

NC STATE Wilson College of Textiles

Textile Protection and Comfort Center

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A Testing Service Report

to

Full Circle Lithium

on

Analysis of Total Fluorine in Liquid and Powder Samples

from

Textile Protection and Comfort Center (TPACC)
Wilson College of Textiles
North Carolina State University
Raleigh, North Carolina

Report Date: April 14, 2025

Analysis of Total Fluorine in Liquid and Powder Samples

Three liquid samples T1, T2 & T3, and one solid sample in powder form T4-T6 were submitted by the **Full Circle Lithium** to the Textile Protection and Comfort Center (TPACC) at the Wilson College of Textiles, North Carolina State University. The test material was evaluated for total fluorine content to determine whether PFAS had been intentionally added to the material. The purpose of this report is to present the test results.

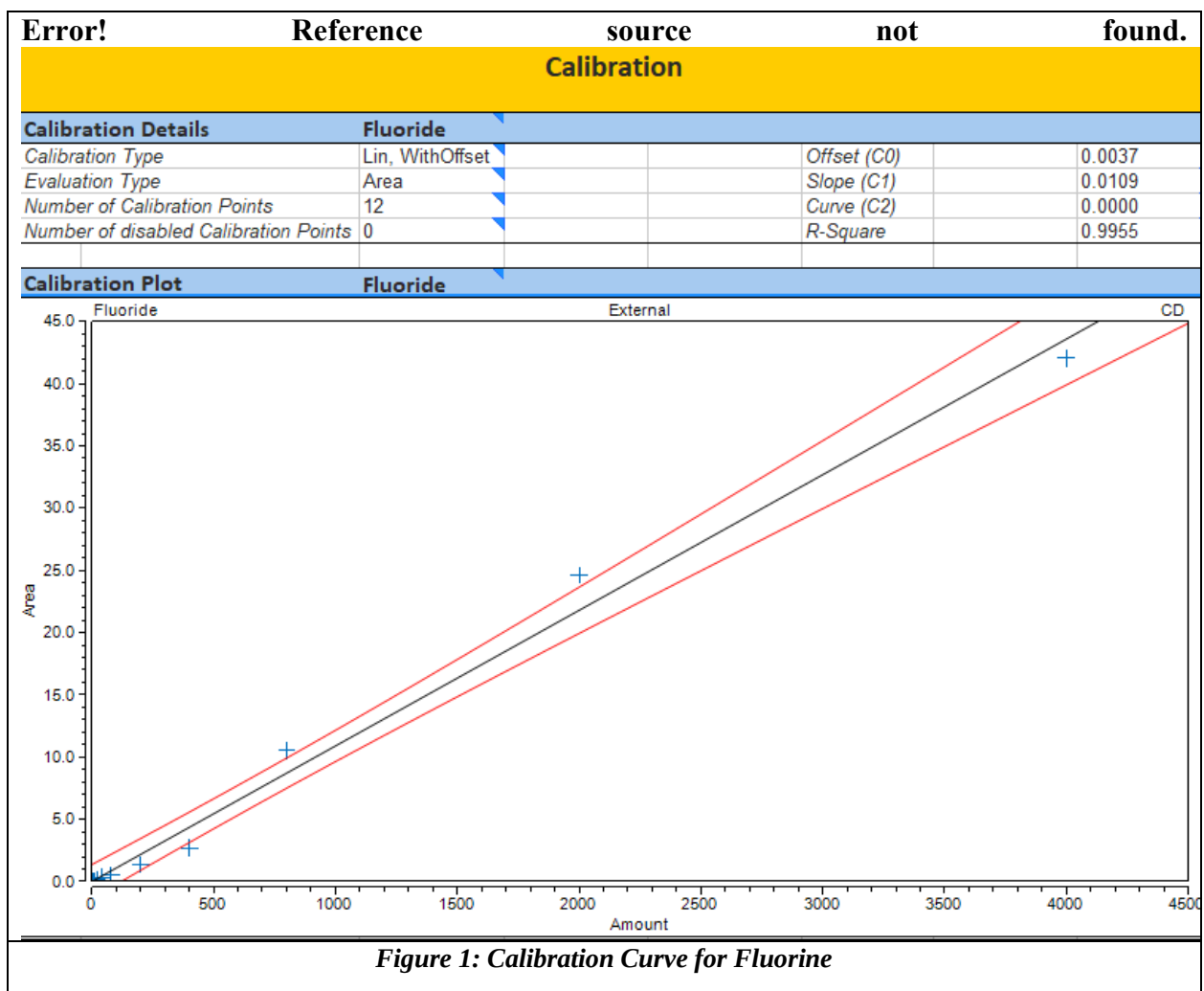
Test Materials

Full Circle Lithium provided three liquid samples in 50 mL conical vials and one solid sample in powder form in a zipper storage bag for evaluation of the total fluorine. Three replicates (~30 μ L) from each of three liquid sample and three replicates approximately 30 mg each from powder sample were weighed in a precleaned cup for the analysis. Fluorobenzoic Acid stock solution was freshly prepared with a concentration of 500 ng/mL (PPB) in UPW. Two working stock solutions with 5 ng/mL (PPB) and 50 ng/mL (PPB) concentrations were prepared for the calibration points. Stock and working stock solutions were injected (1-50 μ L) into pre-baked, clean ceramic cups for combustion to establish the calibration points ranging from 0.8 PPB to 4000 PPB. Total elemental fluorine encompasses organofluoride compounds, such as PFAS and fluoropolymers, in addition to the presence of inorganic fluorine.

Analysis of Total Fluorine

Total fluorine was quantified via direct analysis using a Thermo-TE Instrument XPREP C-IC system (TE Instrument, Netherlands), following the ASTM D7359-23 method. 30 μ L/mg of each sample was pipetted/weighed into pre-baked ceramic cups and combusted at 1000 °C in the XPREP combustion unit under a flow of oxygen ($\approx$ 300 mL/min) and argon ($\approx$ 150 mL/min) mixed with water vapor for approximately 5 minutes. The resulting combustion gases were absorbed in UPW in the XPREP C-IC fraction collection unit. A 1000 μ L aliquot of the absorption solution was injected into a Dionex Integrion HPIC (Thermo Fisher Scientific) equipped with a 500 μ L sample loop, a Dionex™ CR-TC anionic exchange column and a Dionex™ IonPac™ AS19-4 μ m analytical column, operated at 30 °C. Chromatographic separation was achieved using a gradient of aqueous hydroxide mobile phase, ramping from 5 mM to 55 mM at a flow rate of 1 mL/min over 25 minutes. Fluoride was detected using a conductivity detector. Sample quantification was performed using 12-point calibration curves ranging from 0.8 to 4000 ng/mL (PPB).

The calibration plot of fluorine is provided in the **Figure 1**.



Data Analysis

To determine the total fluorine in each sample the following formula was used.

$$\text{Total Fluorine (ng/g)} = \frac{(\text{Calculated Concentration of Fluorine in IC (ng/mL)} \times \text{Volume of Combusted Solution (mL)})}{\text{Weight of Test Specimen (mg)}} \times 1000$$

Test Results

Total fluorine in the samples was determined using a CIC system, with measurements expressed in ng/g based on an external calibration curve created using Fluorobenzoic Acid solutions ranging from 0.8 to 4000 ng/mL (PPB). The instrument can consistently and accurately quantify (**LOQ**) fluorine when the concentration is above 1.5 ng/mL (PPB), however the limit of detection (**LOD**)

of the instrument is 0.5 ng/mL (PPB), which represent samples LOQ ~**350 ng/g (PPB)** and LOD ~**110 ng/g (PPB)** when 30 mg sample is combusted.

No fluorine was detected in the sample T1, and a very low concentration of fluorine was detected in the rest of the three samples T2, T3 and T4-6 (**Table I**). Total fluorine concentration in samples T2 & T4-6 were below the instruments LOQ but above LOD. The total fluorine concentrations in sample T3 were ~471 ng/g (PPB).

The total fluorine concentration in the samples were below **1 PPM**. Typically, **100 µg/g (PPM)** which is **100,000 ng/g (PPB)**, total fluorine level is an indication of intentionally-added PFAS.

Table I: Total Fluorine Concentration in the Liquid and Solid Samples

Sample	Replicate	Total Fluorine (ng/g) /PPB	Average Total Fluorine (ng/g)/PPB	Average Total Fluorine in PPM
T1 (Liquid)	1	ND	ND	ND
	2	ND		
	3	ND		
T2 (Liquid)	1	ND	70.02	0.07
	2	ND		
	3	210.05		
T3 (Liquid)	1	618.8	471.3	0.4713
	2	375.9		
	3	419.1		
T4-6 (Powder)	1	ND	286.53	0.287
	2	644.59		
	3	215.01		
ND: No Detection				