Application note | 004403

Combustion-ion chromatography

# Screening of per- and polyfluoroalkyl substances (PFAS) in cosmetics: Utilizing a new combustion-ion chromatography system for total organic fluorine (TOF) analysis

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## Introduction

Per- and polyfluoroalkyl substances (PFAS) have been widely incorporated into cosmetic formulations due to their unique physicochemical properties. Their ability to impart smooth texture, long wear, water resistance, and improved spreadability has made PFAS appealing ingredients in products such as foundations, mascaras, lotions, and lipsticks. However, these same properties—high chemical stability, hydrophobicity, and resistance to degradation—raise concerns regarding human exposure and ecological impact.

In response, several US states have enacted restrictions on intentionally added PFAS in cosmetics.[1] A key example is California's PFAS-Free Cosmetic Act (AB 2771), which prohibits the sale of cosmetics containing intentionally added PFAS beginning January 1, 2025.[2] The law adopts a broad definition of PFAS as "fluorinated organic chemicals containing at least one fully fluorinated carbon atom," reflecting the regulatory challenge of addressing both well-characterized PFAS and lesser-known or emerging fluorinated compounds.[3]

Traditional PFAS testing in cosmetics has focused on targeted liquid chromatography–mass spectrometry (LC-MS) methods, which provide high sensitivity for specific, well-defined PFAS analytes.[4] However, these methods cannot detect PFAS species lacking analytical standards, including novel PFAS, polymeric materials, and precursor compounds. In addition, targeted LC-MS methods are often time- and resource-intensive, making them less practical for high-throughput screening of large numbers of samples. As a result, there is growing interest in complementary screening approaches capable of capturing total fluorine content from all organically bound fluorine sources.

Combustion-ion chromatography (C-IC) provides such an approach by converting all fluorine into measurable fluoride, allowing laboratories to quantify total organic fluorine (TOF) as a broad indicator of PFAS presence. Although TOF is not explicitly referenced in AB 2771, it serves as a valuable screening tool for identifying samples that may contain intentionally added PFAS and may require follow-up targeted analysis.

To address these analytical needs, the Thermo Scientific™ Cindion™ Combustion Ion Chromatography System integrates the Thermo Scientific™ Dionex™ Inuvion™ ion chromatography system with the Thermo Scientific™ Cindion™ Combustion/Absorption Module, creating an efficient platform for TOF-based PFAS screening. The system features a Z-fold combustion tube design that enhances combustion efficiency and reduces overall footprint. Unified operation through the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) streamlines instrument control, data acquisition, and reporting, making the workflow robust and user-friendly for routine PFAS screening in cosmetic matrices.

## Experimental

### Equipment

- Dionex Inuvion IC system with Reagent-Free™ Ion Chromatography (RFIC)
- Thermo Scientific™ Dionex™ AS-AP Autosampler
- Cindion combustion/absorption module

## Software

- Chromeleon CDS, software version 7.3.2

## Consumables

- Thermo Scientific™ Dionex™ IonPac™ AG24 Guard Column, 2 x 50 mm (Part No. 064151)
- Thermo Scientific™ Dionex™ IonPac™ AS24 Analytical Column, 2 x 250 mm (Part No. 064153)
- Thermo Scientific™ Dionex™ EGC 500 KOH Eluent Generator Cartridge (Part No. 075778)
- Thermo Scientific™ Dionex™ CR-ATC 600 Continuously Regenerated Anion Trap Column (Part No. 088662)
- Thermo Scientific™ Dionex™ ADRS 600 Anion Dynamically Regenerated Suppressor, 2 mm (Part No. 088667)
- Fisherbrand™ Narrow-Mouth Field Sample Bottles, high-density polyethylene (HDPE), 125 mL, 250 mL sizes for storage of standards and samples (Fisher Scientific Part Nos. 02-895A, 02-895B)
- Thermo Scientific™ polyethersulfone (PES) filter, 0.2 µm pore size (Part No. 09-740-113)
- Fisherbrand™ Premium Microcentrifuge Tube, polypropylene, 2 mL (Fisher Scientific Part No. 05-408-138)

## Reagents and standards

- Deionized (DI) water, Type 1 reagent grade, 18 MΩ·cm resistivity or better
- Thermo Scientific™ fluoride standard, 1000 mg/L, (Fisher Scientific Part No. NC1145532)
- Thermo Scientific™ Dionex™ Combined Seven Anion Standard II (fluoride 20 mg/L, bromide 100 mg/L, chloride 100 mg/L, nitrite 100 mg/L, nitrate 100 mg/L, sulfate 100 mg/L, phosphate 200 mg/L) (Fisher Scientific Part No. NC1145568)
- Perfluorooctanesulfonic acid (Fisher Scientific Part No. NC2948459)
- Thermo Scientific™ Methanol, UHPLC-MS grade (Part No. A4581)

## Preparation of solutions and reagents

### Fluoride calibration standard

A seven-point fluoride calibration curve (1–100 mg/L) was prepared by diluting a 1000 mg/L fluoride standard solution with DI water.

### Perfluorooctanesulfonic acid (PFOS) standard

A 0.35 mg/mL PFOS solution in methanol was prepared for combustion efficiency verification and sample spike-recovery experiments.

## Samples

Ten commercially available cosmetic products (six lipsticks, two mascaras, and two foundations) were purchased from an online retailer and analyzed.

## Sample preparation

### Total fluorine (TF) by C-IC

Between 5 and 50 mg of cosmetic sample was weighed into a pre-baked ceramic combustion cup, prepared by blank combustion under the same C-IC conditions, and analyzed directly by C-IC.

### Total inorganic fluorine (TIF) by direct injection

For TIF extraction:

1. Weigh 25–100 mg sample into a 2 mL centrifuge tube.
2. Add 1.5 mL DI water, sonicate for 30 min, then centrifuge at 15,000 × g for 10 min.
3. Filter the supernatant through a 0.2 µm PES filter.
4. Analyze the filtrate by direct injection on the ion chromatography (IC) system.

## TOF by C-IC

TOF was determined using the C-IC system consisting of a Dionex Inuvion IC system with a Dionex AS-AP autosampler and a Cindion C-IC system combustion/absorption module. Detailed instructions for configuring the 2-in-1 system are provided in Technical Note 003853.[5]

Two modes of sample introductions were used:

### Combustion mode (Figure 1)

Samples were placed in ceramic cups and combusted at 1100 °C under argon (100 mL/min) and oxygen (400 mL/min). Acidic combustion products were absorbed into 7 mL DI water, and 25 µL of the absorbate was injected for IC analysis. In this mode, all fluorine derived from organic and inorganic compounds in solid or liquid samples is converted to and determined as fluoride by IC, providing the measure of TF.

### Direct injection mode

Aqueous extracts were introduced directly via the Dionex AS-AP autosampler to quantify TIF.

Fluoride was measured using a Dionex Inuvion IC system equipped with a 25 µL sample loop, a Dionex ADRS 600 suppressor (or the Thermo Scientific™ Dionex™ NGES-A Next Generation Electrolytic Suppressor, the recommended equivalent replacement for anion analysis), and a Dionex EGC 500 KOH cartridge. Fluoride was separated from other anions using a Dionex IonPac AS24 analytical column and guard column maintained at 30 °C. The detailed C-IC conditions are listed in Table 1.

**Table 1. C-IC conditions.**

| IC system conditions | |
|---|---|
| IC system | Dionex Inuvion IC system with Dionex AS-AP autosampler |
| Columns | Dionex IonPac AS24 analytical column (2 x 250 mm) (Part No. 064153)<br>Dionex IonPac AG24 guard column (2 x 50 mm) (Part No. 064151) |
| Eluent | 8 mM KOH from 0–6 min, 8–75 mM KOH from 6–9 min, 75 mM KOH from 9–12 min, 8 mM KOH from 12–20 min |
| Eluent source | Dionex EGC 500 KOH cartridge (Part No. 075778) with Dionex CR-ATC 600 continuously regenerated anion trap column (Part No. 088662) |
| Flow rate | 0.3 mL/min |
| Injection volume | 25 µL |
| Column temperature | 30 °C |
| Detection | Suppressed conductivity, Dionex ADRS 600 anion dynamically regenerated suppressor, 2 mm (Part No. 088667), recycle mode, 56 mA current |
| Run time | 20 min |

| Combustion and absorption conditions | | | |
|---|---|---|---|
| Combustion system | Cindion C-IC system combustion/absorption module | | |
| Furnace temperature | Heater 1: 1050 °C<br>Heater 2: 1050 °C | | |
| Gases | Oxygen (primary): 300 mL/min<br>Oxygen (turbo): 100 mL/min<br>Argon carrier: 100 mL/min | | |
| Absorption solution | 7 mL DI water | | |
| Boat program | Position (mm) | Wait time (s) | Boat speed (mm/s) |
| | 65 | 180 | 2 |
| | 90 | 180 | 2 |
| | 150 | 180 | 1 |

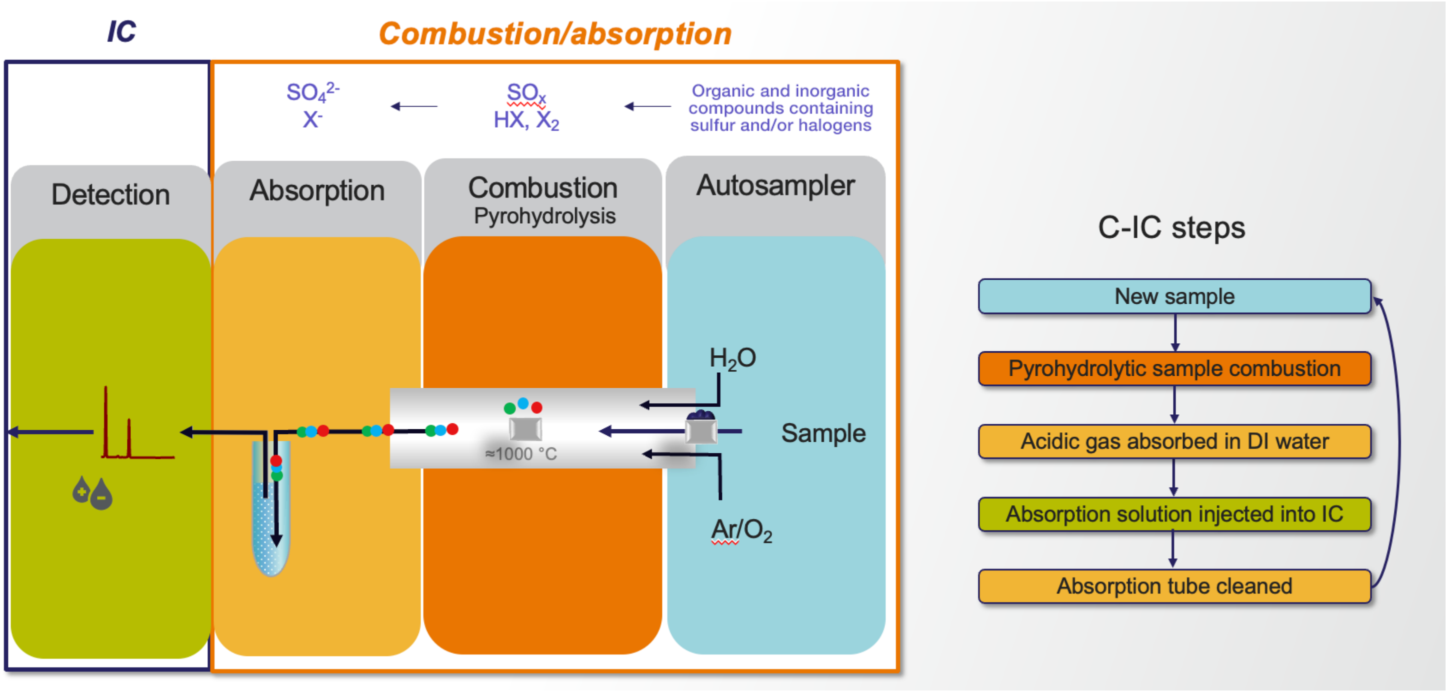

**Figure 1. C-IC instrument workflow.**

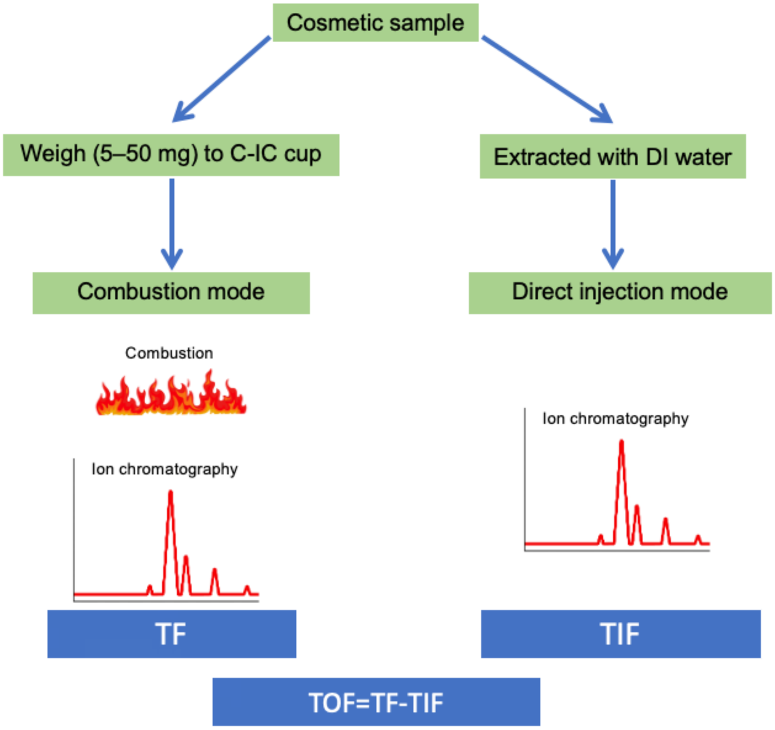

**Figure 2. TOF analysis workflow.**

Figure 2 outlines the overall workflow for determining TF, TIF, and TOF using C-IC. TOF was calculated as TOF=TF-TIF. For TF analysis, the sample amount needed may vary based on the concentration of PFAS in the samples. For the analysis described here, 5–50 mg of cosmetic sample was placed onto a pre-baked ceramic boat for analysis by C-IC operated in combustion mode. To avoid carryover, two to three boat blanks were analyzed after samples expected to contain a high amount of fluorine. To determine TIF, the water extract was analyzed by C-IC operated in direct injection mode. TOF was calculated by subtracting TIF from TF.

## Results and discussion

### Separation

Establishing the appropriate eluent was essential for determining the optimal separation of fluoride from the water dip and other common anions. Carbonate and hydroxide are commonly used as eluents in IC. Hydroxide eluent was chosen due to its effectiveness in separating fluoride from the water dip and its ability to yield a higher signal-to-noise (S/N) ratio after suppression compared to carbonate eluent. The use of hydroxide eluent resulted in increased overall method sensitivity.

Figure 3 illustrates the separation of seven common anions and three organic acids using combustion and direct injection modes. Only fluoride, chloride, and bromide were detected in the chromatogram obtained from the combustion mode, and fluoride was well-separated from the water dip and other anions, enabling accurate quantification.

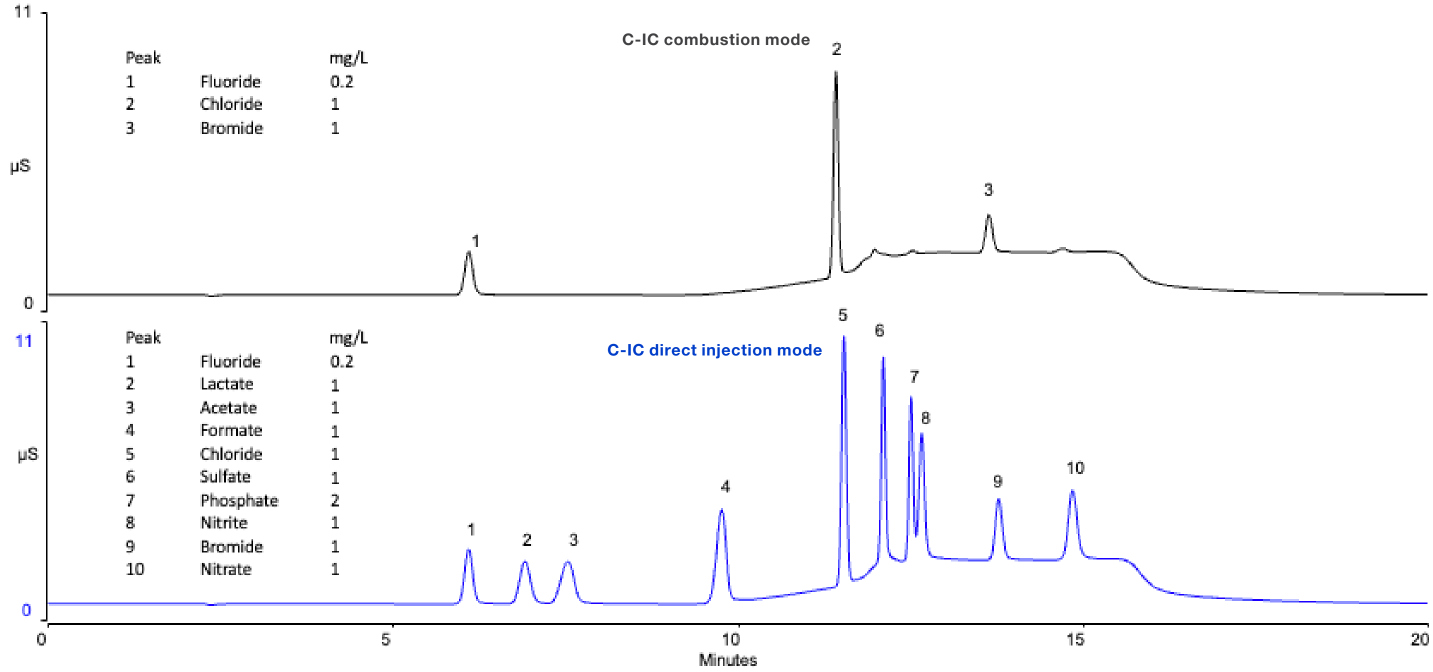

**Figure 3. Separation of anions standard (combustion mode vs. direct injection mode).**

### Calibration

Combustion mode was used to build fluoride calibration curves, in alignment with EPA Method 1621 for the determination of adsorbable organic fluorine (AOF) in aqueous matrices by C-IC.[6] A seven-point calibration curve was prepared over a concentration range of 1–100 mg/L by diluting a certified 1000 mg/L fluoride standard solution with DI water. Standards were pipetted (200 µL) into ceramic boats, combusted, and quantified as fluoride (expressed in ng) as shown in Figure 4. The regression coefficients of the calibration curve were greater than 0.999 with a quadratic fitting. The calculated concentrations of the calibration standards were within 95–110% of the true value for all calibration levels, meeting the EPA requirements of 80–120%. The calibration was also assessed based on the relative standard error (RSE) method as listed in EPA Method 1621, and the RSE for the calibration curve was <10%, meeting the EPA requirement of 20% or less.

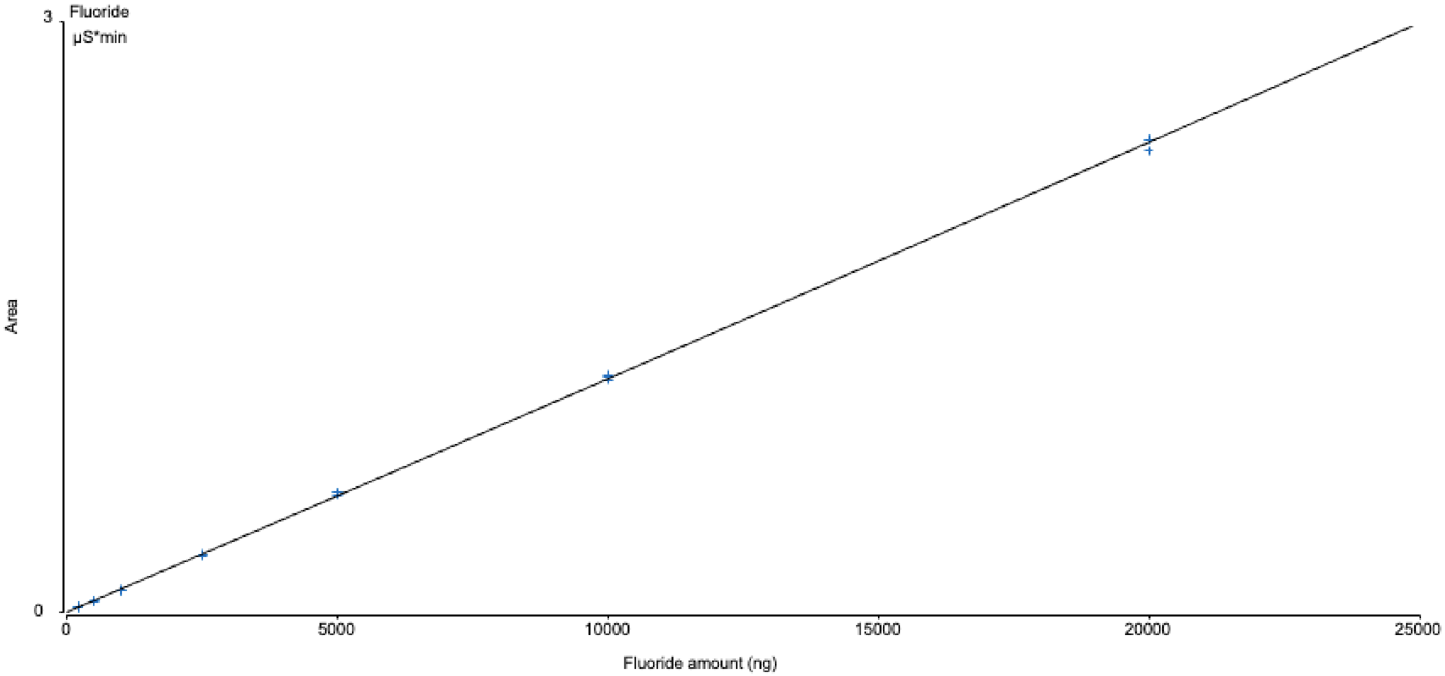

**Figure 4. Fluoride calibration curve.**

### Method detection limit (MDL)

The method detection limit (MDL), which depends on blank values and standard deviations of blank measurements, was calculated according to the guideline in the Definition and Procedure for the Determination of the Method Detection Limit, Revision 2 EPA document.[7] We report $MDL_b$ if the blank for an individual analyte gives a numerical result. The $MDL_b$ is calculated as:

$MDL_b = X + (3.143*\sigma)$

where X is the average of the method blanks, 3.143 is the student's t-test value for n=7, and σ is the standard deviation of the replicate method blanks. $MDL_s$ is reported if the blank does not give a numerical value and a spiked MDL standard was prepared, calculated as:

$MDL_s = 3.14*\sigma$

where 3.143 is student's t-test value for n=7, and σ is the standard deviation.

For TF, seven boats without samples were analyzed, yielding an average blank value of 22 ng and a standard deviation of 8.7 ng. This translates to an $MDL_b$ of 0.9 µg/g, assuming a sample weight of 50 mg.

For TIF, external water blank injection did not yield a value, so a 2 µg/L fluoride MDL standard was used and analyzed in seven replicates. The $MDL_s$ were calculated as 37.8 ng/g, assuming a sample weight of 25 mg.

### Method accuracy and precision

Accuracy was assessed using two approaches. First, combustion of the PFOS standard was conducted to verify the combustion efficiency of the C-IC system. Second, samples were spiked with PFOS standards and subjected to the complete workflow to assess method accuracy. Sample and spiked samples were analyzed in triplicate to evaluate method precision. To monitor background contamination, extraction blanks were processed in every batch. Results were blank corrected by subtracting the corresponding average blank values.

A standard solution of PFOS (0.35 mg/mL in methanol) was prepared for direct combustion analysis. Of this solution, 75 µL was added to the C-IC sample cup, and the fluoride value was determined using the previously established calibration curve. A recovery of 98.5% was achieved, confirming that the combustion conditions effectively convert fluorine to fluoride.

Method accuracy was evaluated further through recovery studies using spiked samples. To assess TF recovery in samples, 50 µL of a 0.35 mg/L PFOS standard solution was added to approximately 5–50 mg of sample, resulting in a spike of approximately 11,302 ng of fluorine in the samples. The samples were incubated at room temperature overnight to ensure absorption of PFAS into the cosmetic matrix. The TF in the samples and spiked samples was determined using the C-IC combustion mode. Recovery was assessed by first comparing the TF difference between the sample and the spiked sample. The TF difference was then divided by the known amount of PFOS standard added to the spiked sample to calculate the final recovery. This comparison helps determine the efficiency and accuracy of the method in recovering the added analyte from the sample matrix. Total recoveries ranging from 98–110% were obtained for all 10 samples, confirming the efficiency and completeness of the combustion as well as the accuracy of the overall workflow.

To assess TIF recovery in samples, 100 µg/L fluoride was spiked into the sample water extracts. As above, recovery was calculated by comparing the TIF differences between the sample and spiked samples with the known amount of fluoride that was added. Recoveries ranging from 95–115% were obtained for all 10 samples, confirming the accuracy of IC for TIF determination. The average relative standard deviation between the triplicates was less than 5%, indicating high precision.

### TOF in cosmetic samples by C-IC

The TF in a sample is defined as the sum of TIF and TOF. In this study, TF was measured using the combustion method, while TIF was determined by directly injecting water-extracted samples into the IC using the Dionex AS-AP autosampler.

As summarized in Table 2, five of the ten cosmetic products (three lipsticks and two mascaras) contained TF >100 ppm. In most samples, the TIF contribution was minimal (<1 ppm), except for one lipstick sample (Sample 4) and the two mascara samples (Samples 7 and 8), where TIF exceeded 8 ppm. Even after subtracting TIF, the TOF values for these five samples remained above 100 ppm, indicating a substantial fraction of fluorine is organically bound and likely consistent with the presence of PFAS.

Representative TF and TIF chromatograms (Sample 4) are shown in Figure 5, illustrating that fluoride is well separated from other anions present in the matrix, enabling reliable quantification.

California AB 2771 prohibits the manufacture and sale of cosmetic products containing intentionally added PFAS but does not specify a numerical TOF threshold.[2] Under this framework, TOF serves as an effective screening metric to identify samples that may contain intentionally added PFAS and warrant follow-up targeted PFAS analysis by LC-MS.

**Table 2. TOF in cosmetics, ppm (µg/g), (n=3, RSD <10% for TF >100 ppm samples, RSD <20% for TF <100 ppm samples).**

| Sample | Sample info | TF | TIF | TOF (TF-TIF) |
|---|---|---|---|---|
| 1 | Lipstick | 48.2 | 0.73 | 47.5 |
| 2 | Lipstick | 712 | 0.78 | 711 |
| 3 | Lipstick | 11.4 | 0.14 | 11.3 |
| 4 | Lipstick | 906 | 13.1 | 893 |
| 5 | Lipstick | 194 | 0.30 | 194 |
| 6 | Lipstick | 4.9 | 0.32 | 4.6 |
| 7 | Mascara | 1974 | 9.44 | 1965 |
| 8 | Mascara | 2098 | 8.50 | 2089 |
| 9 | Foundation | 85.0 | 0.35 | 84.7 |
| 10 | Foundation | 37.1 | 0.59 | 36.5 |

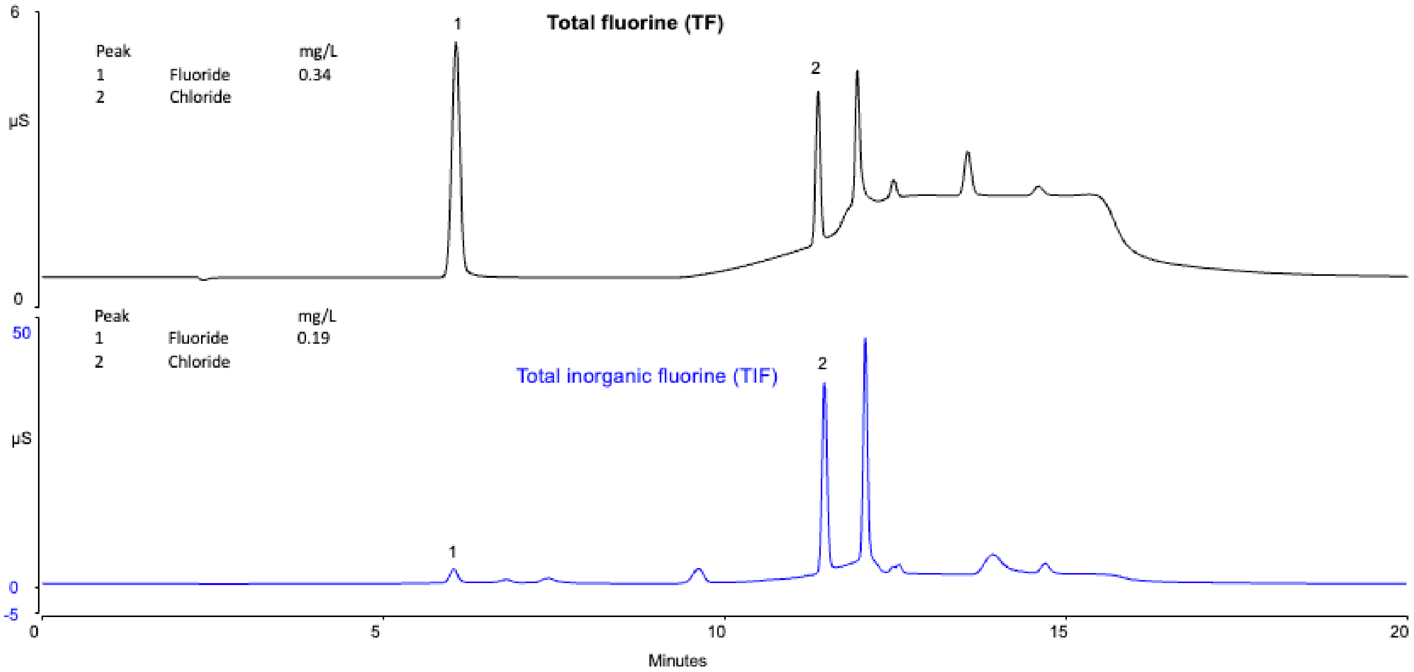

**Figure 5. TF and TIF chromatogram (Sample 4).**
Figure 5 shows the IC chromatograms of TF and TIF for Sample 4. Fluoride is well separated from other anions that may be present in samples, allowing for accurate determination.

## Conclusion

A streamlined TOF workflow using the Cindion C-IC system was developed for PFAS screening in cosmetic products. The method has a TF detection limit of approximately 0.91 ppm, well below the level of 100 ppm often used as a screening threshold for high-fluorine samples, and the method is highly accurate and precise for both TF and TIF determination.

This C-IC-based TOF approach enables cosmetic manufacturers and regulatory laboratories to efficiently flag samples that may contain intentionally added PFAS and require follow-up targeted analysis by LC-MS. The integrated 2-in-1 Cindion C-IC system, operated through the Chromeleon CDS, is well-suited for routine laboratory use and supports compliance with emerging regulations such as California AB 2771.

## References

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## List of abbreviations

PFAS: Per-and polyfluoroalkyl substances

IC: Ion chromatography

C-IC: Combustion ion chromatography

TF: Total fluorine

TOF: Total organic fluorine

TIF: Total inorganic fluorine

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