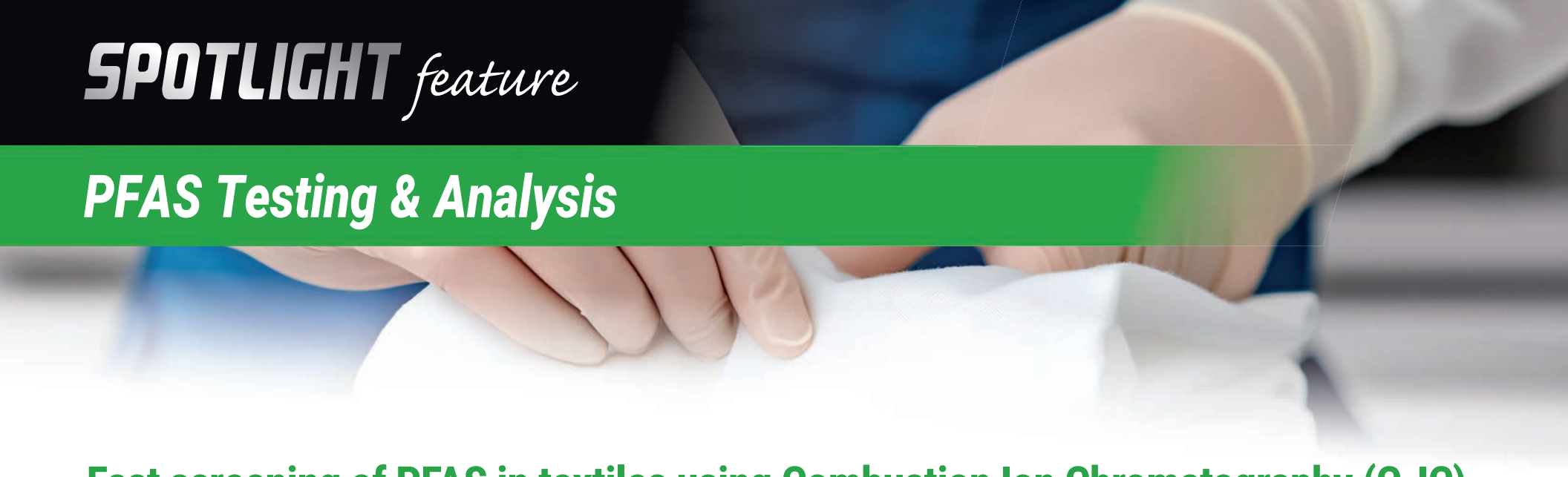
PFAS Testing & Analysis



Fast screening of PFAS in textiles using Combustion Ion Chromatography (C-IC)



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Per- and polyfluoroalkyl substances (PFAS) represent a class of synthetic fluorinated compounds characterised by the presence of at least one fully fluorinated methyl (CF3-) or methylene (-CF2-) carbon atom. This structure imparts PFAS with unique physicochemical properties, including exceptional hydrophobicity and thermal stability, and by June 2024, between 86,000 and 6,000,000 distinct PFAS compounds have been identified by PubChem database.

In the textile industry, PFAS (both polymeric and non-polymeric) plays a role in surface finishes and membrane technologies. Fluorocarbon-based finishes and ePTFE membranes are widely used to improve water, oil, and chemical repellency. These properties are essential for a diverse range of textile applications, including outdoor apparel, upholstery, and industrial fabrics.

PFAS are increasingly examined due to their persistence in the environment, potential to bioaccumulate, and links to adverse health effects. Their resistance to natural degradation has led to widespread environmental contamination, raising concerns about long-term exposure. With expanding regulatory measures, accurate detection and quantification of PFAS in textiles have become increasingly important for regulatory authorities, manufacturers, consumers, and testing laboratories.

Current regulations and limits for PFAS in textiles

Recently, increasing concerns about the potential health risks of these fluorinated compounds have led to new regulations in the United States and Europe aimed at restricting or banning the use of PFAS in textile products. *Table 1* provides an overview of these regulations, specifying limits on either individual PFAS compounds, Total Fluorine (TF), or Total Organic Fluorine (TOF).

Table 1: Regulatory limits on PFAS in textiles.

Regulation	Limit
REACH Annex XVII Entry 68 (02/2023)	Regulation on C9-C14 PFCAs, their salts, and related substances.
OEKO-TEX®100	100 mg/kg TF from 1st January 2024
California Assembly Bill 1817	TOF limits of 100mg/kg by 1st January 2025, and does not allow any exemption for articles exceeding these limits.
New York Conservation Law - ENV § 37-0121	Prohibition against the use of PFAS in apparel and outdoor apparel for severe wet conditions.

Challenges in detecting PFAS

Despite the regulatory efforts in targeting specific PFAS compounds, banning PFAS as a group remains a challenge due to their complex mixtures, including long- and short-chain variants and to evade these regulations, manufacturers continue to develop new PFAS formulations. Detecting PFAS in textiles is further complicated by their presence at trace levels, requiring highly sensitive analytical methods.

Currently, a widely used technique for the analysis (target and non-target) of PFAS is LC-MS/MS. Non-target PFAS analysis is more costly compared to target analysis and requires experienced technicians to perform the LC-MS/MS analysis. Besides, LC-MS/MS may not detect all the PFAS components, leading to an underestimation of total levels. As an alternative TOF and TF screening methods provide broader application but raise concerns about distinguishing PFAS from non-PFAS fluorinated substances used in textile processing.

TF and TOF values, such as those required by the OEKO-TEX® STANDARD 100 and the Californian AB-1817, can vary significantly due to inorganic fluorides in samples, which are common in laundry detergents, fabric dyes, and textile raw materials. Currently, there are no specifications on reference methods since method development is ongoing. This leads to inconsistencies and variance in results across laboratories, due to variations in sample preparation and analysis.

Therefore, TF screening accounting for both organic fluorine and inorganic fluoride represents the solution to provide a fast and cost-effective method for assessing the potential presence of PFAS in textiles, even if it may not directly indicate PFAS presence or concentrations. Unlike TOF, TF analyses do not require any sample preparation, simplifying the analyses and allowing indirect quantification of all PFAS present.

Combustion Ion Chromatography

Combustion Ion Chromatography (C-IC) offers a reliable method for TF analyses by addressing the challenges of a broad detection, with the identification of both known and unknown fluorinated compounds without needing individual standards and a matrix simplification. This allows manufacturers and regulators to efficiently assess PFAS content by faster and more efficient screening and to adapt processes to meet regulatory requirements. Currently, there is one standard under development for the textile sector: ISO/CD 20999:2023. This standard describes the analysis of speciated total halides (Fluorine, Chlorine, Bromine, and Iodine) in textiles by C-IC and it is applicable to all types of textile products.

Xprep C-IC for textile analysis

TE Instruments offers the Xprep C-IC (*Figure 3*), a cost-effective, highly reliable, and efficient solution for TF and TOF analysis in textiles, already compliant with the standard ISO/CD 20999:2023. Operating through oxidative pyrohydrolytic combustion, the system includes a fraction collection unit and a sample injection mechanism that is fully compatible with any renowned IC manufacturer.

Sample introduction

The Xprep C-IC is compatible with automated sample injection systems for liquids, gasses, and solids. For the measurement of textile samples, it operates with the Newton solid autosampler. With this autosampler the boat introduction device is used, the cups are placed automatically into the boat system which moves the cup into the furnace tube. With the Newton autosampler, up to 42 samples can be measured in one sequence.

Sample Combustion

For a standard textile analysis, the sample is loaded into the horizontal furnace through a boat inlet, following complete oxidation via pyrohydrolytic combustion at high temperatures, converting any fluorinated compounds into HF. To prevent corrosion caused by hydrofluoric acid on the furnace components, the system controls the introduction of both oxygen and ultra-pure water into the furnace tube during combustion. After the sample combustion, an absorber solution is added to the output gas stream. This process converts the HF into ionic fluoride (F^-) and ensures complete absorption of the analytes in the fraction collection unit. In textile analysis, the combustion tube and boat inlet include a ceramic setup instead of quartz, to mitigate corrosion risks.

Fraction collection

The combusted samples are collected in one of the sixty-five available positions of the fraction collection unit. The fraction collection unit consists of a dual-channel needle, the outer channel for transferring the absorber solution, containing the sample into a vial, and the inner channel for transferring the sample into the sample loop via a six-way valve, found at the front of the fraction collection unit. Once the sample enters the analytical IC column, the halogen ions are speciated and quantified. The storage of the sample in the fraction collection unit allows the Xprep C-IC to operate as a standalone system or to be fully integrated with an IC system, in this way the collected sample can be analysed either directly by the IC or at later stages and by alternative techniques.

Textile sample analysis

To evaluate system performance, four distinct textile samples were analysed, including personal protective equipment (PPE; samples 1-3) and commercial textiles (sample 4). The

system settings used for each analysis are presented in *Table 2*. The purpose of the study was to assess a wide range of TF content across different textile materials, specifically those known to contain PFAS due to their 'protective' properties, such as the PPE samples (samples 1-3), and compare these with commercial textiles that meet OEKO-TEX® STANDARD 100 (sample 4). For the sample analysis, a sample intake from 30 – 50 mg was selected, and 10 replicate C-IC analyses were performed on each sample.

Table 2: Setting for textiles analysis by Xprep C-IC.

Parameter	Setting
Oxygen Flow	300 mL/min
Argon Flow	100 mL/min
Oxygen Collision Flow	100 mL/min
Furnace Temperature I	1000 °C
Furnace Temperature II	1000 °C
UPW Initial Absorber Volume	3000 µL
Dosing Speed Ultra-Pure Water (UPW)	2 µL/s
Flush Volume Furnace Tube UPW	250 µL
Wash Volume Absorber Solution	2000 µL
Sample Loop IC	100 µL

Calibration

The system was calibrated using inorganic fluoride standards in the range of 0 to 10 mg/kg Fluoride in water (*Table 3*). three replicates per level were injected directly into the IC without combustion. It should be noted that the calibration standards are not combusted and therefore do not undergo a dilution step, compared to the sample analysis. For the sample analysis, 30 mg of combusted sample is collected in 8526 ml of UPW, resulting in a dilution factor of 284.2.

The linear trendline results below show the excellent performance of the Xprep C-IC, producing a calibration curve with an R^2 of 0.99993 and a slope of 0,99996 (*Figure 1*).

Table 3: overview of the calibration standards used to calibrate the IC.

Standard	F- concentration (IC Direct) (mg/kg)
Blanc	0
1	0.005
2	0.01
3	0.05
4	0.1
5	0.5
6	1
7	2.5
8	5
9	10

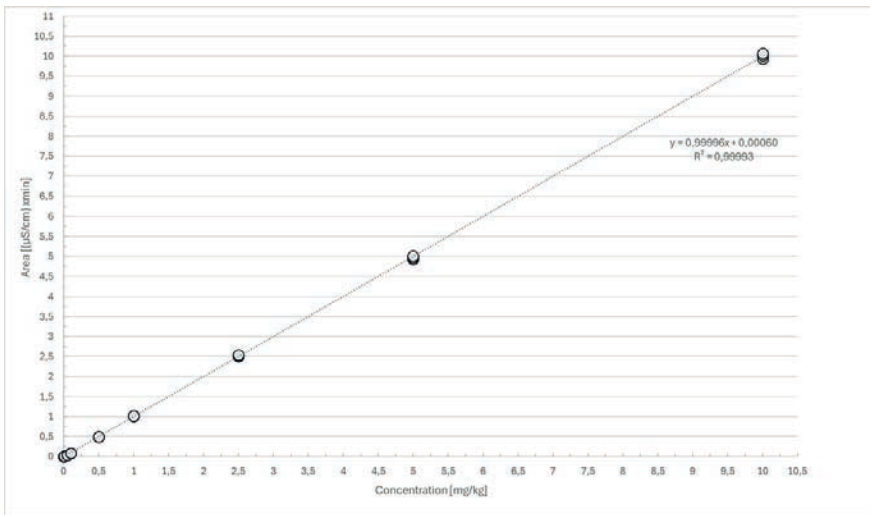


Figure 1: Calibration line by IC direct measurements for TF analyses.

Sample Calculation & Results

After sample analyses, the results need to be corrected by applying the dilution factors. The dilution factors are variables per sample, based on the amount of volume used in the fraction collection unit. The calculated relative standard deviation (RSD %) is based on the final sample concentrations.

The TF content varied across the different samples, with average results presented in mg/kg, as shown in *Table 4*. As expected, the PPE samples (samples 1-3) showed relatively high TF content ranging from ~ 500 mg/kg to ~0.2%. All the samples show good repeatability with an RSD <5%, except for sample 3, with a RSD of 18%. It is important to highlight that the homogeneity of applied coatings on textiles, as well as dyes, can have a significant impact on analysis repeatability, the variability reported for sample 3 may be attributed to this issue. Commercial sample 4 is within the maximum TF limit of 100 mg/kg, as specified by OEKO-TEX 100 regulations. *Figure 4* shows a chromatographic overlay of sample 2.

Table 4: Results of TF analysis in textile samples.

Sample	Sample type	Intake (mg)	TF (mg/kg) n=10	Standard deviation	RSD (%) n=10
Sample 1	PPE-Blue fabric	50	2354	112.9	4.8
Sample 2	PPE- Polyurethane coated polyester orange fabric	30	998	20.4	2.0
Sample 3	PPE-Polyester and cotton orange fabric	50	478	86.2	18.0
Sample 4	Commercial black fabric	50	48	2.1	4.3

Summary

The Xprep C-IC system provides an efficient and reliable method for analysing Total Fluorine in textiles, offering a fast screening solution for PFAS detection. The measurement of TF by C-IC can be used as a powerful screening tool prior to in-depth component-specific LC-MS/MS analysis. Using oxidative pyrohydrolytic combustion, the system eliminates the need for sample pretreatment, allowing direct analysis of textile samples. The combustion and fraction collection process converts fluorinated compounds into ionic fluoride, which is quantified through Ion Chromatography (IC). The Xprep C-IC is ideal for assessing a wide range of TF content in textiles, from personal protective equipment (PPE) to commercial fabrics, with strong repeatability (RSD <5%). The Xprep C-IC is already compliant with the ISO/CD 20999:2023 and this method meets regulatory standards such as OEKO-TEX® 100 and California AB 1817, providing an accurate, cost-effective tool for monitoring PFAS in textile products.



Figure 2 (left): Example of textile sample analysed. Sample 1, PPE-Blue fabric.



Figure 3 (above): Xprep C-IC with Newton solids autosampler.

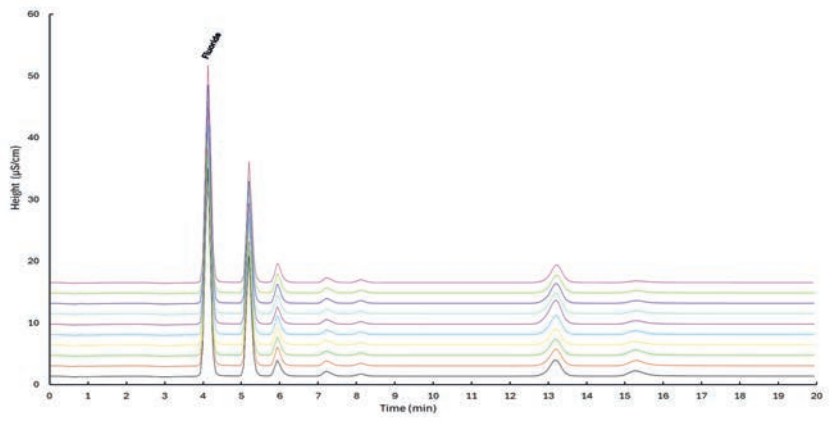


Figure 4: Chromatogram from 10 repetition analyses of sample 2.