DEVELOPMENT AND VALIDATION OF PFAS ANALYTICAL METHODS FOR CHARACTERIZATION OF COMPLEX MATRICES

David Patch, PhD

Background/Objectives:

The widespread use of per- and polyfluoroalkyl substances (PFAS) has resulted in their occurrence in a number of environmental matrices, including water, soils, sediments, organisms, biosolids, and the atmosphere, as well as inclusion in industrial processes and consumer products. Existing PFAS analytical methods can struggle when applied to complex matrices, especially in situations where the PFAS concentration is low and there are high concentrations of other constituents. Classically, most PFAS analytical methods rely on mass spectrometric techniques, which need commercially available analytical reference materials to ensure accurate quantitation of the compounds of interest. With over 14,000 PFAS compounds having been identified, and less than 100 commercially available reference materials, there is a growing gap in the total PFAS that may be present in a sample, and what PFAS can be analyzed. Methods have been developed to address this gap, including the use of high-resolution mass spectrometry, the total oxidizable precursor (TOP) assay, and the absorbable/extractable organic fluorine (AOF/EOF) assays. While these methods are effective, they have their own inherent drawbacks, including difficulty handling complex matrices, and issues with complete mass balance closure. Furthermore, these methods often require significant resource investment, and often have extensive turn-around times when performed by commercial laboratories.

Approach/Activities:

Parsons has developed several in-house analytical methodologies to either augment commercially available PFAS analysis or allow for the real-time determination of PFAS concentrations. The methods developed are refinements of methods published in the academic literature, including the ultraviolet-light (UV) activated TOP assay (Patch et al. 2024), the UV-reducible organic fluorine (UV-ROF) assay (Patch, O'Connor et al., 2025), and the colorimetric active surfactant (CAS) assay. Where possible, methods have been validated using third-party commercial analysis employing EPA Method 1633 or EPA Method 1621 (AOF). These methods have been applied to different matrices, including AFFF-impacted waters, soils, and captured air emissions.

Results/Lessons Learned:

The following conclusions have been identified based on the results of this study.

- PFAS concentrations can be determined at a screening level of confidence using indirect quantitation methods, such as UV-ROF and CAS
- The UV-activated TOP assay can be used to improve PFAS characterization using both external analysis (EPA Method 1633) and in-house screening analysis (UV-ROF, CAS assay)
- The two biggest challenges in performing in-house PFAS analysis are matrix complexity, and initial PFAS concentration
    - Matrix complexity is overcome using different sample preparation methods (e.g. mild oxidation, extractions, filtration)
    - In samples with high PFAS concentrations, sample dilution is used to reduce the PFAS concentration to the linear dynamic range of the detection method
    - In samples with low PFAS concentrations (<50 ppb), sample pre-concentration, using granular activated carbon or ion exchange resins, are used to increase the PFAS concentration to the linear dynamic range of the detection method
- In-house PFAS analysis is supported by a robust quality assurance program, including sample duplicates, matrix spiked replicates (PFAS, inorganic fluoride), and blanks

- The use of in-house screening methods can be performed at a fraction of the cost of commercial laboratories, can provide results significantly faster, and can be used identify which samples are most appropriate to send for additional analysis
- The developed methods are most effectively applied to samples in which PFAS concentrations range from µg/L to mg/L, but sample pre-processing steps can be employed for more sensitive applications

About The Author

David Patch, PhD, is an analytical chemist with academic and industrial experience specializing in the analysis and remediation of legacy and emerging environmental contaminants, including metals, nanomaterials, microplastics, and PFAS. He develops novel analytical methods for complex environmental and product matrices.