Experimental implication of PIGE (Particle Induced Gamma Ray Emission) and standard modifications

M. McCarthy, E. Salas, A. Rodriguez Manso, J. Gauthier, and S.J. Yennello

PIGE (Particle Induced Gamma Ray Emission) is an ion beam analysis technique that is used to determine the composition of a sample, and has been implemented.

When low MeV protons interact with the nucleus of a target atom, excitation of the nucleus occurs due to proton induced nuclear reactions. Upon de-excitation, characteristic gamma rays are emitted. PIGE can be used for low Z elements and is sensitive up to parts per million (ppm) [1]. PIGE has been used for identifying per- and polyfluoroalkyl substances (PFAS). PFAS are commonly used in commercial products for waterproofing and fireproofing consumer products. These include food containers, curtains, couch cushions, fireproof foam. However, health and environmental concerns have been linked to PFAS [2].

In November 2018, we used a 3.4 MeV proton beam delivered by the K150 TAMU cyclotron to study a set of cosmetic products to determine if PFAS were contained in the products, in particular products that were marketed as being waterproof. Gamma rays were detected by a XR-100 CdTe diode detector placed 45° from the beam axis. Fluorine has two gamma lines at 110 and 197 keV that are used as a marker for PFAS. Fig. 1 shows an example of a Teflon gamma ray spectra with the distinctives peaks.
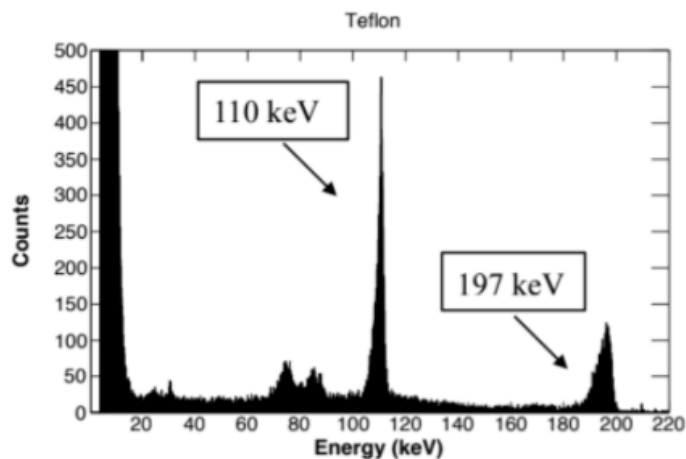

FIG. 1. An example gamma spectra of Teflon. The two distinct Fluorine peaks are marked at 110 and 197 keV.

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The summary of the cosmetic products tested for fluorinated compounds concentrations by utilizing the calibration curve in Fig. 2, is shown in Table I. The samples were prepared by swatching a
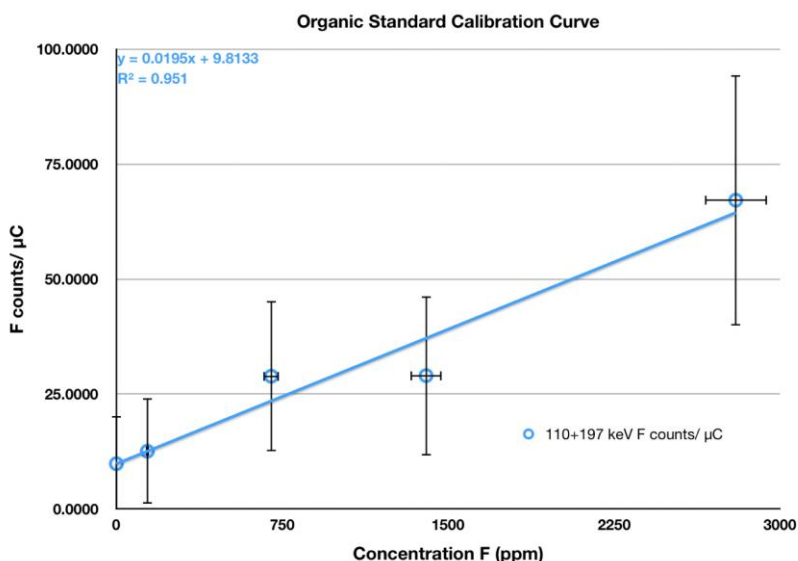

FIG. 2. Organic Standard Calibration Curve. The standards ranged from 0-2800 ppm of F. The plot shows the concentration of the standards versus the normalized Fluorine counts. Errors were computed using statistical and beam errors. The errors are relatively large due to degradation of the standards over the beam exposition.

thin layer on a non fluorinated filter paper. A similar procedure is used where the fluorine gamma lines were integrated and normalized. The trendline in the calibration curve allows the backtrack and determine the concentration from the fluorine counts. These results show a considerable amount of Fluorine was present in multiple cosmetic products labeled as waterproof.

The standards used for the analysis were prepared with PFOA (Perfluorooctanoic acid). PFOA is relatively volatile, and degradation of the standards over beam use has been observed. As a consequence, two new sets of standards were prepared with PFOA and were diluted with methanol and water, and then pipetted on non-flourinated filter paper. Both sets of standards ranged over 0 - 12k ppm of F over two months. These will be tested to determine the longevity of the standards and the degradation due to beam use in future experiments.

Table I. Summary results of the cosmetic products. Over a third of the set shows significant amount of Fluorine present.

Sample Name	Determined Fluorine Concentration (ppm)
Popcorn Bag	4650.0
C10 Pet Proof	Undetectable
Oven Bags	Undetectable
Urban Decay Foundation	Undetectable
Urban Decay Water Proof Powder	2209.74
Clinique Foundation Ever better	Undetectable
Coverfix Foundation	5390.50
Fenty 130 Foundation	Undetectable
Sephora 27 Foundation	Undetectable
Nars Gobi Foundation	Undetectable
Smashbox Concealer Waterproof	Undetectable
Clinique Beyond Perfection Concealer	279.93
Sephora Concealer	1991.19
Muf Primer	Undetectable
Clinique Super Primer	Undetectable
Nars Primer	Undetectable
Laura Geller Matte Primer	Undetectable
Xo Beauty Haroika	658.42
Tarte Shape Tape Concealer	Undetectable
Nars American Women Liquid Lipstick	Undetectable
Jeffree Star Liquid Lipstick	578.75
Wet n Wild Liquid Catsuit	1883.14
Milani Liquid Lipstick	129.37
NYX Stockholm Lipstick	1608.29
Kat Von D Bauha 5	1260.07
Anastasia Beverly Hills Liquid Lipstick	139.24
Dose of Colors Stone Liquid Lipstick	1497.26
Mac Lipstick	Undetectable
Stila Liquid Eyeshadow	Undetectable
Urban Decay Eye Primer	1098.84
Too Faced Melted Lipstick	Undetectable
Tarte Lip Paint Man Bun	Undetectable
Pixi Au Naturelle Liquid Lipstick	Undetectable
Tarte Blush Art Deco	1602.60
Tarte Eyeshadow Caretaker	Undetectable
Fenty Contour	Undetectable
Anastasia Beverly Hills Eyebrow Gel	Undetectable
Anastasia Beverly Hills Brow Pomade	Undetectable

[1] M.B.H. Breese, D.J. Jamieson, and P.J.C. King, *Materials Analysis using a nuclear microprobe*, (John Wiley & Sons, New York, 1996).

[2] Zhanyun Wang, Jamie C. DeWitt, Christopher P. Higgins, and Ian T. Cousins, Environ. Sci. Tech. **51**, 2508 (2017); 10.1021/acs.est.6b04806.