130 Å, 1.7 µm, 100 x 2.1 mm) column; injection volume 5 µL, column oven temp.50°C, autosampler temp.10°C. Eluent flow was initially 0.15mL/min.; eluent A: 2 mM ammonium acetate in 90 % Milli-Q water/10 % methanol, pH adjusted to 9.0, and B: methanol. Eluent programme; 0 min. 10% B; 0.5 min. 10% B, 5.5 min 60% B, 12 min. 95% B at 0.2 mL/min., 17.5min. 95% B at 0.2 mL/min., 17.6 min 10% B at 0.15 mL/min.to end 22 min.

LC-conditions for fluorotelomer alcohols (FTOH):

Waters ACQUITY UPLC CSH C18 (130 Å, 1.7 µm, 100 x 2.1 mm) column; injection volume 5 µL, column oven temp.30°C, autosampler temp.10°C. Eluent flow was initially 0.2mL/min.; eluent A: 2 mM ammonium hydroxide in Milli-Q water, pH adjusted to 8.5, and B: methanol. Eluent programme; 0 min. 70% B; 4 min. 99% B, 6.5 min 99% B, 8 min. 99% B at 0.3 mL/min., 11 min. 99% B at 0.3 mL/min, 12 min 70% B at 0.3 mL/min., 13 min 70% B at 0.3 mL/min., 13.5 min 70% B at 0.2 mL/min to end 17 min.

Quality assurance

The Danish accreditation body (DANAK) supervises the chemical methods applied at the DTU Food – National Food Institute. Routines are established for daily quality control of the methods taken into consideration a suitable composition of the analytical batch with respect to number of samples that are analysed in multiplicity, laboratory and solvent blanks and control charts.

3.2 Nontargeted screening

Samples from migration experiment were also used for nontargeted screening by an LC-Orbitrap system from Thermo Fisher Scientific (Vanquish Flex System, Orbitrap Exploris 120).

The sequence included blanks, injected at the beginning of the analysis and a QC sample analysed after the banks and at regular intervals between samples. The QC sample contained all the standards available (Annex B) at a concentration of 10 ng/mL. The compounds belong to different classes of fluorinated compounds, such as perfluoro carboxylic acids, perfluoro sulphonic acids, fluorotelomer compounds (sulfates, saturated and unsaturated carboxylic acids, alcohols), polyfluoroalkyl phosphate mono- and di- esters, per- and polyfluoroalkyl ether carboxylic acids, chloroperfluoroalkyl ether sulfonates, perfluoroalkyl phosphonic acids, perfluoroalkylphosphinic acids.

The analysis was performed using an Accurate™™ RP-MS LC column (150×2.1 mm, particle size 1.7µm) in the gradient of Aqueous 0.1% HCOOH (A) and MeOH (B) in the flow rate of 0.3mLmin^{-1}. The elution gradient started with 25% B increasing to 60% B at 1.4min, further increasing to 80% B at 8 min and to 90% B at 10min, kept in an isocratic mode until 12.75min,

then, increasing linearly to 100% B at 13 min, and hold for 5 min in an isocratic mode. Finally, at 18.3 min the method returned to the initial condition for 4min. The temperature of the column was 40°C and the temperature of the autosampler was 10°C. The injection volume was 5µL.

HESI settings in negative polarity were as follows: spray voltage −2500 V, ion transfer tube and the vaporizer temperatures 300◦C and 350◦C, respectively, the sheath gas and the auxiliary gas was nitrogen at a pressure of 50 and 10, sweep gas pressure 1. The spectrometric conditions were as follows: the full range used was between 100 and 1000 m/z with a resolution of 120000. ddMS2 spectra were acquired based on an inclusion list of compounds which included the targeted compounds listed in Table 1 with a stepped collision energy (HCD% 20, 70, 120).

The system was calibrated internally using EASY IC. For HRMS data acquisition XCaliburTM 4.7 software was utilized. The samples were analysed with target screening. Spectra were acquired for the standard compounds at a concentration of 25 ng/mL with the same analytical conditions of the FCM samples. Acquired data were processed using Compound Discoverer 3.3.2.31 (SP2) software (Thermo Scientific Fisher) and built in workflow to find and identify PFAS.

The identification of the standards was carried out based on a mass list that comprised all the compounds present in the analysed mixes (Table 1) and spectral features matches were searched in multiple databases including MZCloud, EPA Comptox PFAS Structure list (10,737 chemicals), EPA PFAS Master List (10 901 chemicals), NIST PFAS Suspect List (4951 chemicals), and Chemspider. For annotation of compounds, ddMS2 data were used to compare obtained spectra with fragment libraries such as Fluoromatch Suite Products (805 fragments) and PFAS Fine Signature Fragments (17 fragments). 37 out of 70 compounds were identified with the data treatment workflow and the results were used to create an in-house reference library in MZVault. Compound Discoverer was then used to process the results obtained from the analysis of the FCM samples. The alignment of the retention time was based on one of the QC injections from the central part of the sequence and one of the blank injections was used for background and noise removal. Compound detection was set at 5 ppm with a minimum of 1e5 intensity. The calculations of estimated C, mass/C, mass defect/C, and estimated F were performed by a scripting node developed by Thermo Fisher Scientific.

4 Results and discussion

In the present project, 30 FCM samples from five different regions in Norway were analysed for PFAS content, including PFCAs, PFSAs, monoPAPs, diPAPs, and FTOH- The analysed substances and their specific limits of detection (LOD) are listed in Annex A. Information regarding the type of samples (trade name, sampling date, country of origin etc.) are presented in table 2.

Table 2 Sampling information of paper & board FCM samples.

Region	Case number	Product type	Sampling place	DTU Number
Nord	2024/055647	Airfryer papir	Normal, Storgata, Tromsø	K24-423
Nord	2024/056263	Matposer papir	Normal, Jekta, Tromsø	K24-424
Nord	2024/057306	Muffins forme papir	Normal, AMFI Pyramiden, Tromsø	K24-425
Sør og Vest	2024/85370	Micropopcorn	Europris, Sandnes	K24-426
Sør og Vest	2024/85370	Muffins former papir	Europris, Sandnes	K24-427
Sør og Vest	2024/85370	Papirsugerør	Europris, Sandnes	K24-428
Sør og vest	2024/86196	Pizzaesker	Pizzabakeren Invest, Sandnes (ikke Tega Group)	K24-429
Sør og vest	2024/86196	Pizzaunderlagspapp	Pizzabakeren Invest, Sandnes (ikke Tega Group)	K24-430
Sør og vest	2024/86295	Muffinsformer	Home Brands AS Hovedkontor (Kitch'n/Tilbords)	K24-431
Sør og vest	2024/88810	Papirsugerør	Spar Kjøp, Kokstad, Bergen	K24-432
Sør og vest	2024/90204	Micropopcorn	Bunnpris Sartor, Straume	K24-433
stor Oslo	2024/87057	Papirsugerør	Festutstyr	k24-434
stor Oslo	2024/87088	Popcorn	COOP	k24-435
stor Oslo	2024/87088	Popcorn	COOP	k24-436
stor Oslo	2024/94004	pizzaeske	Eureca	K24-437
stor Oslo	2024/93980	sugrør	Culina	k24-438
stor Oslo	2024/94002	muffinsform papir	Cacas	k24-439
stor Oslo	2024/94002	muffinsform papp	Cacas	k24-440
stor Oslo	2024/96854	pizzaeske	Lyreco	k24-441
Midt	2024/092385	pizzaesker i papp	Sunnmøre Engros AS	k27-449
Midt	2024/092385	pizzaesker i papp	Sunnmøre Engros AS	k27-450
Midt	2024/092361	papptallerkener	Nor Engros Lerstad (Ødegaard engros)	k27-451
Midt	2024/092361	papirsugerør	Nor Engros Lerstad (Ødegaard engros)	k27-452
Øst	2024/95148	Micropopcorn	KIWI Landfalløya, 3023 Drammen	k24-442
Øst	2024/101843	pizzaesker i papp	NorEngros Drammen	K24-443
Øst	2024/101805	papirsugerør	Nille Gulskogen, Drammen	K24-444
Øst	2024/102124	muffinsformer	Dollarstore Buskerud Storsenter	K24-445
Øst	2024/102124	mikropopcorn	Dollarstore Buskerud Storsenter	K24-446
Øst	2024/102159	muffinsformer	Jula Mjøndalen	K24-447
Øst	2024/103029	Air Fryer papir	Jernia CC Drammen	K24-448

PFAS were detected in 11 out of the 30 analysed food contact material (FCM) samples: three muffin forms, three paper straws, three pizza boxes, one popcorn beaker, and one paper plate. The concentrations of individual PFAS compounds, expressed in µg/kg of food, are presented in Table 3. These results were calculated by first determining the concentrations in the extracts, then converting them to the amount extracted per dm² of FCM and finally estimating the concentration in µg/kg of food. This final conversion was based on the assumption that 1 kg of food is in contact with a surface area of 6 dm² (multiplication factor of 6).

The sum of concentrations of 11 PFCAs detected in 9 samples ranged from 0.004 – 0.905 µg/kg food. PFOS and PFHxS belonging to PFSA were detected only in two samples, however, only in the range of 0.007-0.011 µg/kg food. Among analysed MonoPAPs and DiPAPs, only 6:2DiPAP was one tested sample, namely cupboard pizza tray at concentration 0.05 µg/kg food. Among measured FTOH, only 6:2FTOH was detected in four samples at concentrations of 0.503-9.16 µg/kg food. One muffin form and one paper plate contained a total of 4 different substances comprising ∑PFAS concentrations of 5.16 and 1.42 µg/kg food respectively (sample K24-431 and K24-451). Moreover, the highest concentrations of ∑PFAS were found in extracts from one sample of paper plate and two muffin forms (∑PFAS: 9,161; 5,161; 3,983 µg/kg food (sample K24-44; K24-431; K24-439)).

Migration tests in triplicate were performed for all 11 samples in which PFAS were found at detectable levels. The migration conditions: 6 cm2 in 1.5 mL 50% ethanol/water 2 hours at 70°C were chosen, as the collected FCM are expected to be subjected to a high temperature during food preparation to mimic microwaving as well as high temperature conditions during baking.

The results of the individual PFAS and the ∑PFAS in µg migrant /kg food are presented in table 4 as migration in triplicate (1, 2, 3). The results of PFAS from the migration studies showed similar value compared to the PFAS results of the extracts in most of the samples. Interestingly, in paper plate samples a simultaneous decrease of 6:2FTOH and increase in PFPeA was observed. The reason for this could be oxidation of 6:2FTOH to corresponding PFCAs under migration conditions in high temperature. Similar observations were made by (Chen et al., 2024) who found a potential transformation relationship between the detected PFAS was proposed, starting from X:2 FTOH and finally transforming to CnF2n+1COOH.

In contrast to the previous investigation conducted in 2017 by (Granby & Håland, 2018), PFASs were detected at lower amounts. In that study, PFAS were found in 13 of 35 FCM analysed, that is in seven of 11 plates, two cupcake cups, one cup, a fish food bag, a popcorn beaker and a pizza tray. The sum of concentrations of 11 detected perfluorcarboxylic acids (PFCA) and polyfluorsulphonic acids (PFSA) in the extracts ranged from 0.01- 13.1 µg/kg food. In addition, fluorotelomer alcohols were detected in nine samples at concentrations 0.8-13.5 µg/kg. The highest concentrations of perfluoralkyl substances (∑PFAS) found were in three samples of plates, all from the same producer from USA and in two Chinese cupcake cups contained 12 different substances.

Detected amounts are also lower than those found in the recent study conducted by (Vázquez Loureiro et al., 2024) where PFAS content was investigated in FCM originating from Danish and Spanish markets. In this study, among analysed samples, the highest concentrations of PFAS were found in a muffin cup made of cellulose (PFCA ~ 1.41 µg/kg food, FTOH ~ 11.5 µg/kg food).

(Schwartz-Narbonne et al., 2023) studied occurrence of PFAS in Canadian food packaging. In this study FCM subjected to further investigation based on high total fluorine (TF) content. In the 8 samples with high total F revealed 4–15 individual PFAS in each sample, with 6:2 fluorotelomer methacrylate (FTMAc) and 6:2 fluorotelomer alcohol (FTOH) typically dominating.

(Di Mario et al., 2024) were studying occurrence of PFAS in one hundred and ten FCM made of paper and board, sugar cane paper analogues selected on the Belgian market. In this study among 25 PFAS targeted, 11 were detected in the samples, mainly perfluoroalkyl carboxylic acids (PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoDA, PFTrDA) and PFOS. It was found that all the paper analogue samples contained PFAS, while 43% of the paper and board samples showed the presence of these chemicals. Except for one sample, most detections suggest contamination rather than intentional use.

Table 3. Concentrations of ∑PFAS (PFCA and PFSA) in µg/kg food extracted from paper and board FCM. PFAS with TWI set by EFSA are marked red.

n.d. – not detected; <LOD – below limit of detection,

DTU Number	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDeA	PFUnA	PFDoDA	PFTrDA	PFTeDA	PFBS	PFHxS	PFOS	PFDS	4HPFOS	PFOSA	4:2 FTOH	6:2 FTOH	8:2 FTOH	10:2 FTOH	6:2 mono PAP	6:2 di PAP	8:2 mono PAP	8 2 di PAP
LOD	0,030	0,013	0,011	0,014	0,006	0,004	0,006	0,007	0,005	0,008	0,010	0,015	0,004	0,005	0,009	0,012	0,017	1,37	0,37	0,58	0,58	0,04	0,04	0,09	0,05
K24-423	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
K24-424	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
K24-425	n.d.	n.d.	n.d.	n.d.	0,006	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
K24-426	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
K24-427	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
K24-428	<LOD	n.d.	n.d.	<LOD	0,080	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD
K24-429	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	<LOD
K24-430	0,107	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.
K24-431	<LOD	0,015	0,018	0,015	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5,113	<LOD	<LOD	n.d.	n.d.	n.d.	n.d.
K24-432	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	<LOD	n.d.	n.d.	n.d.	n.d.
K24-433	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
k24-434	n.d.	n.d.	<LOD	n.d.	0,046	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
k24-435	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
k24-436	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
K24-437	n.d.	n.d.	n.d.	n.d.	n.d.	0,004	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,011	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	<LOD
k24-438	n.d.	n.d.	n.d.	n.d.	0,042	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.
k24-439	<LOD	n.d.	0,101	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	3,882	<LOD	<LOD	<LOD	n.d.	<LOD	n.d.
k24-440	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	<LOD
k24-441	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	0,05	n.d.	<LOD
k27-449	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	<LOD	<LOD	n.d.	<LOD
k27-450	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD
k27-451	0,894	n.d.	0,011	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,007	n.d.	n.d.	n.d.	n.d.	n.d.	0,503	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD
k27-452	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
k24-442	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
K24-443	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	<LOD
K24-444	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.
K24-445	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
K24-446	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	9,161	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
K24-447	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
K24-448	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD

Table 4. Concentrations of ∑PFAS (PFCA and PFSA) in µg/kg food extracted (0)/migrated (A,B,C) from paper and board FCM. PFAS with TWI set by EFSA are marked red.

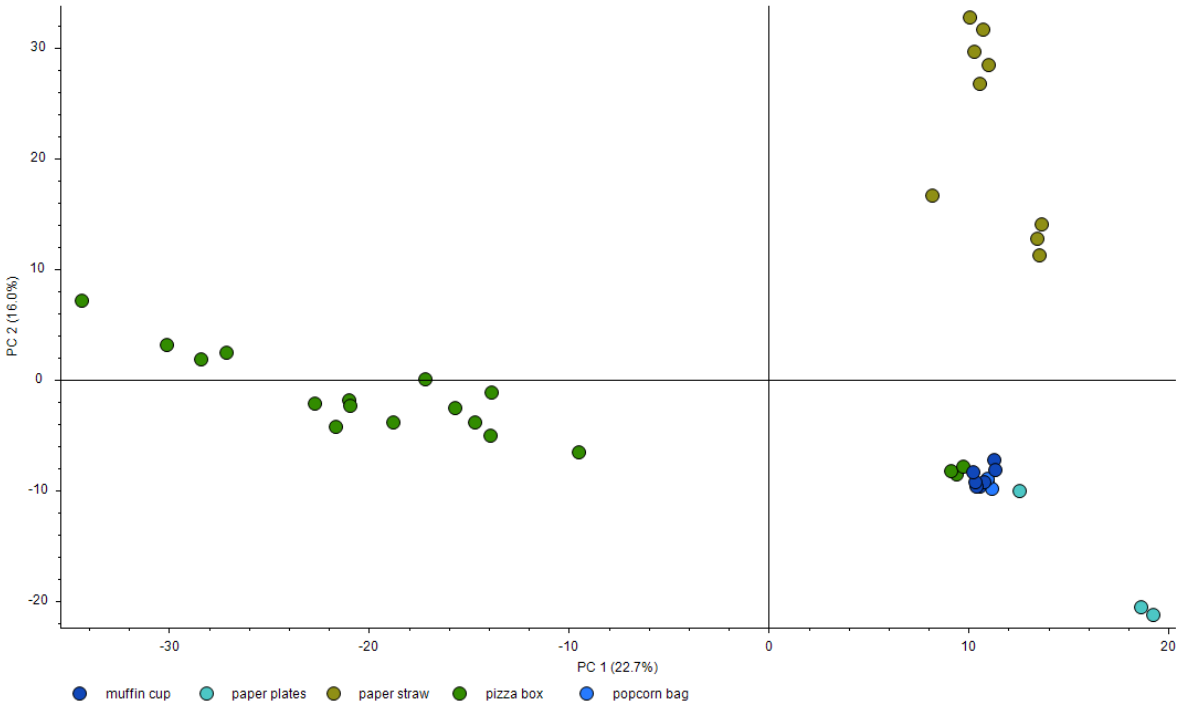
n.d. – not detected; <LOD – below limit of detection,

DTU number		PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDeA	PFUnA	PFDoDA	PFTrDA	PFTeDA	PFBS	PFHxS	PFOS	PFDS	4HPFOS	PFOSA	4:2 FTOH	6:2 FTOH	8:2 FTOH	10:2 FTOH	6:2 mono PAP	6:2 di PAP	8:2 mono PAP	8 2 di PAP
K24-425	0	n.d.	n.d.	n.d.	n.d.	0,006	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	A	<LOD	n.d.	<LOD	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,012	n.d.	n.d.	n.d.	n.d.								
	B	<LOD	n.d.	<LOD	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,004	n.d.	n.d.	n.d.	n.d.								
	C	0,000	n.d.	<LOD	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,012	n.d.	n.d.	n.d.	n.d.								
K24-428	0	<LOD	n.d.	n.d.	<LOD	0,080	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD
	A	<LOD	n.d.	<LOD	n.d.	0,072	n.d.	n.d.	n.d.	<LOD	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.								
	B	0,048	n.d.	<LOD	n.d.	0,069	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	<LOD								
	C	0,035	n.d.	<LOD	<LOD	0,067	n.d.	n.d.	n.d.	<LOD	n.d.	<LOD	n.d.	0,005	n.d.	n.d.	n.d.	n.d.								
K24-430	0	0,107	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.
	A	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.								
	B	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,013	n.d.	n.d.	n.d.	n.d.								
	C	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,008	n.d.	n.d.	n.d.	n.d.								
K24-431	0	<LOD	0,015	0,018	0,015	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5,113	<LOD	<LOD	n.d.	n.d.	n.d.	n.d.
	A	<LOD	0,014	0,152	0,015	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,012	n.d.	n.d.	n.d.	n.d.	n.d.	3,610	n.d.	n.d.				
	B	<LOD	0,013	0,126	0,013	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	2,529	n.d.	n.d.				
	C	<LOD	0,013	0,137	0,014	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,014	n.d.	n.d.	n.d.	n.d.	n.d.	2,773	<LOD	<LOD				
k24-434	0	n.d.	n.d.	<LOD	n.d.	0,046	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	A	<LOD	<LOD	n.d.	n.d.	0,035	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.								
	B	<LOD	<LOD	n.d.	n.d.	0,033	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.								
	C	<LOD	<LOD	<LOD	n.d.	0,033	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.								
K24-437	0	n.d.	n.d.	n.d.	n.d.	n.d.	0,004	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,011	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	<LOD
	A	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**n.d.**	n.d.	n.d.	n.d.	n.d.	**n.d.**	n.d.	n.d.								
	B	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**n.d.**	n.d.	n.d.	n.d.	n.d.	**n.d.**	n.d.	n.d.								
	C	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**n.d.**	n.d.	n.d.	n.d.	n.d.	**n.d.**	n.d.	n.d.								
k24-438	0	n.d.	n.d.	n.d.	n.d.	0,042	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.
	A	n.d.	n.d.	n.d.	n.d.	0,034	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,014	n.d.	n.d.	n.d.	n.d.								
	B	<LOD	n.d.	n.d.	n.d.	0,034	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.								
	C	<LOD	n.d.	n.d.	n.d.	0,037	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,006	n.d.	n.d.	n.d.	n.d.								
k24-439	0	<LOD	n.d.	0,101	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	3,882	<LOD	<LOD	<LOD	n.d.	<LOD	n.d.
	A	<LOD	0,014	0,091	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,008	n.d.	n.d.	n.d.	n.d.	<LOD	2,181	<LOD	<LOD				
	B	<LOD	<LOD	0,083	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	<LOD	2,445	<LOD	<LOD				
	C	<LOD	<LOD	0,085	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,000	n.d.	n.d.	n.d.	n.d.	<LOD	2,093	<LOD	<LOD				
k24-441	0	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	0,05	n.d.	<LOD
	A																						n.d.	<LOD	n.d.	<LOD
	B																						n.d.	<LOD	n.d.	<LOD
	C																						n.d.	<LOD	n.d.	<LOD
k27-451	0	0,894	n.d.	0,011	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,007	n.d.	n.d.	n.d.	n.d.	n.d.	0,503	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD
	A	0,504	0,031	0,013	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,003	n.d.	n.d.	n.d.	n.d.	n.d.	0,367	n.d.	n.d.				
	B	0,472	0,019	0,012	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,418	n.d.	n.d.				
	C	0,454	0,017	0,011	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,004	n.d.	n.d.	n.d.	n.d.	n.d.	0,351	n.d.	n.d.				
K24-446	0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	9,161	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	A																		n.d.	8,946	n.d.	n.d.				
	B																		n.d.	9,241	n.d.	n.d.				
	C																		n.d.	8,896	n.d.	n.d.				

All 11 samples obtained in migration study were further investigated via nontargeted screening approached by LC-HRMS to investigate presence of other PFAS not included in the scope of targeted method. Post-acquisition data handling of HRMS consists of data analysis and interpretation. The first step of data handling aims to transform raw data consisting of the histograms of detected ions in a small-time frame into a format allowing interpretation of the characteristics of detected ions. These characteristics are called features and include m/z, intensities and RT (Katajamaa & Orešič, 2007). A total of 5200 features were detected in the analysed samples, of which 1159 were suggested to contain three or more fluorine atoms with a Δm annotated (ppm) between -2.00 and 2.00. However, none of the target compounds included in the in-house library was identified in any of the FCM present in the study. PFAS quantified by LC-MS/MS were found at very low levels, below 0.5 ng/mL extract which is below LOD of current LCHRMS platform (LOD= 0.5 ng/mL corresponds to 0.082 ng/kg of food).

Looking at results from the LCHRMS analysis on the migration test solutions and compounds containing fluorine atoms it is possible to distinguish sample types by principal component analysis (PCA). The preliminary PCA plot is presented in Figure 2. To explain the observed phenomena, further investigation is necessary, focusing on the identification of features contributing to the observed pattern.

Figure 2. PCA of FCM samples divided by sample type based on the screened features containing fluorine atoms. Dataset is centred and normalized.



5 Conclusion

The sum of concentrations of 11 analysed perfluorcarboxylic acids (PFCA) and 4 polyfluorsulphonic acids (PFSA) in the extracts ranged from 0.006- 912 µg/kg food. Fluorotelomer alcohols were detected in four sample extracts at concentrations 0.503-9.161 µg/kg. MonoPAPs and diPAPS were found at quantifiable amount only in one pizza box (0.05 µg/kg). All eleven samples in which PFAS were found in quantifiable amounts were subjected to migration tested in triplicates. The highest migration of the sum of PFAS were found in popcorn beaker and muffin forms (ΣPFAS: 9.028; 3.145; 2.333 µg/kg food). In contrast to the previous investigation conducted in 2017, PFASs were detected at lower amounts. It was demonstrated a potential for migration of PFASs from FCM to foods, indicating that FCM could be a source of PFASs in food, but also rising awareness of such phenomena and limited use. In contrast to the previous investigation conducted in 2017, PFASs were detected at lower amounts.

Nontargeted screening reviled the presence of 5200 features were in the analysed samples, of which 1159 were suggested to contain three or more fluorine atoms with accuracy <2 ppm. However, none of the target compounds included in the in-house library was identified in any of the FCM present in the study.

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Annex A LC-MS/MS Conditions

Spay Voltage	**-3000V**
Cone temperature	**350°C**
Heated probe temp.	**350°C**
Cone gas flow	**20**
Heated probe gas flow	**50**
Nebulizer gas flow	**50**
Exhaust gas	**ON**

	Abbr.	**Limit of detection µg/dm²**	**Brutto formula (M: precursor)**	***m/z* $(M-H)^-$**	***m/z* product quantifier**	***m/z* product qualifier**	**t_R (min)**
PFBA	C4-PFCA	0.001	C4F7O2H	213.0	168.9	167.9	5.5
PFPeA	C5-PFCA	0.001	C5F9O2H	262.7	218.8	262.7	9.0
PFHxA	C6-PFCA	0.001	C6F11O2H	312.9	268.7	119.0	9.4
PFHpA	C7-PFCA	0.001	C7F13O2H	362.8	318.7	168.8	10.2
PFOA	C8-PFCA	0.001	C8F15O2H	412.7	368.6	168.8	10.8
PFNA	C9-PFCA	0.001	C9F17O2H	462.9	418.7	218.8	11.3
PFDA	C10-PFCA	0.001	C10F19O2H	512.9	468.6	218.8	11.6
PFUnDA	C11-PFCA	0.001	C11F21O2H	562.8	518.6	268.7	12.2
PFDoDA	C12-PFCA	0.001	C12F23O2H	612.8	568.6	168.9	12.5
PFTrDA	C13-PFCA	0.001	C13F25O2H	662.8	618.5	218.8	12.8
PFTeDA	C14-PFCA	0.001	C14F27O2H	712.8	668.4	318.6	13.2
PFBS	C4-PFSA	0.001	C4F9SO3H	298.8	80.1	99.0	8.8
PFHxS	C6-PFSA	0.002	C6F13SO3H	398.8	80.1	99.0	10.3
PFOS	C8-PFSA	0.001	C8F17SO3H	498.9	80.1	99.0	11.3
4H-PFOS	6:2 FTSA	0.001	C8F13H4SO3H	427.0	406.7	81.0	11.0
PFDS	C10-PFSA	0.002	C10F21SO3H	598.8	80.1	99.0	12.2
PFOSA	C8--	0.010	C8H2F17NO2 S	498.1	78.0	168.9	11.0
6:2 monoPAPs		0.001	C8H6F13O4P	442.9	442.7	97.1	8.9
8:2 monoPAPs		0.001	C10H6F17O4P	542.9	542.7	97.1	10.3
6:2/6:2 diPAPs		0.001	C16H8F26O4P	788.9	442.7	97.1	11.8
8:2/8:2 diPAPs		0.001	C20H8F34O4P	988.7	542.6	97.1	12.8
4:2 FTOH		0.3	C6H5F9O	263.0	155.0	203.0	3.1
6:2 FTOH		0.1	C8H5F13O	363.0	255.0	303.0	4.7
8:2 FTOH		0.1	C10H5F17O	463.0	403.0	355.0	5.7
10:2 FTOH		0.1	C12H5F21O	563.0	503.0	455.0	6.4

Annex B Target list of PFAS contained in the standard mixes for which MS2 spectra were acquired.

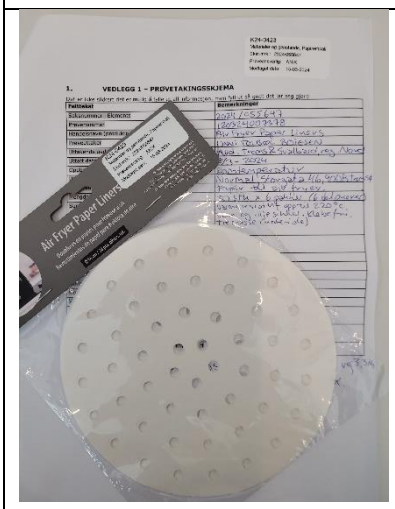
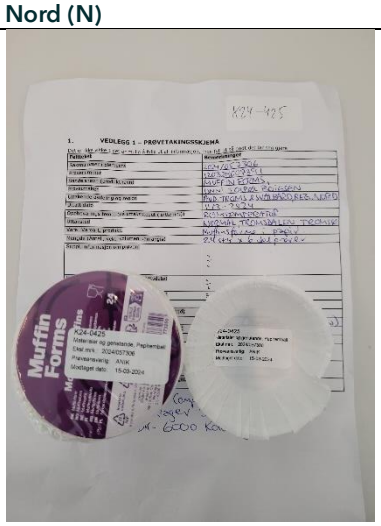
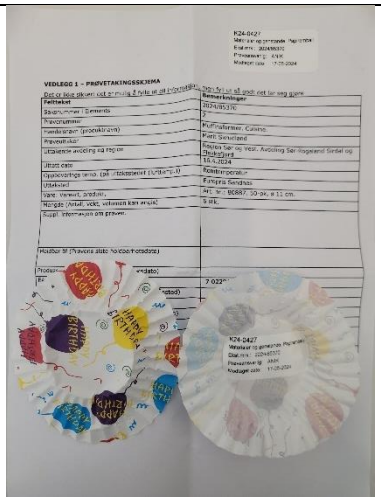
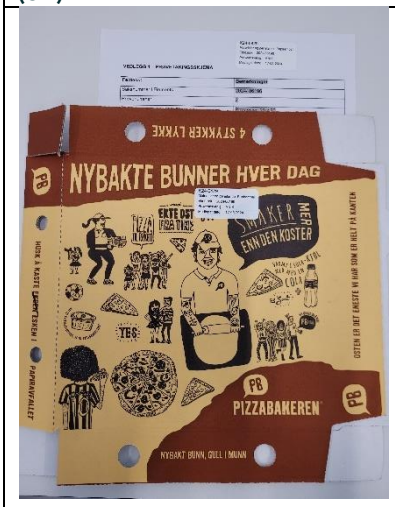
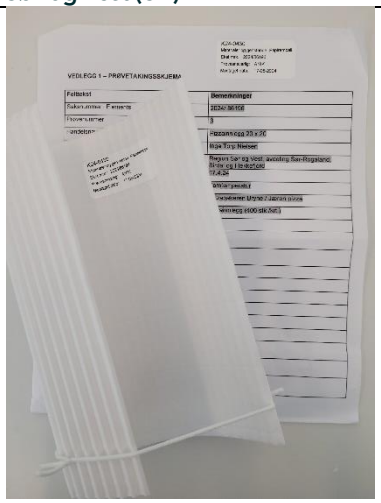
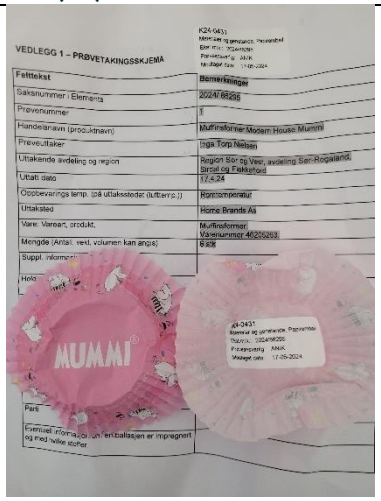
The compounds detected and identified with the data analysis workflow in Compound Discoverer are marked in the last column. The spectra acquired for these compounds were used to create the mzVault target library.

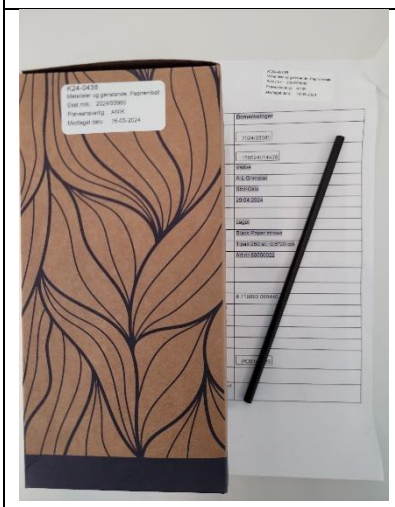
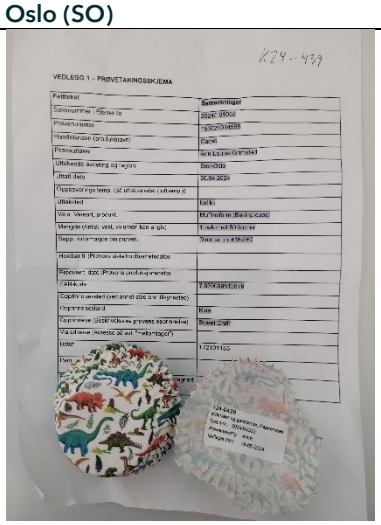
Compound name	Chemical Formula	m/z	Identified CD
PFBA_perfluoro-n-butanoic acid	C4HF7O2	213.0305	X
PFPeA_perfluoro-n-pentanoic acid	C5HF9O2	263.0380	X
PFBS_perfluoro-1-butanesulfonate	C4F9SO3	299.0917	X
PFHxA_perfluoro-n-hexanoic acid	C6HF11O2	313.0455	X
4:2FTS_1H,1H,2H,2H-perfluorohexanesulfonate (4:2)	C6H5F9SO3	327.9816	X
FPePA_3-perfluoropentyl propanoic acid (5:3)	C8H5F11O2	341.0987	X
FHUEA_2H-perfluoro-2-octenoic acid (6:2)	C8H2F12O2	357.0733	X
PFHpA_perfluoro-n-heptanoic acid	C7HF13O2	363.0530	X
DONA_dodecafluoro 3h 4,8dioxanonanoate	C7H2F12O4	377.9758	X
PFHxS_perfluoro-1-hexanesulfonate	C6F13SO3	399.9439	X
PFOA_perfluoro-n-octanoic acid	C8HF15O2	413.0605	X
Cl-PFHxPA_6-Chloroperfluorohexylphosphonic acid	C6H2ClF12PO3	415.4780	X
6:2FTS_sodium 1H,1H,2H,2H-perfluorooctanesulfonate (6:2)	C8H5F13SO3	427.9749	X
FHpPA_3-perfluoroheptyl propanoic acid (7:3)	C10H5F15O2	441.1137	X
FOUEA_2H-perfluoro-2-decenoic acid (8:2)	C10H2F16O2	457.0883	X
PFNA_perfluoro-n-nonanoic acid	C9HF17O2	463.0680	X
FOEA_2-perfluorooctyl ethanoic acid (8:2)	C10H3F17O2	477.0946	X
PFOS_ perfluoro-1-octanesulfonate	C8F17SO3	499.9375	X
N-MeFOSA_N-methylperfluoro-1-octanesulfonamide	C9H4F17NO2S	512.1636	X
PFDA_perfluoro-n-decanoic acid	C10HF19O2	513.0755	X
N-EtFOSA_N-ethylperfluoro-1-octanesulfonamide	C10H6F17NO2S	526.1901	X
8:2FTS_1H,1H,2H,2H-perfluorodecanesulfonate (8:2)	C10H5F17SO3	527.1749	X

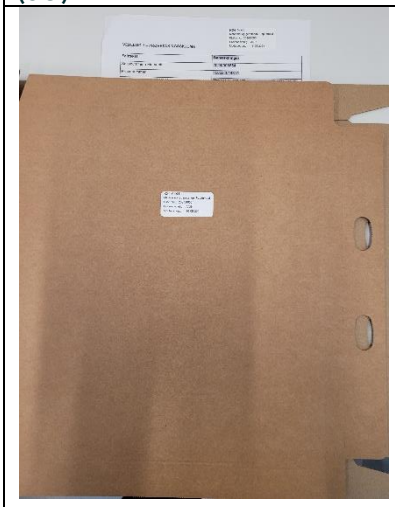
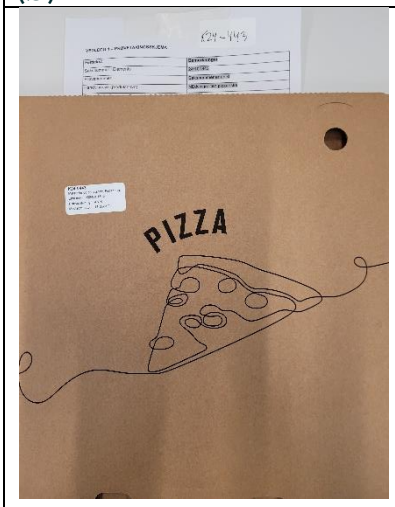
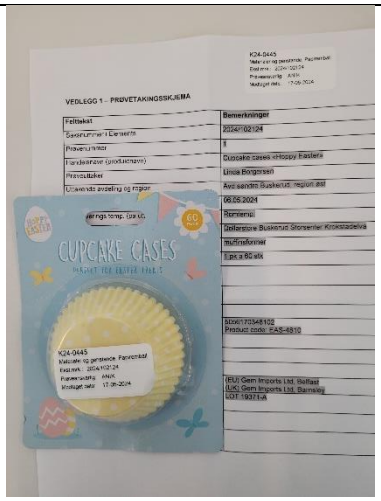
FDUEA_2H-perfluoro-2-dodecenoic acid (10:2)	C12H2F20O2	557.1033	X
PFUdA_perfluoro-n-undecanoic acid	C11HF21O2	563.0830	X
FDEA_2-perfluorodecyl ethanoic acid (10:2)	C12H3F21O2	577.1096	X
PFDS_ perfluoro-1-decanesulfonate	C10F21SO3	599.9311	X
PFDoA_perfluoro-n-dodecanoic acid	C12HF23O2	613.0905	X
10:2FTS_ 1H,1H,2H,2H-perfluorododecanesulfonate (10:2)	C12H5F21SO3	627.1899	X
PFTrDA_perfluoro-n-tridecanoic acid	C13HF25O2	663.0980	X
PFTeDA_perfluoro-n-tetradecanoic acid	C14HF27O2	713.1055	X
6:6PFPi_sodium bis(perfluorohexyl)phosphinate	C12HF26PO2	701.9299	X
6:2diPAP_ bis(1H,1H,2H,2H-perfluorooctyl) phosphate	C16H8F26PO4	789.9823	X
PFHxDA_perfluoro-n-hexadecanoic acid	C16HF31O2	813.1205	X
6:8PFPi_sodium perfluorohexylperfluorooctylphosphinate	C14HF34PO2	800.9162	X
8:8PFPi_bis(perfluorooctyl)phosphinate	C16HF34PO2	901.9171	X
PF4OPeA_Perfluoromethoxypropionic acid	C4HF7O3	229.0299	
Cl-PFOS_ Perfluoro-8-chloro-1-octanesulfonic acid	C8HClF16SO3	515.9079	
FPrPA_3-perfluoropropyl propanoic acid (3:3)	C6H5F7O2	241.0837	
FBET_2-perfluorobutyl ethanol (4:2)	C6H5F9O	263.0811	
L-PFPrS_sodium perfluoro-1-propanesulfonate	C3F7SO3Na	271.0661	
PF5OHxA_PFMBA	C5HF9O3	279.0374	
3,6-OPFHpA	C5HF9O4	295.0368	
5:2sFOTH_1-perfluoropentyl ethanol (5:2 secondary)	C7H5F11O	313.0886	
HFPO-DA_hexafluoropropylene oxide dimeric acid	C6HF11O3	329.0449	
FHET_2-perfluorohexyl ethanol (6:2)	C8H5F13O	363.0961	
PFPeS_ perfluoro-1-pentanesulfonate	C5F11SO3	371.0811	
FHEA_2-perfluorohexyl ethanoic acid (6:2)	C8H3F13O2	377.0796	
7:2sFTOH_1-perfluoroheptyl (7:2 secondary)	C9H5F15O	413.1036	
T-PFOA_T-PFOA	C8F15O2NH4	430.0911	
KPFHxS_potassium perfluoro-1-hexanesulfonate	C6F13SO3K	437.1971	
6:2PAP_ 1H,1H,2H,2H-perfluorodecyl phosphate	C8H4F13PO4	443.9796	
FOET_2-perfluorooctyl ethanol (8:2)	C10H5F17O	463.1111	

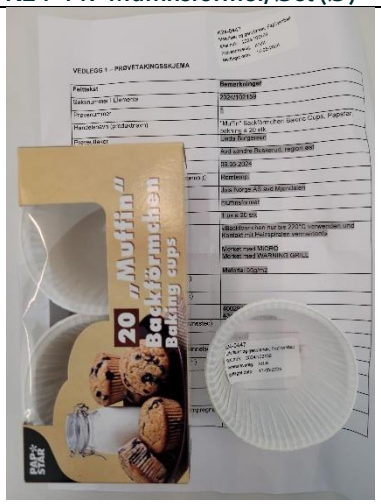
L-PFHpS_sodium perfluoro-1-heptanesulfonate	C7F15SO3Na	471.0961	
KPFECHS_KPFECHS	C8F15SO3K	499.2153	
8:2FTOAc_1H,1H,2H,2H-perfluorodecyl acetate	C12H7F17O2	505.0097	
8:2FTAcr_1H,1H,2H,2H-perfluorodecyl acrylate	C13H7F17O2	517.0097	
KPFOS_potassium perfluoro-1-octanesulfonate	C8F17SO3K	537.2121	
8:2PAP_ 1H,1H,2H,2H-perfluorooctyl phosphate	C10H4F17PO4	543.9732	
N-MeFOSE_2-(N-methylperfluoro-1-octanesulfonamido)ethanol	C11H8F17NO3S	556.2161	
FDET_2-perfluorodecyl ethanol (10:2)	C12H5F21O	563.1261	
N-EtFOSE_2-(N-ethylperfluoro-1-octanesulfonamido)ethanol	C12H10F17NO3S	570.2427	
L-PFNS_sodium perfluoro-1-nonanesulfonate	C9F19SO3Na	571.1111	
10:2FTOAc_1H,1H,2H,2H-perfluorododecyl acetate	C14H7F21O2	605.0033	
10:2FTAcr_1H,1H,2H,2H-perfluorododecyl acrylate	C15H7F21O2	617.0033	
L-PFUdS_sodium perfluoro-1-undecanesulfonate	C11F23SO3Na	671.1261	
L-PFDoS_sodium perfluoro-1-dodecanesulfonate	C12F25SO3Na	721.1336	
L-PFTrDS_sodium perfluoro-1-tridecanesulfonate	C13F27SO3Na	771.1411	
6:2/8:2diPAP_sodium (1H,1H,2H,2H-perfluorooctyl-1H,1H,2H,2H-perfluorodecyl) phosphate	C18H8F30PO4Na	911.1615	
PFODA_perfluoro-n-octadecanoic acid	C18HF35O2	913.1355	
8:2diPAP_ bis(1H,1H,2H,2H-perfluorodecyl) phosphate	C20H8F34PO4	989.9696	

Annex C Photos of the surveyed samples

K24-423 Airfryer papir, Nord (N)	K24-424 Matposer papir, Nord (N)	K24-425 Muffins forme papir, Nord (N)
		
K24-426 Micropopcorn, Sør og Vest (SV)	**K24-427 Muffins forme papir, Sør og Vest (SV)**	**K24-428 Papirsugerør, Sør og Vest (SV)**
		
K24-429 Pizzaesker, Sør og Vest (SV)	**K24-430 Pizzaunderlagspapp, Sør og Vest (SV)**	**K24-431 Muffinsformer, Sør og Vest (SV)**
		

K24-432 Papirsugerør, Sør og Vest (SV)	K24-433 Micropopcorn, Sør og Vest (SV)	K24-434 Papirsugerør, stor Oslo (SO)
		
K24-435 Popcorn, stor Oslo (SO)	**K24-436 Popcorn, stor Oslo (SO)**	**K24-437 Pizzaeske, stor Oslo (SO)**
		
K24-438 Sugrør, stor Oslo (SO)	**K24-439 Muffinsform papir, stor Oslo (SO)**	**K24-440 Muffinsform papir, stor Oslo (SO)**
		

K24-441 Pizzaeske, stor Oslo (SO)	K24-449 Pizzaeske, Midt (M)	K24-450 Pizzaeske, Midt (M)
		
K24-451 Papptallerkener, Midt (M)	**K24-452 Papirsugerør, Midt (M)**	**K24-442 Micropopcorn, Øst (Ø)**
		
K24-443 Pizzaesker i papp, Øst (Ø)	**K24-444 Papirsugerør, Øst (Ø)**	**K24-445 Muffinsformer, Øst (Ø)**
		

K24-446 Mikropopcorn, Øst (Ø)	K24-447 Muffinsformer, Øst (Ø)	K24-448 Air Fryer papir, Øst (Ø)
		

Sammen trygger vi framtiden for mennesker, dyr og natur

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