

# The Analysis of PFAS in AFFF Products: Application of Isotope Dilution Mass Spectrometry and TOP

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**Background/Objectives.** Per- and poly-fluorinated alkyl substances (PFAS) are ubiquitous in the environment from multiple industrial sources such as aqueous film forming foams (AFFF) for fire fighting, and from household sources such as food packaging and waterproofing coatings. Some perfluorinated carboxylic (PFCA) and sulfonic (PFSA) acids such as PFOA and PFOS are persistent in the environment and have been linked to a number of health concerns. Sites affected by the historical use of AFFF foams containing PFOS (currently phased out in North America), represent a significant characterization and remediation challenge. Replacement AFFF products containing other PFAS need to be characterized, both for residual concentrations of recalcitrant carboxylic (PFCA) and sulfonic (PFSA) acids such as PFOS, PFOA and PFHxS, and for the presence and potential of “precursors” that can transform into stable PFAS in the environment. The analysis of AFFF products for residual PFAS is challenging due to the need to balance residue limits in consideration by regulatory agencies with the high and unpredictable PFAS levels in the product, with the presence of high levels of matrix components, and with the potential surfactant nature of the products. Our study’s objectives are to develop and validate analytical procedures to reproducibly assess residual PFAA/select precursor content in a variety of current-use AFFF products, to report on residual levels from these products, and to assess the potential of precursors to form PFAA using the total oxidisable precursor assay (TOP).

**Approach/Activities.** Method development for the analytical method utilized existing LC-MS/MS procedures validated in water for 29 PFAS analytes including C4-C14 PFCA, C4-C12 PFSA (excluding C11) and select PFAS precursors including 4:2-8:2 fluorotelomer sulfonates (FTS). Briefly, the product samples are pre-screened to qualitatively assess overall levels, then this pre-screen data is used to select a sample size that balances expected levels with a desired reporting limit of 10 ng/mL (ppb). Samples are then diluted in reagent water, spiked with an isotopically labeled internal standard mix, and extracted using weak anion exchange to remove matrix interferences. The extracts are then analyzed for a relevant sub-set of the 29 PFAS using LC-MS/MS. For the total oxidizable precursor assay, a very small (0.1 mL or less) of the product is oxidized with persulfate in an alkaline environment for 6 hours following the standardized TOP protocol to convert all oxidizable precursors into PFCAs. The difference between the PFCAs measured before and after the TOP assay represents the precursor potential of the product sample. Six different product formulations in current use will be tested.

**Results/Lessons Learned.** Preliminary results from testing of archived AFFF products indicate that the weak anion exchange cleanup procedure was effective in removing matrix interferences. The combination of isotope dilution and careful sample size selection resulted in good data quality. Preliminary TOP analysis in AFFF-affected groundwater samples showed a 300% increase in PFCAs with increase patterns indicative of the use of a 6:2 telomer AFFF product, and provided insight into sample size limitations that will apply to the application of the method in AFFF products. Method validation, quality evaluation and results from the testing of five to six AFFF products will be presented.