# Current and Future Federal and State Sampling Guidance for Per- and Polyfluoroalkyl Substances in Environmental Matrices

Justin Kidd[a*], Emily Fabricatore[a], Dennis Jackson[a]

a. Savannah River National Laboratory (SRNL), Aiken, South Carolina, 29808, United States

* Corresponding Author:
*E-mail Address*: justin.kidd@srnl.doe.gov

## Abstract

Per- and polyfluoroalkyl substances (PFAS) are a class of emerging contaminants composed of an estimated 5,000 to 10,000 human-made, fluorinated, organic chemicals. Due to the complexity of PFAS, the need for multiple environmental matrix considerations and the absence of a promulgated federal standard for environmental sampling and analysis, U.S. states have begun developing health-based regulatory and/or guidance values for a limited number of PFAS in environmental matrices. As there is a growing body of science to inform PFAS sampling guidance standard development, it is important to understand which U.S. states are implementing sampling guidelines and how they plan to handle emerging PFAS. This critical review discusses the current and impending federal and state sampling guidelines for PFAS in environmental matrices, the data gaps surrounding PFAS sampling guidance in U.S. states, and the future impacts of impending guidance documents and regulations. Ten federal guidance documents are available for PFAS sampling guidance and analysis. The maximum number of PFAS covered in these guidance documents is 25 analytes spanning across 8 unique media. While the EPA has developed several different sampling and analytical guidelines for PFAS, there is no formal regulation of PFAS or requirements of states to enforce these guidelines. As a result, only 31 states have informally adopted sampling guidelines, while the other 19 states have no guidance documentation in place for PFAS. The introduction of new PFAS sampling guidelines by the EPA, as well as updated analytical guidelines that target more PFAS or total organofluoride, is expected to continuously shift the landscape of federal and state guidance for PFAS sampling moving forward.

## 1. Introduction

Per- and polyfluoroalkyl substances (PFAS) are a class of emerging contaminants composed of an estimated 5,000 to 10,000 human-made, fluorinated, organic chemicals (EPA, 2018; ITRC, 2020; Dorrance et al. 2017). The actual number of PFAS is continuously changing, as some are no longer produced due to regulatory actions, while others are developed as their alternatives (Dean et al. 2020; Wang et al. 2019; Sheng et al. 2019). The carbon-fluorine bond of PFAS is one of the strongest bonds in nature, making PFAS resilient to biological, chemical, and thermal degradation (Kucharzyk et al. 2017; Merino et al. 2016; Frömel and Knepper, 2010). PFAS have unique chemical properties (e.g., low surface tension, oil-repellent, water soluble) (Rayne and Forest, 2009; Kim et al. 2015; Ahrens et al. 2009) that enable their use in biocides, hydraulic fluids, fire-fighting foam, and household products (Buck et al. 2012; Glüge et al. 2020; Høisæter et al. 2019; Backe et al. 2013; Herzke et al. 2012). There is a concern for potential environmental contamination due to the presence of PFAS in numerous industrial and consumer products, as well as their persistence in the environment, likely increasing their concentrations over time (Brendel et al. 2018). PFAS are potentially capable of producing a wide range of adverse health effects depending on human exposure circumstances, including negative immune function (Grandjean et al. 2017; Abraham et al. 2020), negative thyroid function (Melzer et al. 2010; Winquist and Steenland, 2014), liver disease and cancer (Sakr et al. 2007; Darrow et al. 2016; Massoud and Charlton, 2018), and other human diseases (Steenland et al. 2020; Di Nisio et al. 2022).

The discharge of PFAS to the environment presents additional challenges, increasing the complexity of PFAS detection and quantification. PFAS are commercially manufactured as isomeric mixtures of linear and branched chains by electrochemical fluorination or as linear isomers by telomerization (Benskin et al. 2010). While most studies report both linear and branched isomers as “Total PFAS”, the differing properties of linear and branched PFAS isomers can affect the partitioning and migration of PFAS in aquatic environments (Chen et al. 2015). PFAS have both hydrophobic and hydrophilic properties that influence their fate and transport in the environment. Long-chain PFAS adsorb on soils (Filipovic et al. 2015; Moody and Field, 1999) and particles in solution (Gagliano et al. 2020) and accumulate between air-water and soil-water interfacial regions (Guelfo et al. 2021; Karoyo and Wilson, 2016), Short-chain PFAS dissolve in solution and have high mobility in water bodies (Ahrens, 2011; Ateia et al. 2019; Brendel et al, 2018), increasing the potential for human exposure to PFAS. Analytical challenges for PFAS include an increasing number of PFAS subclasses (ITRC, 2020), applicability of analytical methods to a limited number of environmental matrices (Winchell et al. 2021), a lack of differentiation between organic and inorganic fluorine (McDonough et al. 2020), poor instrument sensitivity (Menger et al. 2021), and nonstandardized analytical methods (Al Amin et al. 2020).

Common methods for PFAS analysis include liquid chromatography with tandem mass spectrometry (LC/MS/MS), Total Oxidizable Precursor (TOP) assay, and Total Organic Fluorine (TOF). PFAS analysis has primarily been through LC/MS/MS; however, results do not provide a comprehensive measurement on the magnitude of the total PFAS that may exist in environmental matrices, nor does it measure the potential for targeted PFAS formation due to transformation of precursors over time. TOP and TOF assays are lower cost alternatives that provide a more comprehensive measure of total organic fluorine as a proxy for total PFAS contamination, but lack in specificity and sensitivity. For LC/MS/MS, the use of “dilute and shoot”, or direct injection, of samples is the most common technique for analyzing PFAS samples. Other methods, like the Environmental Protection Agency’s PFAS methods 533 and 537.1, call for PFAS extraction from water samples using solid phase extraction (SPE) with a weak anion exchange cartridge to provide high recoveries of shorter- and longer-chain PFAS.

Due to the complexity of PFAS, the need for multiple environmental matrix considerations and the absence of a promulgated federal standard, states have begun developing health-based regulatory and/or guidance values for a limited number of PFAS in environmental matrices. Historically, many states have relied on standards promulgated by federal agencies to sample, analyze, and regulate chemicals, while others have had the authority to develop their own standards (Post, 2020; EPA, 2016). When no federal standard exists, states may rely on United States Environmental Protection Agency (EPA) suggested values or similar reference documents from other states. Other states have adopted the EPA lifetime health advisory (LHA) for drinking water and/or groundwater to guide their efforts upon detection of PFAS (EPA, 2002).

There is a growing body of science to inform sampling and analysis standard development, an absence of a federally enforceable standard, and pressures from the public and legislative bodies to take regulatory action against PFAS. Therefore, it is important to know which states are developing their own sampling guidelines, which states are using federal guidelines, and which states are not actively sampling for PFAS. This is essential so that states can make informed decisions when implementing their own regulations and/or risk communication practices. The goal of this review is to (1) identify the current and impending federal and state sampling guidelines for PFAS in environmental matrices, and (2) determine the data gaps surrounding PFAS sampling and analytical guidance in the U.S. and (3) assess the future impacts of impending guidance documents and regulations.

## 2. Methods

### *2.1. Information Sources and Data Management*

PFAS information from each state was collected by accessing each state's environmental or health department website. For websites that did not provide adequate information on PFAS sampling and analytical state guidelines, an email was sent out to a point-of-contact within the state environmental department and the information was retrieved via email or telephone. The SciFinder electronic database was used to assess the number of publications of PFAS in published literature and find the number of articles (on any topic) mentioning each PFAS compound. The number of studies found was bound from 2002 to 2020. This range of dates was chosen because in 2002 the Environmental Protection Agency first attempted governmental risk management of PFAS by publishing a Significant New Use Rules (SNUR) to require notification to the EPA before any future manufacture (including import) of 75 PFAS (EPA, 2002). The SciFinder search was conducted for each PFAS by combining results found when searching for the chemical name, the systematic IUPAC name, and the acronyms for the relevant PFAS. The information found from each state environmental department and the data found from the SciFinder search were housed on secure internal servers and analyzed on Microsoft Excel and R Statistical Software (R Core, 2020).

## 3. Current and Emerging Federal Methods for PFAS Sampling and Analysis in Environmental Matrices

### *3.1.Classification of PFAS*

Of the thousands of PFAS predicted to be in the environment, current literature has focused primarily on perfluoroalkyl acids (PFAAs) and PFAA precursors, which are part of the nonpolymer subclass of PFAS (Figure 1). PFAAs are perfluorinated substances where fluorine atoms have replaced all hydrogen atoms attached to carbon atoms (with exception of those associated with functional groups). As a result, these compounds are recalcitrant to degradation and are persistent in the environment (Cousins et al. 2020; Ahrens and Bundschuh, 2014). The PFAA subclass includes perfluoroalkyl carboxylic acids (PFCAs), perfluoroalkyl sulfonic acids (PFSAs), perfluoroalkyl phosphonic acids (PFPAs), perfluoroalkyl phosphinic acids (PFPiAs), perfluoroether carboxylic acids (PFECAs), and perfluoroether sulfonic acids (PFESAs). PFAA precursors are chemicals that have the potential to degrade into perfluoroalkyl substances (Hamid et al. 2018; Bräunig et al. 2017; Dauchy et al. 2017). These PFAA precursors are chemicals used in surface coatings and surface protection formulations, as raw materials for surface treatment products (e.g., surfactants, emulsifiers), or are formed as intermediate environmental transformation products (Langberg et al. 2020); Houtz et al. 2013; Lau et al. 2007). Recent research has also reported several PFAA precursors in personal care products like cosmetics (Whitehead et al. 2021; Schultes et al. 2018).

Figure 2 highlights the current published literature on PFAAs and their precursors. PFCAs and PFSAs have the most publications among PFAA subclasses, with perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS) being the two chemicals with the most publications since 2002. This is the year the EPA published the Significant New Use Rules (SNUR) to require notification to EPA before any future manufacturing or importing of 75 PFAS (EPA, 2002).

## *3.2.Approved Standard Methods for PFAS Sampling and Analysis*

### *3.2.1. Comparison of PFAS sampling between approved standard methods*

There are ten approved standard methods available for PFAS sampling and analysis developed by U.S. governmental agencies and international organizations (EPA, 2020a; EPA, 2020b). These methods have been through a laboratory validation process following a rulemaking or guidance effort and are available to support regulatory guidance activities. These ten methods are used in a variety of environmental matrices to assess different PFAS. Table 1 highlights the different methods, what environmental matrices they can be applied to, what analytical instrument is being used to analyze PFAS, and the number of PFAS available for analysis by each method. These 10 standard PFAS sampling and analytical methods cover 40 different PFAS analytes across 8 unique media, while using 7 different analytical methods to detect and quantify PFAS in samples. PFAS sampling guidance for each standard method was also reviewed to assess if each method contained information on appropriate personal protective equipment (PPE), sample collection, sample preparation, sample decontamination, sample shipment, and sample storage (Table 2). While each method provided guidance on using polypropylene containers to collect PFAS samples, there is no consensus between standard methods on any of the other PFAS sampling parameters. Some of these PFAS sampling parameters are not available or discussed within the methods. The results here highlight the need for standardization in PFAS sampling procedures and methods. The wide variety of environmental matrices, sampling procedures, analytical methods, and PFAS in these 10 standard methods leads to a lack of data comparability between research laboratories and potential cross-contamination of PFAS samples.

### *3.2.2. Comparison of PFAS between approved standard methods*

Figure 3 highlights which PFAS each of the ten standard methods sample for and analyze. Every standard method samples for and analyzes PFOA and PFOS. The majority of PFAS that are evaluated using these standard methods belong to the PFAA and PFSA subclasses. These methods do not sample or analyze for chemicals within PFPA or PFPiA subclasses. Additionally, Figure 4 compares the number of journal publications versus the number of standard methods available for each PFAS. The general trend indicates that the number of standard methods available for PFAS increases with journal article publications, with more methods for PFCAs highly represented in publications. However, what is important is that there is no universal standard method to sample and analyze common PFAS. States with PFAS-contaminated locations are not likely to contain similar PFAS as other states because region-specific biogeochemical conditions may influence PFAS fate and transport in the environment (Ahrens et al. 2009; Yang et al. 2011; Writer et al. 2011; Labadie and Chevreuil, 2011). Some states also have unique PFAS contamination scenarios, such as North Carolina (Kotlarz et al. 2020), where fluorochemical manufacturing has led to contamination with novel PFAS. The combination of diverse sources and environmental processes will lead to unique PFAS compositions. Therefore, it is valuable to understand what PFAS are of concern to which states and what sampling and analytical methods or guidance they are using or developing to assess emerging PFAS.

Table 3 compares the detection limits of PFAS for each approved standard PFAS sampling and analytical method. For the majority of PFAS and methods in water matrices, the limit of detection is between 1.0 and 10 ng/L. The limit of detection for PFAS varies in soil (2 – 22 ng/kg), food (3 – 67 ng/kg), bovine muscle (0.5 – 13 ng/kg), and serum (1 – 6 ng/mL). Additionally, only PFOA and PFOS are analyzed in each of the 10 accepted methods. The variability in PFAS detection limits can be attributed to a lack of standardization of PFAS analytical methods across a suite of environmental matrices. The use of different PFAS, different detection limits, and sample preparation methods with different selectivity between the accepted standard PFAS sampling and analytical methods potentially reduces the reliability of the methods. Standardizing PFAS sampling and analytical methods would facilitate confidence in researchers that the PFAS data they are gathering is from the same empirical phenomena.

The manufacturing method of PFAS can yield either branched or linear isomers. Branched isomers of PFAS may produce lower responses for product ions, which may lead to low bias measurements of these isomers if used for quantitation using LC/MS/MS. Each approved PFAS sampling and analytical method has their own unique determination for the analysis of PFAS isomers. CDC Method 6304.08 allows for the differentiation between linear and branched isomers and includes recommended standards for both. ISO Method 25101:2009 is specified as a method for linear isomers. Both ASTM D7968-17a and D7979-17 identify that PFAS tested in the methods are comprised of isomeric mixtures and that the entire isomeric group is quantified in the method. For FDA Method C-010.02, PFOA and PFOS are quantified and reported as the sum of linear and branched isomers; however, the other analytes are linear isomers. USDA Method CLG 2.03 does not disclose information on PFAS isomers in the method. EPA Methods 3512, 8327, 537.1 and 533 note that standards for the target PFAS in the method may contain both branched and linear isomers; however, the CAS for each standard is for normal-chain isomers. These results indicate that while PFAS linear isomers are intended for standard analytical methods, there will likely be branched isomers present which may create obstacles when trying to quantify PFAS in environmental matrices using LC/MS.

### *3.2.3. Approved EPA Methods for PFAS Sampling and Analysis in Environmental Matrices*

The EPA is responsible for providing sampling and analytical methodologies of emerging contaminants in support of the Safe Drinking Water Act (SDWA) and the Clean Water Act (CWA). As there currently is no nation-wide regulation of PFAS, many federal agencies, state departments, national and private labs, and public research universities rely on EPA sampling and analytical guidance documents when assessing emerging contaminant concerns. The EPA has developed three approved standard methods for sampling and analyzing PFAS in potable and non-potable waters: EPA Methods 533,537.1, and 8327 (EPA, 2020a). EPA Method 533 is an EPA isotope dilution method for the determination of 25 PFAS in drinking water and was developed to support the SDWA and the Fifth Proposed Unregulated Contaminant Monitoring Rule (UCMR5) sampling effort. This method targets "short chain" PFAS, which are PFSAs with carbon chain lengths less than $C_6$ or PFCAs with carbon chain lengths less than $C_8$, including PFCAs, PFSAs, fluorotelomers, and poly/perfluorinated ether carboxylic acids. EPA Method 537.1 is a method for the determination of 18 PFAS in drinking water. It was first published in 2009 but has been modified twice to accommodate additional PFAS that have the potential to contaminate drinking water or have been identified as PFOA/PFOS alternatives in manufacturing. EPA Method 8327 is a direct injection method for the determination of 24 PFAS in non-drinking water (e.g., surface water, wastewater) aqueous samples that was developed primarily for the CWA.

### *3.3.Emerging EPA Standard Methods for PFAS Sampling and Analysis in Environmental Matrices*

The EPA is developing eight additional standard methods for PFAS sampling and analysis in addition to EPA 533, EPA 537.1, and EPA 8327, the three currently approved protocols, which are primarily used for drinking water and non-potable water. Table 4 lists the EPA standard methods being developed and the environmental matrix they will be used in (EPA, 2020b). EPA method 8328 will target non-potable water and environmental media. OTM-45 and TO-15 will target source air emissions. Other methods being developed will target near-source, semi-volatile, and volatile PFAS in ambient air. Lastly, the EPA is developing methods for total organic fluorine and total organic precursors in environmental media. The methods that target environmental media, source air emissions, and ambient air are significant because current EPA PFAS methods are only suitable for drinking water and non-potable water, even though PFAS contamination extends beyond water matrices. Additionally, current methods may significantly underestimate the total number of PFAS present in contaminated regions when only targeted analytical methods are used. Total organic fluorine or total organic precursor methods, which target bulk organofluorine, may provide additional insight into the currently unidentified fractions of PFAS in contaminated regions. The EPA is developing draft methods or considering the need for a thorough multi-laboratory validation study for each of these methods with anticipation times of completion up to the end of 2022. In one such case, the EPA has recently developed EPA draft method 1633, which is a single laboratory validation study of PFAS by isotope dilution using liquid chromatography-tandem mass spectrometry (LC-MS/MS). This method is designed for analyzing 40 PFAS in aqueous matrices (wastewater, surface water, groundwater, landfill leachate), solids (soil, sediment, biosolids), and fish and clam tissues (EPA, 2021). This method is being tested further in a multi-laboratory validation study to be completed in 2022.

### *3.3.1. Standard Methods for PFAS in Non-Potable Water and Environmental Media*

The EPA is collaborating with the United States Department of Defense (DoD) to develop an isotope dilution method (EPA 8328) for non-drinking water aqueous matrices. Matrices able to be analyzed with method 8328 include surface water, groundwater, wastewater influent/effluent, landfill leachate. This method will also be used for fish tissues, biosolids, soils, and sediments. This method will sample for and analyze the 24 PFAS that are covered under EPA 8327 with the addition of GenX substances. GenX technology has been used in the United States since 2005 to produce fluorinated polymers (Wang et al. 2013; Wang et al. 2015). These GenX substances were developed to replace PFOA; however, these substances are under governmental review due to possible similar carcinogenic and toxic effects as other PFAS (Coperchini et al. 2020; Temkin et al. 2020; EPA, 2018). This method is currently undergoing single lab validation and will undergo multi-lab validation upon completion, followed by public comment.

### *3.3.2. Standard Methods for PFAS in Source Air Emissions*

Human exposure to PFAA precursors come from consumer and industrial products available in people's homes (Prevedouros et al. 2006; Shoeib et al. 2005; Langer et al. 2010). Airborne PFAS are thought to break off these products and attach to dust or freely float in indoor air (Ericson Jogsten et al. 2012). However, PFAS air emissions are not as widely understood as PFAS soil and water emissions. Additionally, the lack of toxicity studies on human health risks for airborne PFAS compounds and lack of a standardized method for measuring PFAS air emissions are both significant obstacles for PFAS regulation. PFAS source air emissions are diverse and can be generated by chemical manufacturers, commercial applications, and thermal treatment incineration processes. The EPA is developing test methods for measuring PFAS source air emissions. Modified Test Method 5 Sampling Train OTM-45 (EPA, 2020b) is currently being evaluated for this application. It employs an isotope dilution train approach for targeted and non-targeted gas chromatography-mass spectrometry (GC-MS) analysis of semi-volatile and non-volatile PFAS. Modified Method TO-15 (EPA, 2020b) is also being evaluated for PFAS in source air emissions. This method uses SUMMA® canisters for GC-MS targeted and non-targeted analysis of volatile PFAS. SUMMA canisters are electropolished, passivated stainless steel vacuum sampling devices which are used to collect whole-air samples for volatile organic compounds (VOCs) in source air emissions and ambient air. They are advantageous sampling cannisters for PFAS due to (a) their electropolished inner walls, which reduce PFAS adsorption during sampling events, (b) they do not require a pump during sampling events, and (c) can be used for both source air emissions and ambient air sampling events. These source air emission PFAS methods are currently in development through state and industry collaborations.

### *3.3.3. Standard Methods for PFAS in Ambient Air*

The EPA is considering both targeted and non-targeted analysis of PFAS in ambient air measurements (EPA, 2020b). Anticipated applications include fenceline monitoring for fugitive emissions, deposition, and receptor exposures. A near-source method (unnamed EPA near-source PFAS method) is being developed using a time-of-flight chemical ionization mass spectrometer for real time detection and measurement of PFAS in ambient air. A second method is being developed to assess semi-volatile PFAS in ambient air (unnamed EPA semi-volatile PFAS method) following guidance in EPA TO-13a, which is for the determination of polycyclic aromatic hydrocarbons (PAHs) using GC-MS. A third method is being developed to assess volatile PFAS in ambient air (unnamed EPA volatile PFAS method) using SUMMA canisters and sorbent traps and using GC-MS targeted and non-targeted analysis.

### *3.3.4. Standard Methods for Total PFAS in Environmental Media*

In 2012, the Total Oxidizable Precursors (TOP) Assay was developed to assess the presence of PFAA precursors in aqueous and solid matrix samples (Houtz and Sedlak, 2012). The assay includes steps to oxidize PFAA precursors using heat and alkaline activated persulfate. During oxidation, the assay generates an excess of hydroxyl radicals to fully convert PFAA precursors to

PFCAs. The converted PFCAs can then be quantified using liquid chromatography-tandem mass spectrometry (LC-MS/MS). However, this assay method does not identify individual precursor compounds, provides a conservative estimate of the total concentration of PFAA precursors in any given sample, and is expensive as each sample needs to be analyzed twice for accuracy. Additionally, the TOP assay has not been applied reliably to complex matrices and is primarily validated for water, due to complexities inherent in ensuring repeatable conversion in more complex matrices. The EPA is considering the development of a method based on this TOP assay to identify PFAS precursors that may transform into more persistent PFAS.

The EPA Office of Water – Office of Science and Technology (OW-OST) is also developing a potential rapid screening tool called Total Organic Fluorine (TOF) analysis to identify total PFAS presence and absence in environmental samples. The TOF assay includes steps to undergo pyrohydrolysis in a furnace between 900-1000°C in a humid, $O_2$-rich environment. Under these conditions, PFAS are oxidized, and their C-F bonds are broken. The hydrofluoric acid (HF) formed after PFAS combustion dissociates to form $H^+$ and $F^-$ ions in an absorption solution which is measured using ion chromatography (IC). This method requires the removal of background inorganic $F^-$ from the sample to ensure that only organic F- is reported. Methods to measure TOF in samples include combustion ion chromatography (CIC) or particle-induced gamma ray emission spectroscopy (Duchesne et al. 2020; D'Agostino and Mabury, 2017; Yeung et al. 2013; Wang et al. 2013). There are limitations with this method, such as potentially losing PFAS during combustion, losing F- during sample preparation, and high detection limits of aqueous samples, compared to other environmental matrices (McDonough et al. 2019; Banzhaf et al. 2017).

## 4. U.S. State Guidance for Sampling of PFAS in Environmental Matrices

In 2016, the EPA updated its short-term Provisional Health Advisory values for PFOA (400 nanograms per liter, ng $L^{-1}$) and PFOS (200 ng $L^{-1}$) to a Lifetime Health Advisory (LHA) of 70 ng $L^{-1}$ for PFOA and PFOS, individually or in combination, in drinking water (EPA, 2016). The LHA is a non-regulatory and non-legally enforceable value, but is intended to provide guidance to federal, state, and municipal governments for addressing PFOA and PFOS contamination in public water systems and private potable wells. In 2019, more than 180 countries agreed to phase out and ban production and use of PFOA and PFOS under the Stockholm Convention on Persistent Environmental Pollutants. However, PFOA and PFOS are still environmentally persistent and may still be used in countries that did not agree to their ban. It is likely that consumer products imported from these countries may still contain these chemicals. In February 2019, the EPA released its PFAS Action Plan in which the agency committed to make a "regulatory determination" under the Safe Drinking Water Act (SDWA) by the end of 2019, but the agency has not yet released the determination to the public. The EPA has released an update to their action plan in February 2020 (EPA, 2020c); however, the EPA stated they will continue to make progress on addressing SDWA concerns over the next year. Currently, the U.S. has no federally enforceable PFAS standard methods, leaving individual states to navigate various avenues for addressing PFAS contamination. Some states have established legally enforceable values for certain PFAS in drinking water, groundwater, surface water, soil, or other environmental media. Other states and regulatory agencies have opted for non-enforceable values such as guidance levels, screening numbers, or advisories that may apply to PFAS for which standard methods do not exist. The following section describes state-specific guidance for PFAS sampling and analysis in environmental media.

### 4.1. *The Environmental Council of the States*

The Environmental Council of the States (ECOS) is a national nonprofit, nonpartisan association of state and territorial environmental agency leaders based in Washington D.C. In 2019, ECOS compiled information on state PFAS standards, advisories, and guidance values ("guidelines"). ECOS and its working group of state environmental agency officials surveyed states on their processes, rulemaking requirements, and other considerations for establishing PFAS guidelines. They examined responses from 23 states and formulated a white paper to summarize their results (ECOS, 2021). No guidelines were presented in the ECOS paper that focused on PFAS sampling and analytical guidance. Conclusions from the white paper indicate that states are taking different stances on setting official PFAS guidance, in hopes that the EPA will establish a federally enforceable maximum contaminant level (MCL). Also, state regulators are looking for more transparency about the uses of existing PFAS, the ongoing development of new PFAS by industry, and PFAS approval by the EPA under statutes like the Toxic Substances Control Act (TSCA). Not all states responded to the survey so there are still unknowns around how each develops PFAS guidelines and how these compare to other states and federal agencies.

### *4.2. The Interstate Technology and Regulatory Council*

The Interstate Technology and Regulatory Council (ITRC) is a state-led coalition based in Washington D.C. with the goal of environmental sustainability through innovative solutions. In September 2020, ITRC established a PFAS technical and regulatory guidance document to support state and federal environmental staff, as well as stakeholders, project managers, and decision makers, gaining a working knowledge of the current state of PFAS science and practice (ITRC, 2020). The ITRC document was developed by a team of over 400 environmental practitioners drawn from state and federal government, academia, industry, environmental consulting, and public interest groups. The ITRC document covers information on PFAS physico-chemical properties, environmental fate and transport processes, environmental matrix-specific occurrence, human and ecological health effects, site risk assessment and characterization, regulations, sampling and analytical methods, treatment technologies, and stakeholder perspectives.

For PFAS sampling, the consensus from the ITRC document is that current state guidance and procedures should remain the basis for a PFAS sampling protocol. Method modifications and special considerations for PFAS sampling include (1) using unique sampling equipment or materials depending on the amount of background PFAS present, (2) field and equipment blanks above what is normally required, (3) the need for low laboratory quantification limits, (4) low state and federal screening levels, and (5) the need for decontamination and cleanup measures. Specific PFAS sampling information related to sampling equipment and supplies, collection bottle selection, sample preservation, shipping, storage and hold times, and sample decontamination can be found in the ITRC document. These recommendations are based on guidance supplied by EPA standard methods EPA 533 and EPA 537.1. For PFAS analysis, the ITRC document recommends multilaboratory-validated and published methods, specifically EPA standard methods 533 and 537.1. Information is provided on guidance for sample preparation, analytical instrumentation, standard preparation and storage, and instrumentation calibration. Data gaps and research needs identified by the ITRC document include a lack of human half-lives and toxicokinetic data for some PFAS in drinking water and other environmental matrices, a lack of information on the formation and potential toxicity of reactive PFAS precursors, and a lack of information on state-specific guidance for sampling and analysis of PFAS.

### *4.3. State-Specific Sampling and Analytical Guidance*

While coalitions like ECOS and ITRC have been developed to assess sampling and analytical guidance for PFAS, there is currently no comprehensive assessment of PFAS sampling and analytical guidance across all 50 U.S. states. After accessing state environmental department websites and discussing PFAS sampling and analytical guidance with state representatives via phone or email, we were able to compile a complete list of state-specific PFAS sampling and analytical guidance.

Figure 5 encompasses PFAS sampling guidelines for all states across ten environmental media. States follow up to three guidelines, (1) EPA 537.1 standard method, (2) ITRC sampling guidelines, (3) or state-developed guidelines. There are 31 states that are actively sampling for PFAS using one of these three sampling guidelines. Of the remaining 19 states that do not actively sample for PFAS, 13 states (AL, AR, KS, LA, MO, MS, NE, OK, OR, RI, SD, UT, WY) recommend EPA 537.1 as a PFAS sampling guideline. Two states (IA, ID) recommend ITRC as a PFAS sampling guideline. Four states (CO, MD, WA, WI) recommend Michigan state sampling guidelines as the preferred PFAS sampling guide to use when sampling for PFAS. States that do not actively sample for PFAS will refer these guidelines to organizations within the state that are sampling for PFAS. The data from Figure 5 shows that there is currently no state sampling for PFAS in air. This is likely due to the EPA not having a standard method for detecting PFAS in ambient or source air environments. It is also important to note that 26 states only sample or refer to sampling guidelines for drinking water and no other environmental matrix. This is likely due to the lack of standard methods available for non-potable water and environmental media. This compilation of data will likely change after 2021, when the EPA has completed a multi-laboratory assessment of their emerging standard methods and makes them available for public use.

States have also begun to develop their own general maximum contaminant levels (MCLs) for individual PFAS in drinking water. In 2020, New York Department of Health issued an MCL of 10 ng/L for PFOA and PFOS (NYDH, 2020), while Vermont issued MCLs of 20 ng/L for five PFAS (Vermont DEC, 2020). In 2021, the Pennsylvania Department of Environmental Protection issued MCL's for seven PFAS that ranged from 6 – 108 ng/L (Pennsylvania DEP, 2021). Other states have begun implementing state-specific regulations of PFAS while waiting for federal regulations to become enforced.

### 4.4. *International Perceptions on PFAS Sampling and Guidance*

In 2019, more than 180 countries agreed to phase out and ban production and use of PFOA and PFOS under the Stockholm Convention on Persistent Environmental Pollutants, meaning that there is a global awareness of the dangers of PFAS on global health and environment. Some international countries have also taken an active approach to reducing the global PFAS footprint. In 2018 Canada set a drinking water guideline for PFOS (600 ng/L) and PFOA (200 ng/L) (Canada, 2018). Australia has set drinking water health limits for drinking water (70 ng/L PFOS and 560 ng/L PFOA), recreational water (700 ng/L PFOS and 5.6 mg/L PFOA), and ecological protection risk values at 230 ng/L for PFOS and 19 µg/L for PFOA (NSW Health, 2017). The European Union has PFOS, PFOA and PFHxS restrictions. Denmark and Sweden have drinking water guidance values for over 11 PFAS, while Germany guidance values for PFOS range between 100 and 500 ng/L depending on general population. The U.K. PFOS drinking water guideline is 100 ng/L. International challenges of PFAS regulations include evolving information of environmental and human impacts of individual PFAS compounds and PFAS as a group, and a lack of resources to implement and monitor PFAS regulations and policies on a large scale (European Environment

Agency, 2019). Additionally, the ASTM and ISO standard sampling and analytical methods are internationally accepted methods for PFAS sampling and analysis.

## 5. Conclusions

To date, ten federal guidance documents are available for PFAS sampling guidance and analysis. The maximum number of PFAS covered in these guidance documents is 25 analytes spanning across 8 unique media. These 25 analytes only cover a small fraction of PFAS subclasses and are unlikely indicative of the total amount of PFAS present in contaminated regions (Nakayama et al. 2019; Ahrens and Bundschuh, 2014). While the EPA has developed several different sampling and analytical guidelines for PFAS and is on the verge of introducing a handful of new guidance documents this next year, there is no formal regulation of PFAS or requirements of states to enforce these guidelines. As a result, only 31 states have informally adopted sampling guidelines, while the other 19 states have no guidance documentation in place for PFAS. Additionally, some states have taken a more proactive approach (e.g., MI, CA, NY) in developing state guidelines that other states have begun recommending to organizations within their own state. The introduction of new PFAS sampling guidelines by the EPA, as well as updated analytical guidelines that target more PFAS or total organofluoride, is expected to continuously shift the landscape of federal and state guidance for PFAS sampling in the coming years.

## 6. Acknowledgments

This material is based upon work supported by the U.S. Department of Energy, Office of Environmental Management Technology Development Program. Funding for this material was provided by Kurt Gerdes.

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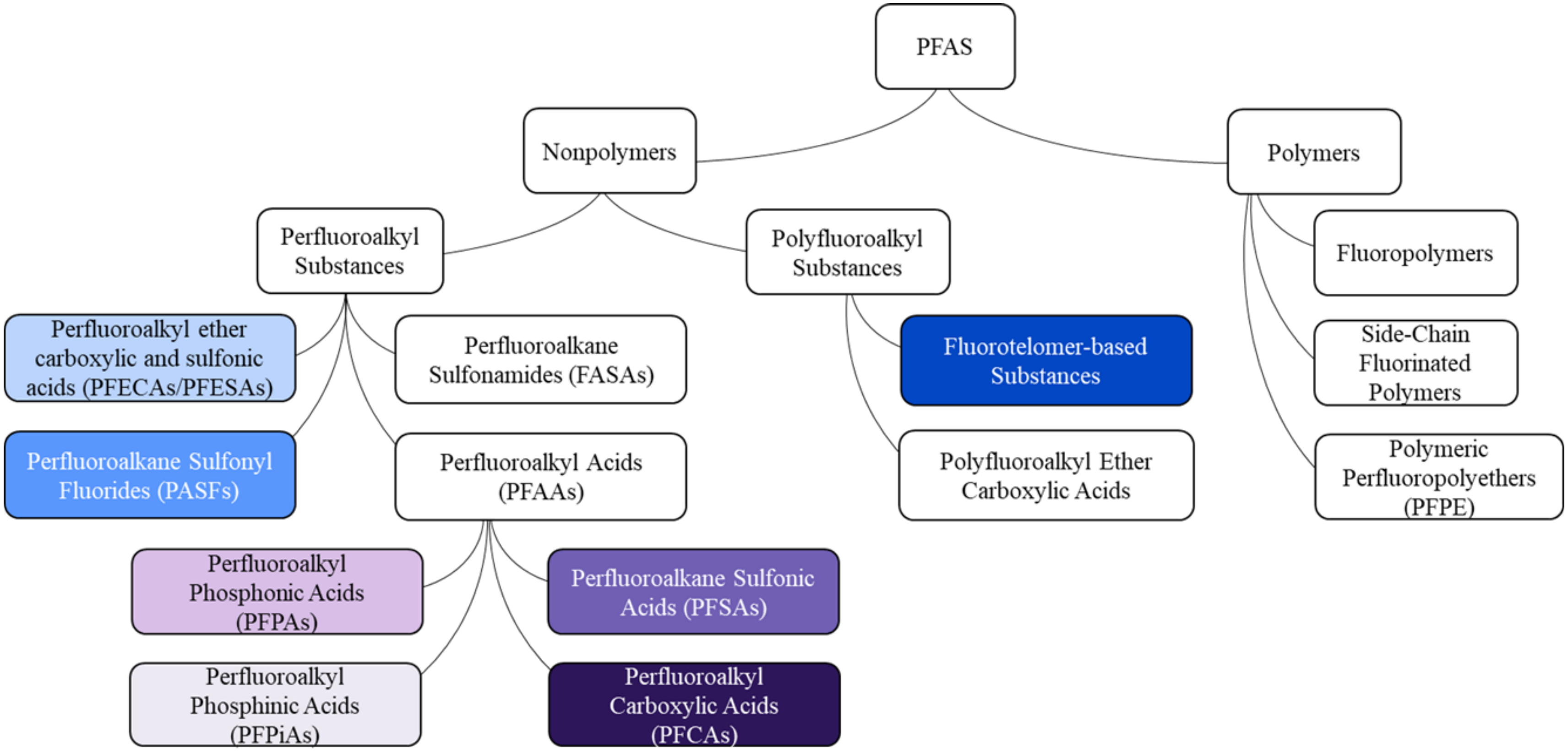


**Figure 1**. Hierarchy Tree for PFAS Family of Chemicals. The colored boxes indicate the most common PFAS used in manufacturing and industry. The color of each box correlates with the Figure 2 bar chart color scheme.

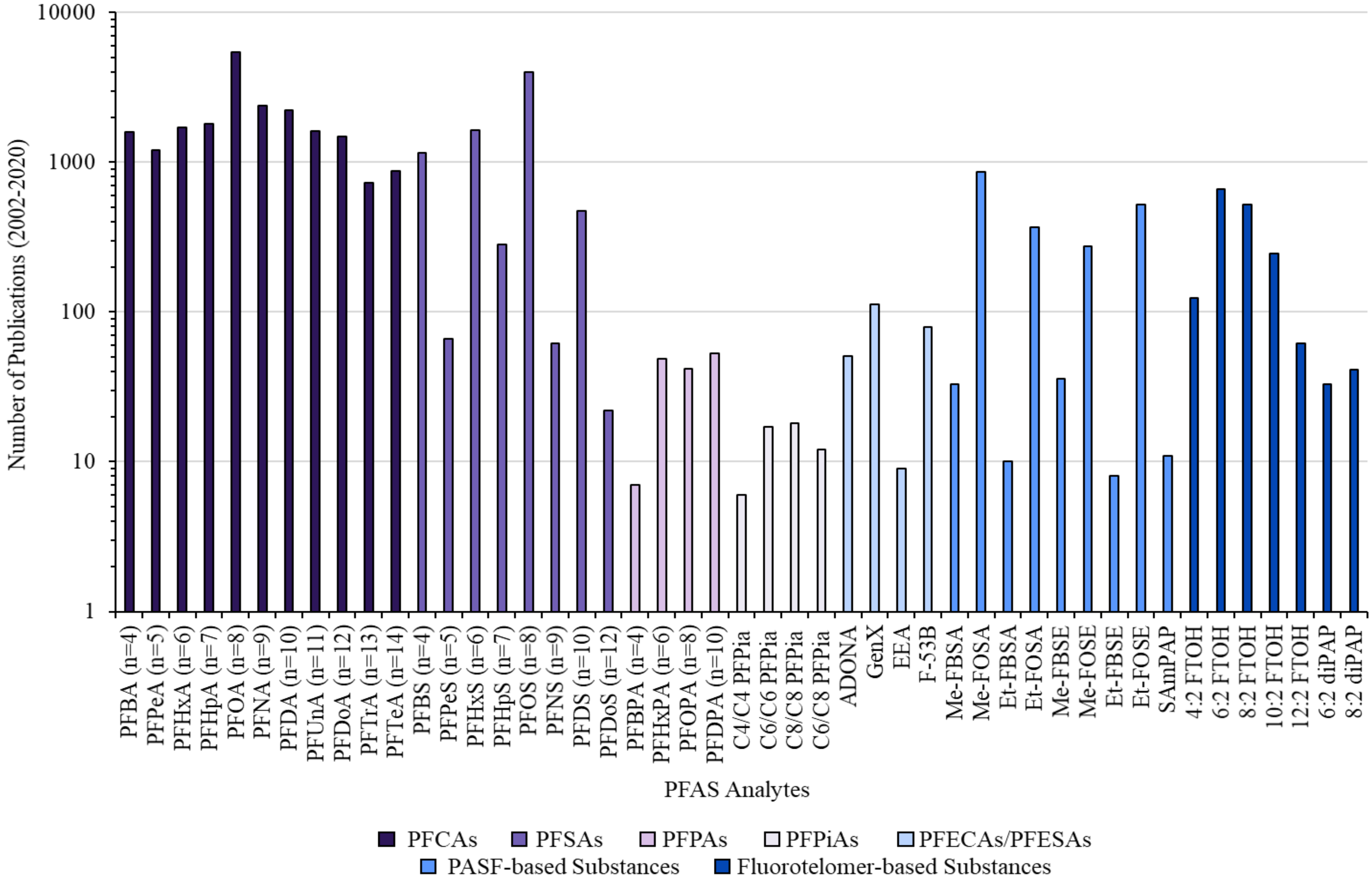


**Figure 2**. Number of journal publications of PFAS since 2002. The “n” refers to the carbon chain length of the PFAS. The y-axis is in log scale. The number of publications (related to all aspects of research) were retrieved from SciFinder on Dec. 15, 2021. PFAS full chemical names are provided in the SI.

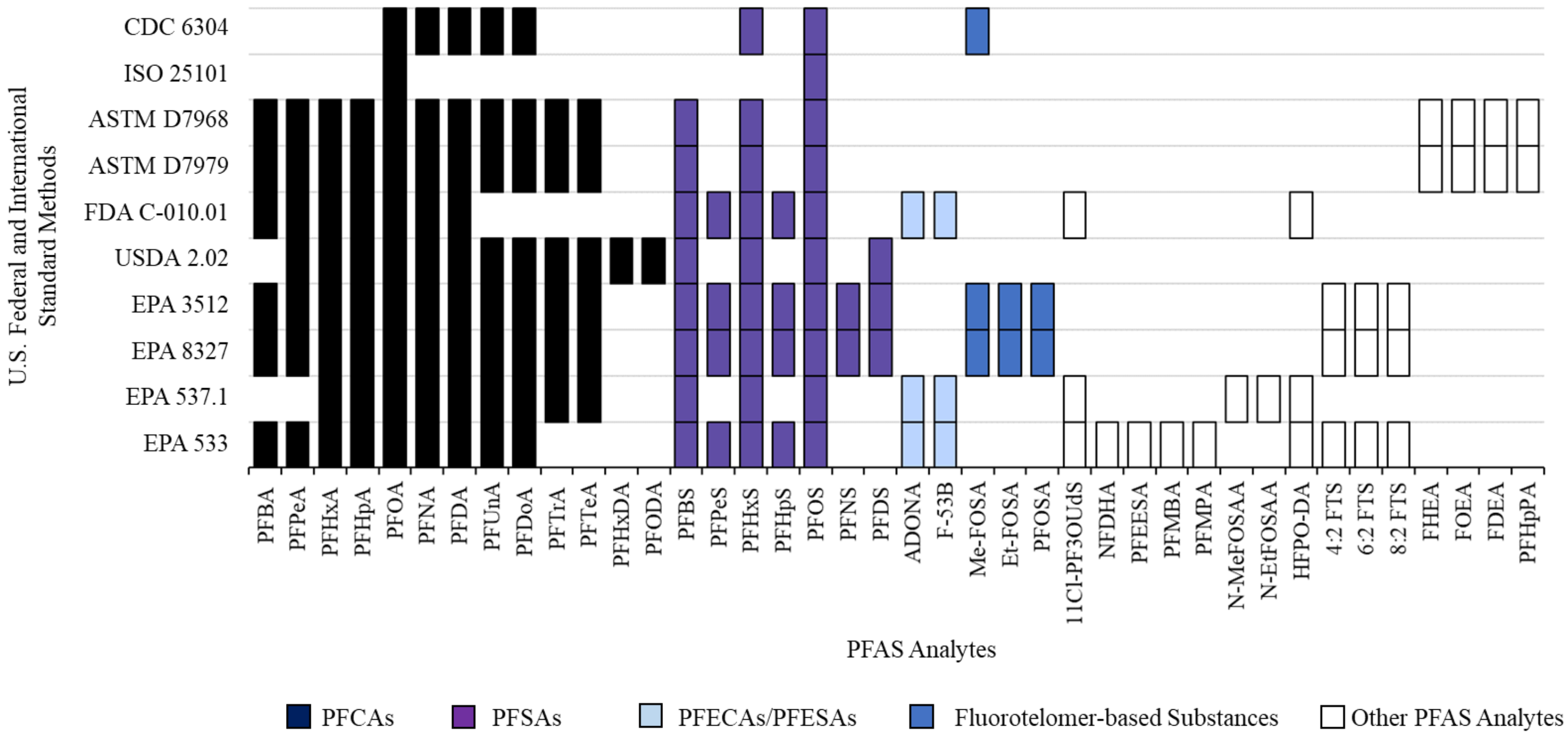


**Figure 3**. The PFAS that are analyzed in each U.S. government- and International Agency-approved standard method. The color schematic is representative of different subclasses of PFAS. Standard approved methods do not analyze for PFAS in PFPA, PFPiA, and PASF-based substance subclasses. PFAS full chemical names provided in the SI.

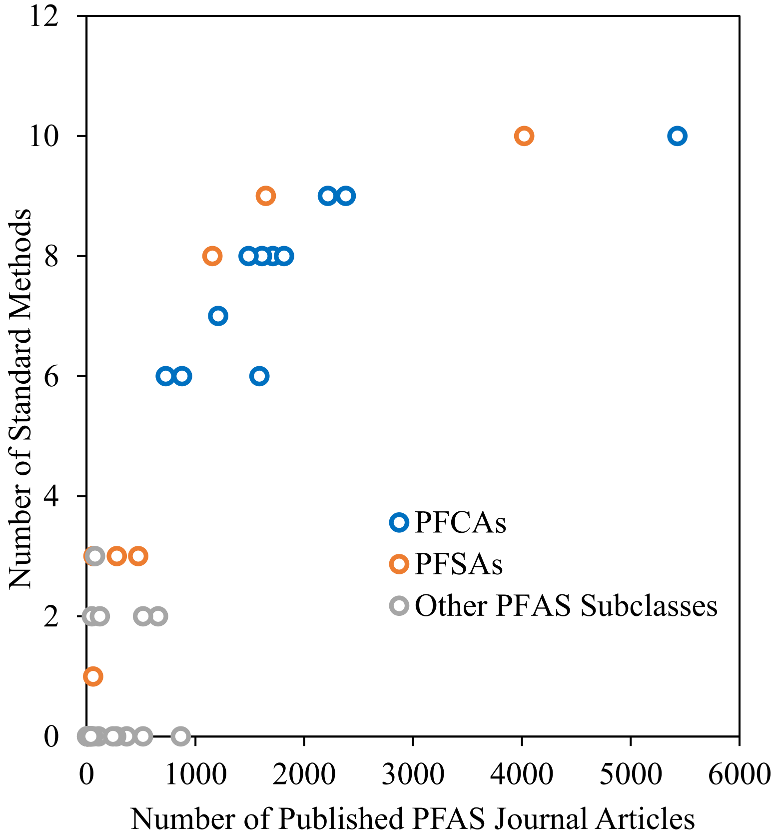
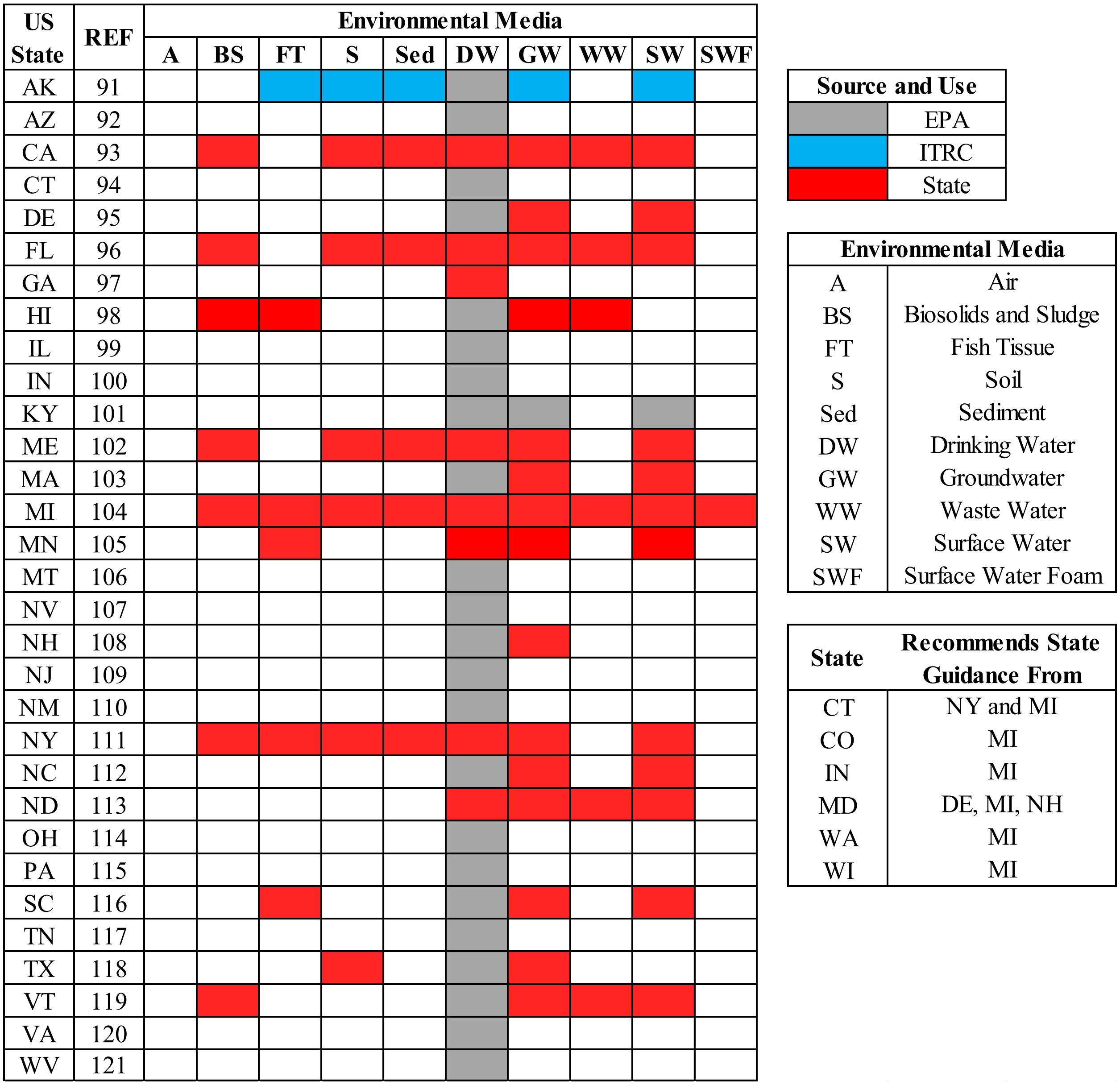


**Figure 4**. Comparison of the number of published PFAS journal articles versus the number of standard methods available for each PFAS. The number of publications (related to all aspects of research) were retrieved from SciFinder.



| US State | REF | Environmental Media | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | A | BS | FT | S | Sed | DW | GW | WW | SW | SWF |
| AK | 91 | | | 🟦 | 🟦 | 🟦 | ▨ | 🟦 | | 🟦 | |
| AZ | 92 | | | | | | ▨ | | | | |
| CA | 93 | | 🟥 | | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 | |
| CT | 94 | | | | | | ▨ | | | | |
| DE | 95 | | | | | | ▨ | 🟥 | | 🟥 | |
| FL | 96 | | 🟥 | | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 | |
| GA | 97 | | | | | | 🟥 | | | | |
| HI | 98 | | 🟥 | 🟥 | | | ▨ | 🟥 | 🟥 | | |
| IL | 99 | | | | | | ▨ | | | | |
| IN | 100 | | | | | | ▨ | | | | |
| KY | 101 | | | | | | ▨ | ▨ | | ▨ | |
| ME | 102 | | 🟥 | | 🟥 | 🟥 | 🟥 | 🟥 | | 🟥 | |
| MA | 103 | | | | | | ▨ | 🟥 | | 🟥 | |
| MI | 104 | | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 |
| MN | 105 | | | 🟥 | | | 🟥 | 🟥 | | 🟥 | |
| MT | 106 | | | | | | ▨ | | | | |
| NV | 107 | | | | | | ▨ | | | | |
| NH | 108 | | | | | | ▨ | 🟥 | | | |
| NJ | 109 | | | | | | ▨ | | | | |
| NM | 110 | | | | | | ▨ | | | | |
| NY | 111 | | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 | 🟥 | | 🟥 | |
| NC | 112 | | | | | | ▨ | 🟥 | | 🟥 | |
| ND | 113 | | | | | | 🟥 | 🟥 | 🟥 | 🟥 | |
| OH | 114 | | | | | | ▨ | | | | |
| PA | 115 | | | | | | ▨ | | | | |
| SC | 116 | | | 🟥 | | | ▨ | 🟥 | | 🟥 | |
| TN | 117 | | | | | | ▨ | | | | |
| TX | 118 | | | | 🟥 | | ▨ | 🟥 | | | |
| VT | 119 | | 🟥 | | | | ▨ | 🟥 | 🟥 | 🟥 | |
| VA | 120 | | | | | | ▨ | | | | |
| WV | 121 | | | | | | ▨ | | | | |

| Source and Use | |
|---|---|
| ▨ | EPA |
| 🟦 | ITRC |
| 🟥 | State |

| Environmental Media | |
|---|---|
| A | Air |
| BS | Biosolids and Sludge |
| FT | Fish Tissue |
| S | Soil |
| Sed | Sediment |
| DW | Drinking Water |
| GW | Groundwater |
| WW | Waste Water |
| SW | Surface Water |
| SWF | Surface Water Foam |

| State | Recommends State Guidance From |
|---|---|
| CT | NY and MI |
| CO | MI |
| IN | MI |
| MD | DE, MI, NH |
| WA | MI |
| WI | MI |

**Figure 5**. Per- and Poly-fluoroalkyl Substance (PFAS) Environmental Sampling Guidance for U.S. States. EPA guidelines are denoted black, Interstate Technology and Regulatory Council (ITRC) guidelines are denoted blue, and state guidelines are denoted red. The following states are not in the figure as they do not actively sample for PFAS currently: AL, AR, CO, ID, IA, KS, LA, MD, MS, MO, NE, OK, OR, RI, SD, UT, WA, WI, and WY.

**Table 1**. U.S. Government - and International Organization-approved PFAS sampling and analytical methods.

| Ref | Entity | Method | Environmental Matrices | Analytical Method | Number of PFAS |
|---|---|---|---|---|---|
| 122 | EPA | 533 | Drinking Water | SPE-LC-MS/MS | 25 |
| 123 | EPA | 537.1 | Drinking Water | SPE-LC-MS/MS | 18 |
| 124 | EPA | 8327 | Non-Potable Water | MRM-LC-MS/MS | 24 |
| 125 | EPA | 3512 | Non-Potable Water | LC-MS/MS | 24 |
| 126 | USDA | CLG 2.03 | Animal Muscle and Plasma | UPLC-MS/MS | 16 |
| 127 | FDA | C-010.02 | Food | LC-MS/MS | 16 |
| 128 | ASTM | D7979-17 | Water, Sludge, Influent, Effluent, and Wastewater | LC-MS/MS | 21 |
| 129 | ASTM | D7968-17a | Soil | LC-MS/MS | 21 |
| 130 | ISO | 25101:2009 | Unfiltered Drinking Water, Groundwater, Surface Water | HPLC-MS/MS | 2 |
| 131 | CDC | 6304.08 | Serum | SPE-HPLC-TIS-MS/MS | 8 |

Note: SPE = online solid phase extraction. MRM = multiple reaction monitoring. UPLC = ultra-performance liquid chromatography. HPLC = high-performance liquid chromatography. TIS = turbo ion spray. LC/MS/MS = liquid chromatography with tandem mass spectrometry. USDA = United States Department of Agriculture. FDA = United States Food and Drug Administration. ASTM = American Society for Testing and Materials. ISO = International Organization for Standardization. CDC = Center for Disease Control and Prevention.

**Table 2. PFAS Sampling Guidance for approved PFAS Sampling and Analytical Methods**

| PFAS Standard Method | PFAS Sampling Guidance | | | | | | |
|---|---|---|---|---|---|---|---|
| | PPE | Sample Collection | Sample Preparation | Sample Decontamination | Sample Shipment | Sample Storage | Sample Holding Time |
| CDC 6304 | Standard PPE | Polypropylene Container | - | - | Ice | 4°C | - |
| ISO 25101 | Standard PPE | Polypropylene Container | Sodium sulfate | - | - | 4°C | 2 Weeks |
| ASTM D7968-17a | - | Polypropylene Container | - | EPA SW-846 Guide | Ice | 0°C - 6°C | 4 Weeks |
| ASSTM D7979-17 | - | Polypropylene Container | - | EPA SW-846 Guide | Ice | 0°C - 6°C | 4 Weeks |
| FDA C-010.02 | - | Polypropylene Container | Sample Homogenization | - | - | - | - |
| USDA CLG 2.03 | - | Polypropylene Container | Sample Homogenization | - | - | - | - |
| EPA 3512 | Standard PPE | Polypropylene Container | Samples diluted, filtered, and pH adjusted | Detergent | - | 0°C - 6°C | 4 Weeks |
| EPA 8327 | Standard PPE | Polypropylene Container | Samples diluted, filtered, and pH adjusted | Detergent | Ice | 0°C - 6°C | 4 Weeks |
| EPA 537.1 | Nitrile Gloves | Polypropylene Container | Trizma reagent | PFAS free Containers and LC Tubing | Below 10°C | 0°C - 6°C | 4 Weeks |
| EPA 533 | Nitrile Gloves | Polypropylene Container | Ammonium acetate | PFAS free Containers and LC Tubing | Below 10°C | 0°C - 6°C | 4 Weeks |

**Table 3. PFAS Detection Limits for approved PFAS Sampling and Analytical Methods**

| PFAS Analytes | Limit of Detection / Lower Limits of Quantitation | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | CDC 6304.08 ng/mL | ISO 25101 ng/mL | ASTM D7968 ng/kg | ASTM D7979 ng/L | FDA C-010.02 ng/kg | USDA CLG 2.03 ng/g | EPA 3512 ng/L | EPA 8327 ng/L | EPA 537.1 ng/L | EPA 533 ng/L |
| PFBA | | | 22.0 | 4.6 | 31 - 67 | | 10 | 10 | | 13 |
| PFPeA | | | 20.9 | 4.6 | 17 - 31 | 0.5 | 10 | 10 | | 3.9 |
| PFHxA | | | 15.4 | 2 | 18 - 48 | 0.5 | 10 | 10 | 1 | 5.3 |
| PFHpA | | | 5.8 | 1 | 11 - 43 | 0.5 | 10 | 10 | 0.7 | 2.6 |
| PFOA | 1.0 | 10.0 | 6.2 | 1.7 | 12 - 24 | 0.5 | 10 | 10 | 0.53 | 3.4 |
| PFNA | 1.0 | | 2.8 | 1.1 | 7 - 29 | 0.5 | 10 | 10 | 0.7 | 4.8 |
| PFDA | 1.0 | | 5.5 | 1.4 | 12 - 22 | 0.5 | 10 | 10 | 1.6 | 2.3 |
| PFUnA | 1.0 | | 2.5 | 1.2 | | 0.5 - 1.3 | 10 | 10 | 1.6 | 2.7 |
| PFDoA | 1.0 | | 3.6 | 1.2 | | 0.5 | 10 | 10 | 1.2 | 2.2 |
| PFTrA | | | 5.3 | 0.7 | | 0.5 - 1.3 | 15 | 15 | 0.7 | |
| PFTeA | | | 6.8 | 1.2 | | 0.5 - 1.3 | 10 | 10 | 1.1 | |
| PFHxDA | | | | | | 1.3 - 12.5 | | | | |
| PFODA | | | | | | 0.5 - 1.3 | | | | |
| PFBS | | | 6.5 | 0.8 | 3 - 21 | 0.5 | 10 | 10 | 1.8 | 3.5 |
| PFPeS | | | | | 15 - 28 | | 10 | 10 | | 6.3 |
| PFHxS | 1.0 | | 7.8 | 1,2 | 13 - 35 | 0.5 - 12.5 | 10 | 10 | 1.4 | 3.7 |
| PFHpS | | | | | 20 - 36 | | 10 | 10 | | 5.1 |
| PFOS | 6.0 | 2.0 | 18.8 | 2.2 | 7 - 28 | 0.5 | 10 | 10 | 1.1 | 4.4 |
| PFNS | | | | | | | 10 | 10 | | |
| PFDS | | | | | | 0.5 - 1.3 | 10 | 10 | | |
| ADONA | | | | | 7 - 25 | | | | 0.9 | 3.4 |
| F-53B | | | | | 6 - 20 | | | | 1.4 | 1.4 |
| Me-FOSA | 1.0 | | | | | | 15 | 15 | | |
| Et-FOSA | | | | | | | 15 | 15 | | |
| PFOSA | | | | | | | 10 | 10 | | |
| 11Cl-PF3OUdS | | | | | 3 - 17 | | | | 1.5 | 1.6 |
| NFDHA | | | | | | | | | | 16 |
| PFEESA | | | | | | | | | | 2.6 |
| PFMBA | | | | | | | | | | 3.7 |
| PFMPA | | | | | | | | | | 3.8 |
| N-MeFOSAA | | | | | | | | | 2.4 | |
| N-EtFOSAA | | | | | | | | | 2.8 | |
| HFPO-DA | | | | | 10 - 34 | | | | 1.9 | 3.7 |
| 4:2 FTS | | | | | | | 10 | 10 | | 4.7 |
| 6:2 FTS | | | | | | | 60 | 60 | | 14 |
| 8:2 FTS | | | | | | | 15 | 15 | | 9.1 |
| FHEA | | | 199.0 | 92.9 | | | | | | |
| FOEA | | | 258.4 | 106.8 | | | | | | |
| FDEA | | | 137.5 | 47.2 | | | | | | |
| PFHpPA | | | 5.1 | 3.3 | | | | | | |

**Table 4**. Standard Methods in Development for PFAS Analysis by the EPA

| EPA Standard Methods in Development[132] | |
|---|---|
| **Standard Method** | **Environmental Matrices** |
| 8328 | Non-drinking water, Soil, Sediment, Solid Waste |
| 1633 | Aqueous, Solid, Biosolid, Tissue |
| OTM-45 | Source (Air) Emissions |
| TO-15 | Source (Air) Emissions |
| Near-Source | Ambient Air |
| Semivolatile | Ambient Air |
| Volatile | Ambient Air |
| Total Organic Fluorine | Environmental Media |
| Total Organic Precursors | Environmental Media |