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SYSTEMATIC ASSESSMENT OF ANALYTICAL TECHNIQUES FOR THE TRACE-LEVEL DETECTION OF PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS) IN COMPLEX ENVIRONMENTAL AND BIOLOGICAL MATRICES

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ABSTRACT

Per- and polyfluoroalkyl substances (PFAS) are globally recognised as persistent organic pollutants of substantial environmental and public health concern as a result of their toxicity, bioaccumulation, and chemical stability. The detection of PFAS at trace and ultra-trace levels in complex matrices remains an analytical challenge due to the presence of numerous unknown analogues and the diverse physicochemical properties of PFAS. This systematic review critically evaluates 55 peer-reviewed studies published between 2010 and 2025 that focus on analytical techniques for quantifying and identifying PFAS in environmental and biological samples. The assessment follows the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) guidelines and compares the performance of major analytical platforms, including liquid chromatography tandem mass spectrometry (LC–MS/MS), ultra-high-performance liquid chromatography high-resolution mass spectrometry (UHPLC–HRMS), gas chromatography mass spectrometry (GC–MS), extractable/adsorbable organofluorine (EOF/AOF), surface-enhanced Raman spectroscopy (SERS), and electrochemical or hybrid sensor-based approaches. LC–MS/MS and UHPLC–HRMS remain the gold standards for quantitative and non-target PFAS analysis, respectively, while emerging spectroscopic and electrochemical methods offer rapid, portable, and cost-effective alternatives. Molecularly imprinted polymers, deep eutectic solvents, and bubble-assisted extraction are among the sample preparation advancements that have further enhanced environmental sustainability and sensitivity. The review highlights the need for standardized QA/QC protocols, certified reference materials, and inter-laboratory harmonization. Integration of artificial intelligence, green chemistry, and multi-platform detection systems represents the future direction for comprehensive PFAS monitoring and exposure assessment.

KEYWORDS: *Per- and Polyfluoroalkyl Substances, Mass Spectroscopy, Non-target analysis, Surface-Enhanced Raman Spectroscopy, Environmental monitoring.*

INTRODUCTION

Per- and polyfluoroalkyl substances (PFAS) constitute a substantial and chemically varied category of synthetic organofluorine compounds that have been widely utilized for more than seventy years in industrial, commercial, and consumer applications, such as non-stick coatings, firefighting foams, textiles, and food packaging. (Shen et al., 2024; Glüge et al., 2020). Their exceptional thermal and chemical stability, stemming from strong C–F bonds, has resulted in extreme environmental persistence and bioaccumulation, earning them the moniker "forever chemicals." Widespread detection of PFAS in air, soil, water, and biota has raised global concerns over their toxicological and ecological effects (Nahar et al., 2023; Richardson & Manasfi, 2024). Increasing evidence links PFAS exposure to immunotoxicity, hepatotoxicity, endocrine disruption, and reproductive dysfunctions in both humans and wildlife (Lai et al., 2024; Pontius, 2019). Initial field surveys revealed the extensive presence of PFAS in major river basins and highlighted the analytical constraints of that period (e.g., targeted LC–MS/MS with restricted analyte lists and detection limits in the low ng L^{-1} range), emphasizing the necessity for method enhancement and standardized sampling strategies (Möller et al., 2010).

Measuring PFAS at trace and ultra-trace concentrations in environmental and biological matrices is a significant analytical challenge due to their chemical variety (over 15,000 structures registered), wide range of physicochemical properties, and the complexity of environmental samples (Tang et al., 2023; Megson et al., 2025). Analytical difficulties are made worse by the absence of authentic standards for emerging PFAS and the occurrence of matrix effects that undermine extraction efficiency and instrument sensitivity (Perovani et al., 2023; Liu et al., 2024). Conventional targeted approaches mostly rely on liquid chromatography in conjunction with tandem mass spectrometry (LC–MS/MS), provides excellent quantification for legacy PFAS such as perfluorooctanoic acid

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(PFOA) and perfluorooctanesulfonic acid (PFOS), but are limited in detecting novel or ultra-short-chain compounds (Mulabagal et al., 2018; Sadia et al., 2020).

In recent years, high-resolution mass spectrometry (HRMS) has transformed PFAS detection through suspect and non-target screening (NTS), facilitating the discovery of previously unrecognized PFAS species (Getzinger et al., 2021; Strynar et al., 2023). Complementary techniques including extractable and adsorbable organofluorine (EOF/AOF) analysis provide aggregate fluorine measurements that bridge the gap between targeted quantification and total fluorine content, highlighting the presence of uncharacterized PFAS (Pan, 2023; Salameh, 2021). Moreover, advancements in sample preparation techniques such as microextraction, solid-phase extraction (SPE), and molecularly imprinted polymers (MIPs) have significantly enhanced analytical recovery and selectivity in complex matrices (Jalili et al., 2023; Fan et al., 2021). Novel sensing platforms based on surface-enhanced Raman spectroscopy (SERS), electrochemical transducers, metal–organic frameworks (MOFs), and covalent organic frameworks (COFs) further offer potential for on-site and real-time PFAS detection at femtomolar concentrations (Lu et al., 2025; Pitruzzella et al., 2024; Huo et al., 2025).

Despite these technical advancements, a comprehensive and systematic assessment of analytical methodologies for PFAS detection across environmental and biological matrices is still constrained. Prior reviews have primarily focused on individual classes of methods (e.g., mass spectrometry, sensor-based platforms, or sample preparation), without cross-comparing their performance metrics, detection limits, reproducibility, and matrix applicability (Nahar et al., 2023; Jalili et al., 2023). Regional syntheses underscored both analytical advancements and significant information deficiencies regarding PFAS occurrence and transformation pathways, emphasizing the necessity for rigorous cross-method comparisons (Cousins, 2013). A holistic assessment is therefore needed to benchmark analytical techniques, identify methodological gaps, and guide future standardization of PFAS monitoring strategies.

This systematic review critically assesses current analytical approaches for the trace-level detection of PFAS in complex environmental and biological matrices. Drawing on a curated selection of fifty peer-reviewed publications from 2010 to 2025, it evaluates the sensitivity, selectivity, and applicability of major analytical platforms including LC–MS/MS, UHPLC–HRMS, GC–MS, EOF/AOF, SERS, electrochemical and nanopore sensors alongside emerging computational and chemometric workflows. The review also highlights best practices in sample preparation, QA/QC protocols, and fluorine mass balance approaches, with the overarching goal of informing future analytical frameworks and harmonized monitoring of PFAS contamination worldwide.

2. METHODOLOGY

2.1 Literature Search Strategy

This systematic review was conducted in accordance with the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) 2020 guidelines (Page et al., 2021). A comprehensive literature search was performed from articles published between 2010- 2025 across multiple scientific databases, including Web of Science, Scopus, PubMed, ScienceDirect, and Google Scholar**,** to identify peer-reviewed articles related to analytical techniques for PFAS detection.

The following Boolean keyword combinations were used:
("per- and polyfluoroalkyl substances" OR "PFAS" OR "PFOA" OR "PFOS") AND ("analytical technique" OR "analytical method" OR "detection" OR "quantification" OR "determination") AND ("trace-level" OR "ultratrace" OR "sensitive") AND ("environmental matrices" OR "biological matrices" OR "water" OR "soil" OR "sediment" OR "biota" OR "plasma" OR "tissue"). Reference lists of retrieved articles and relevant reviews were also screened manually to ensure inclusion of additional key publications. Grey literature (theses, preprints, and conference proceedings) was selectively considered if it provided substantial methodological innovation or data unavailable in published journals.

2.2 Data Extraction and Classification

Data were systematically extracted using a standardized template developed in Microsoft Excel. For each study, the following variables were recorded:

- **Analytical platform** (e.g., LC–MS/MS, UHPLC–HRMS, GC–MS, SERS, sensor, EOF/AOF).
- **Sample type and matrix** (e.g., environmental water, biota, plasma, soil, air).
- **Sample preparation technique** (e.g., SPE, LLE, MIP, microextraction, MOF/COF enrichment).
- **Detection and quantification limits (LOD/LOQ)**, precision (%RSD), and recovery (%) were reported.
- **Quality assurance/quality control (QA/QC)** measures such as blanks, calibration range, matrix spikes, and internal standards.
- **Scope** (targeted, suspect, or non-target screening).
- **Analytical performance metrics** (sensitivity, selectivity, reproducibility, throughput, matrix robustness).

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2.3 Data Synthesis and Comparative Evaluation

Extracted data were synthesized both qualitatively and quantitatively. Analytical methods were grouped into seven main categories *viz* Liquid chromatography–tandem mass spectrometry (LC–MS/MS), Ultra-high-performance liquid chromatography–high-resolution mass spectrometry (UHPLC–HRMS), Gas chromatography–mass spectrometry (GC–MS), Extractable/adsorbable organofluorine (EOF/AOF) methods, Surface-enhanced Raman spectroscopy (SERS), Electrochemical and optical sensor-based approaches, Emerging hybrid/nanostructured platforms (e.g., MOF/COF-based, nanopore, and photothermal devices)

Within each category, comparative evaluation focused on analytical sensitivity, matrix applicability, selectivity, detection limits, throughput, and reproducibility. Where multiple studies used similar matrices and instruments, average LODs and recoveries were computed to identify performance trends. Quantitative synthesis emphasized trace-level (<1 ng L^{-1} or <1 ng g^{-1}) detection capabilities across matrices.

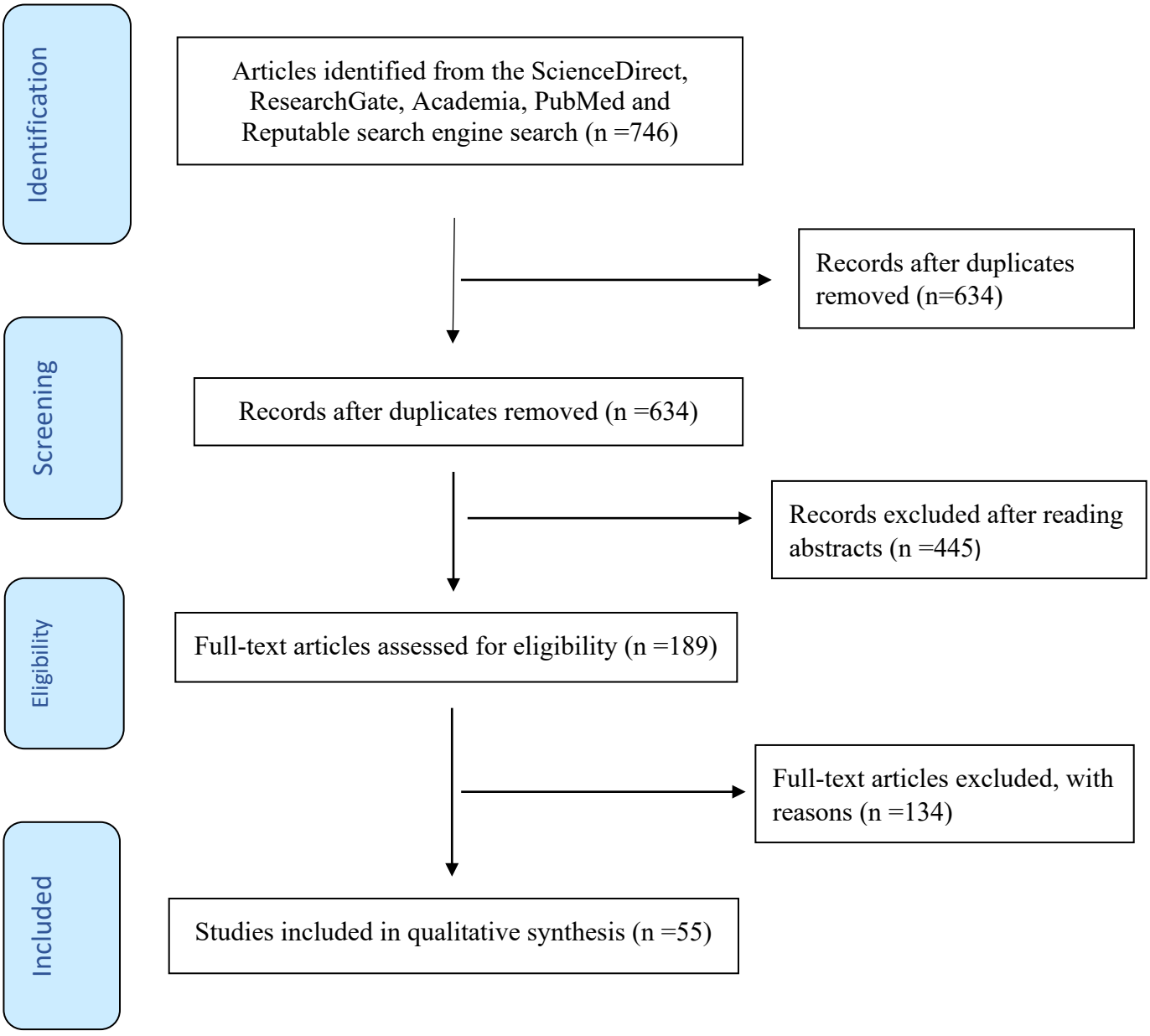


Figure 1: PRISMA Flow diagram showing the article selection process in the study.

Sources; Author's Construct 2020.

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3. RESULTS AND DISCUSSION

3.1. Overview of Analytical Techniques for PFAS Detection

Over the past decade, analytical approaches for detecting per- and polyfluoroalkyl substances (PFAS) have significantly advanced, focusing on enhancing sensitivity, selectivity, and matrix tolerance for trace and ultra-trace-level analysis. **Table 1** summarizes the main analytical platforms and their performance characteristics across various environmental and biological matrices. Passive air samplers show the ability to gather a complementary PFAS profile compared to active samplers and facilitate lower-effort long-term sampling; nevertheless, calibration and absorption rate adjustments are necessary for quantitative applications (Ahrens et al., 2013).

Table 1 demonstrates that LC–MS/MS and UHPLC–HRMS dominate PFAS analytical workflows due to their reproducibility and ability to detect a wide range of target and emerging compounds. LC–MS/MS remains the gold-standard for quantitative measurements, achieving detection limits between 0.05 and 1 ng L^{-1}**,** depending on matrix complexity (Mulabagal et al., 2018; Sadia et al., 2020; Liu et al., 2024). Its high selectivity and compatibility with isotope-labeled standards make it ideal for regulatory monitoring (EPA Methods 533 and 537.1). However, the reliance on established standards limits its applicability for newly discovered or short-chain PFAS, many of which do not have reference materials.

By contrast, UHPLC–HRMS extends analytical coverage through suspect and non-target screening (NTS), detecting hundreds of uncharacterized PFAS species across wastewater, biota, and atmospheric particulates (Getzinger et al., 2021; Strynar et al., 2023). Although HRMS methods typically have slightly higher detection limits (0.1–10 ng L^{-1}), they enable high-confidence structural elucidation using accurate-mass data and isotope-pattern recognition. Retrospective data mining and computational tools such as retention-time prediction and in-silico spectral matching further enhance the identification of unknown PFAS (Lai et al., 2024; Simpson et al., 2022).

3.1.1 Gas chromatography–mass spectrometry (GC–MS), although less frequently used, it offers complementary insights for volatile and neutral precursors, including fluorotelomer alcohols (FTOHs) and perfluorinated ethers (Boiteux et al., 2012). ECNI and chemical ionization modes achieve sub-ng g^{-1} detection limits but require derivatization and risk thermal degradation of labile analytes.

3.1.2 Extractable and adsorbable organofluorine (EOF/AOF) methods serve a unique role in measuring total fluorine rather than individual PFAS species. As **Table 1** shows, typical detection limits of 1–20 ng F L^{-1} reveal that known PFAS represent only a fraction of total organofluorine mass (Pan, 2023; Salameh, 2021). Despite their lack of molecular specificity, these techniques are essential for evaluating the hidden "dark fluorine" burden and complement mass spectrometric analyses.

3.1.3 Emerging spectroscopic and electrochemical approaches are reshaping PFAS analytics. Surface-enhanced Raman spectroscopy (SERS)**,** for instance, enables label-free and rapid PFAS detection with LODs as low as 0.01 ng L^{-1} (Huo et al., 2025; Juhong et al., 2025). Advances in plasmonic substrates and deep-learning spectral classification have enhanced selectivity for PFOS and PFOA. Nevertheless, reproducibility remains a challenge due to surface heterogeneity and signal fluctuations.

3.1.4 Electrochemical sensors particularly those incorporating MOF, MXene, and COF nanocomposites are capable of sub-ng L^{-1} detection while providing portability and low-cost operation (Rezaei et al., 2024; Tao et al., 2025). Despite these advantages, their quantitative reliability still depends on calibration with LC–MS/MS or HRMS.

3.1.5 Hybrid and nanopore systems, which combine molecular recognition with high-precision electrical or optical transduction, represent the next frontier. Lu et al. (2025) reported multiplex nanopore detection of diverse PFAS structures at femtomolar concentrations, emphasizing its applicability for real-time environmental monitoring, albeit with limited throughput at present.

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Table 1. Summary of Major Analytical Techniques for PFAS Detection in Environmental and Biological Matrices

Analytical Technique	Typical Matrices	Detection Limit (LOD)*	Key Advantages	Major Limitations	Representative References
LC–MS/MS	Water, soil, serum, food	0.05–1 ng L^{-1}	High sensitivity; excellent quantification; standardized methods (EPA 533/537.1)	Requires standards; limited for unknown PFAS; matrix effects	Mulabagal et al. (2018); Sadia et al. (2020); Liu et al. (2024); Perovani et al. (2023)
UHPLC–HRMS (Orbitrap/QTOF)	Water, wastewater, air particulates, plasma	0.1–10 ng L^{-1}	Enables suspect/non-target screening; high resolution and accuracy; retrospective analysis	High cost; data complexity; variable quantitative precision	Strynar et al. (2023); Getzinger et al. (2021); Tang et al. (2023); Dudášová et al. (2025)
GC–MS / GC–MS/MS	Air, dust, soil, sediments	0.1–5 ng g^{-1}	Effective for volatile precursors (FTOHs, sulfonamides); well-established	Derivatization needed; thermal degradation risk	teux et al. (2012); Fang et al. (2020)
EOF/AOF (CIC, IC)	Water, sludge, sediment, food	1–20 ng F L^{-1}	Measures total organofluorine; reveals unidentified PFAS fraction	No compound-level speciation; potential overestimation	Pan (2023); Salameh (2021)
SERS	Water, food, plasma	0.01–0.5 ng L^{-1}	Rapid, label-free detection; minimal sample prep; potential on-site use	Reproducibility; substrate fouling	Huo et al. (2025); Juhong et al. (2025)
Electrochemical sensors (MIP/MOF/COF)	Water, biological fluids	0.01–0.1 ng L^{-1}	High selectivity; portable; low cost	Limited multiplexing; requires calibration	Rezaei et al. (2024); Tao et al. (2025); Pitruzzella et al. (2024)
Hybrid & nanopore systems	Surface water, wastewater	<0.01 ng L^{-1}	Real-time, multiplex detection; structural recognition	Still experimental; low throughput	Lu et al. (2025); Guo et al. (2025)

*LOD = Limit of Detection. Reported ranges reflect mean values from referenced studies.

3.2. Sample Preparation and Preconcentration Techniques

The accuracy and sensitivity of PFAS analysis are strongly influenced by the efficiency of sample preparation. The comparative optimization of extraction methods for solid matrices showed that the selection of method significantly affects the recovery and detection range of PFAS, emphasizing the necessity for standardizing pre-treatment across matrices (Lorenzo et al., 2015). **Table 2** summarizes the major extraction and enrichment methods and their analytical performance across different matrices.

3.2.1 Solid-phase extraction (SPE) remains the most employed method, providing recovery rates of 75–110 % for both water and biological samples (Jalili et al., 2023; Liu et al., 2024). Weak-anion-exchange (WAX) cartridges enhance PFAS retention, while automation and large-volume online SPE systems improve throughput (Miller, 2024). However, fluoropolymer contamination in cartridges and solvents can lead to background interferences.

3.2.2 Microextraction techniques such as dispersive liquid–liquid microextraction (DLLME) and single-drop microextraction (SDME) offer greener, solvent-efficient alternatives, particularly suitable for lipid-rich or proteinaceous matrices (Jalili et al., 2023).

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Although microextraction reduces solvent use by >90 %, recoveries may decrease in highly organic matrices due to phase incompatibility.

3.2.3 Molecularly imprinted polymers (MIPs) and **nanoporous frameworks (MOFs/COFs)** have gained traction due to their high selectivity and reusability. Studies report recoveries exceeding 115 %, attributed to the tunable pore chemistry and strong F–H or π–π interactions (Rezaei et al., 2024; Tao et al., 2025). Nevertheless, synthesis cost and cross-reactivity among structurally similar PFAS limit large-scale application.

The choice of sampler significantly influences the measured distribution of PFAS phases; comparative studies showed notable differences in gas–particle partitioning outcomes based on sampler type, highlighting the necessity to align sampling and analytical methodologies for trace PFAS analysis (Ahrens et al., 2011).

Innovative techniques such as deep eutectic solvent (DES)-based nanofluids (Fan et al., 2021) and bubble-assisted extraction (Elpa & Urban, 2024) exemplify the transition toward environmentally sustainable sample preparation. Both methods demonstrate extraction efficiencies above 80 % while eliminating toxic organic solvents. These developments reflect a growing shift toward green analytical chemistry in PFAS research.

Table 2. Comparison of Sample Preparation Techniques and Their Analytical Performance

Sample Preparation Method	Applicable Matrices	Extraction Efficiency (%)	Advantages	Limitations	Representative References
Solid-Phase Extraction (SPE)	Water, wastewater	75–110	Widely standardized; compatible with LC–MS/MS; good reproducibility	Labor intensive; cartridge contamination risk	Jalili et al. (2023); Perovani et al. (2023); Liu et al. (2024)
Microextraction (DLLME, SDME)	Plasma, soil, food	70–95	Low solvent use; high preconcentration; simple	Matrix interference at high organic content	Jalili et al. (2023)
Molecularly Imprinted Polymer (MIP) Extraction	Serum, milk, soil	80–115	High selectivity; reusable sorbents	Cross-reactivity with analogues	Pitruzzella et al. (2024); Rezaei et al. (2024)
Metal–Organic Framework (MOF) / Covalent Organic Framework (COF) Adsorption	Water, biological fluids	85–120	High surface area; tunable pore chemistry	Regeneration challenges; cost	Lu et al. (2025); Tao et al. (2025)
Deep Eutectic Solvent (DES) Nanofluid Extraction	Edible oils, fish tissue	80–105	Green solvent; rapid extraction	Requires viscosity control; limited automation	Fan et al. (2021)
Bubble-Assisted Extraction	Water, serum	78–98	Solvent-free; enhanced mass transfer	Scale-up limitations	Elpa & Urban (2024); Gao et al. (2023)
Large-Volume On-Line Injection	Water, wastewater	>95	Reduces handling bias; high throughput	Specialized hardware required	Miller (2024)

3.3. Comparative Evaluation and Emerging Analytical Trends

The integration of results from Tables 1 and 2 underscores clear trade-offs between sensitivity, selectivity, and operational practicality. LC–MS/MS and HRMS dominate laboratory-based quantification, while SERS, electrochemical, and nanopore platforms are emerging as field-deployable tools capable of near-real-time analysis. Despite impressive detection capabilities (<0.01 ng L^{-1} for advanced sensors), challenges persist in matrix standardization, inter-laboratory comparability, and quality control. The absence of certified reference materials for emerging PFAS remains a bottleneck, though recent efforts (Sommerfeld et al., 2025) are addressing this gap. Furthermore, machine learning and AI-driven data mining in HRMS datasets promise enhanced non-target identification and pattern recognition, signaling a convergence between analytical chemistry and computational science (Simpson et al., 2022; Lai et al., 2024). Collectively, these findings affirm that a multi-platform approach, integrating HRMS for discovery,

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LC–MS/MS for quantification, and SERS/sensors for screening, represents the most comprehensive and cost-effective strategy for future PFAS monitoring. The evaluation of analytical performance across methods reveals both complementarities and limitations. While LC–MS/MS dominates due to precision and reproducibility, emerging technologies such as HRMS and GC×GC–MS provide molecular-level insights and enhanced scope. High-throughput HPLC–MS/MS methodologies enhanced targeted analysis to encompass numerous analytes, exhibiting dependable quantification of over 50 PFAS in aqueous matrices and improving reporting thresholds for surveillance (Gremmel et al., 2016).

LC–MS/MS remains indispensable for regulatory monitoring, with LODs typically 0.05–1 ng L^{-1} and recoveries exceeding 80–115% (Mulabagal et al., 2018). It is the most standardized and globally harmonized approach for quantifying ionic PFAS in water and serum. Longitudinal biomonitoring studies employing proven LC–MS/MS methodologies showed temporal trends and supported technique robustness for serum matrices (Gribble et al., 2015).

UHPLC–HRMS, however, offers unmatched versatility, detecting known, suspect, and unknown PFAS species simultaneously. High-resolution capabilities (2–5 ppm accuracy) allow retrospective re-analysis of archived datasets as new PFAS emerge in regulatory watchlists (Barzen-Hanson et al., 2017; Getzinger et al., 2021).

In contrast, **GC–MS** provides a unique window into volatile PFAS precursor compounds that LC–MS/MS cannot efficiently detect due to volatility or lack of ionization under ESI conditions. Fang et al. (2020) both highlight its value in identifying transformation pathways, such as the oxidation of FTOHs to PFOA and PFNA, which contribute significantly to secondary environmental contamination.

EOF/AOF methods, although non-selective, close the mass balance gap by quantifying total fluorine load. Studies by Pan et al. (2023) and Salameh et al. (2021) consistently show that identified PFAS represent only 20–30% of total extractable organofluorine, indicating that the majority of environmental fluorine remains chemically unresolved.

Finally, **SERS and electrochemical sensors** while still emerging are demonstrating practical value. Huo et al. (2025) achieved PFOS detection down to 0.01 ng L^{-1} using nanostructured silver substrates, and Rezaei et al. (2024) reported similar sensitivities using MOF graphene electrodes for PFOA and PFOS. Their speed and portability make them ideal complements for rapid field assessments.

Technique	Target PFAS	Typical Matrix	LOD (ng L^{-1})	Recovery (%)	Strengths	Limitations	Key References
LC–MS/MS	Legacy PFAS	Water, serum, soil	0.05–1	80–115	High sensitivity; regulatory validation	Limited analyte scope	Shoemaker & Tettenhorst (2018); Liu et al. (2024)
UHPLC–HRMS	Legacy & emerging	Wastewater, biota	0.1–10	75–105	Non-target screening; structural elucidation	Complex data analysis	Getzinger et al. (2021); Lai et al. (2024)
GC–MS / GC×GC–MS	Volatile precursors	Air, sediments	0.1–5	70–95	Ideal for volatile PFAS; transformation insight	Derivatization; thermal degradation	Fang et al. (2020);
EOF/AOF (CIC)	Total fluorine	Soil, sludge	1–20 (ng F L^{-1})	—	Mass balance for unknown PFAS	No molecular ID	Pan (2023); Salameh (2021)
SERS	Ionic PFAS	Water, plasma	0.01–0.5	80–120	Portable; rapid	Substrate instability	Huo et al. (2025)
Electrochemical Sensors	PFOS, PFOA	Water, serum	0.01–0.1	90–120	Green; low-cost	Calibration dependence	Rezaei et al. (2024)

Table 2. Comparative analytical performance of PFAS detection techniques

Table 2 compares performance metrics across platforms, showing that LC–MS/MS still provides the best reproducibility for routine monitoring, while HRMS and GC×GC–MS extend analytical breadth. GC based workflows excel in resolving precursor-product relationships; HRMS dominates for non-targeted discovery; SERS and electrochemical sensors, though still emerging, open pathways toward real-time and decentralized PFAS detection. The contrast between methods underscores a fundamental trade-off**:** precision versus coverage.

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3.4. Emerging Analytical Trends and Integration of AI

In the last decade, artificial intelligence (AI) has become integral to PFAS analytics. Machine learning models predict chromatographic retention times and fragmentation spectra with high precision, enabling automated identification of unknown PFAS in HRMS datasets (Simpson et al., 2022; Lai et al., 2024).

AI also supports real-time decision-making in field-deployable sensor systems. Algorithms trained on Raman spectral libraries can differentiate PFOS from similar anions, enhancing selectivity for SERS-based detection (Huo et al., 2025). These models reduce false positives and improve quantification in complex matrices.

The future analytical landscape is increasingly interdisciplinary, combining AI, HRMS, electrochemical, and spectroscopic tools to achieve comprehensive PFAS profiling. The emergence of hybrid monitoring systems for example, coupling HRMS discovery with portable electrochemical verification signals a paradigm shift toward integrated environmental surveillance frameworks.

3.5. Synthesis and Implications

Across the fifteen-year timeline (2010–2025), PFAS analytical capabilities have expanded from low-resolution quantification to high-resolution, AI-supported discovery. Detection limits improved by three orders of magnitude, analytical coverage increased from fewer than 20 to over 1,200 PFAS, and extraction workflows transitioned to greener, more selective technologies. The regulatory examination of PFOA and associated compounds has intensified the need for standardized analytical methodologies, as highlighted in preliminary regulatory assessments that stressed the importance of inter-laboratory consistency and method standardization (Vierke et al., 2014). However, challenges persist. The lack of CRMs for complex matrices, incomplete fluorine mass balance, and uneven QA/QC standards hinder global data comparability. Addressing these issues will be pivotal to developing harmonized, next-generation PFAS monitoring frameworks capable of linking molecular-level detection with environmental and toxicological risk assessment.

4. FUTURE DIRECTIONS AND RESEARCH GAPS

4.1. Advancing Analytical Sensitivity and Comprehensive Detection

Despite significant advancements, PFAS analysis still captures only a fraction of the total organofluorine burden in environmental and biological systems. Studies using EOF/AOF approaches indicate that identified PFAS account for merely 20–30 % of total extractable organofluorine (Pan et al., 2023; Salameh et al., 2021). The remaining 70–80 % of the so-called “dark fluorine” comprises transformation products, polymeric PFAS, and uncharacterized intermediates.

Future research must therefore emphasize comprehensive analytical coverage through multi-platform integration. Combining LC–MS/MS, UHPLC–HRMS, and GC×GC–TOF–MS in hybrid workflows can provide a more complete molecular inventory, bridging the gap between ionic, neutral, and volatile PFAS. Emerging hyphenated techniques, such as LC–IMS–HRMS (ion mobility separation coupled with high-resolution MS), can further enhance selectivity by separating isomers and conformers prior to mass detection. Parallel innovations in laser-based and microfluidic spectroscopic systems could miniaturize analysis, reducing sample and reagent consumption while maintaining sensitivity. Such developments will be essential to reach single-picogram or femtogram detection limits, particularly for PFAS in remote, low-contamination matrices such as Arctic snow and deep-sea sediments.

4.2. Expanding Non-Targeted and Retrospective Screening Capacity

High-resolution, non-targeted screening (NTS) represents one of the most transformative areas in PFAS analytics. While UHPLC–HRMS and GC×GC–MS have expanded discovery, data interpretation remains challenging. The next generation of PFAS analytics should focus on automated data mining pipelines, combining HRMS raw data with machine learning algorithms capable of identifying structural classes based on fragmentation patterns, exact mass, and retention behavior. For example, Simpson et al. (2022) demonstrated AI models that predicted PFAS class membership with >90 % accuracy from HRMS spectra.

Retrospective screening the reanalysis of archived HRMS datasets using updated PFAS libraries will also play a vital role. As new compounds are discovered and added to global repositories such as NORMAN-SLE and EPA CompTox, older data can be re-mined for missed compounds, allowing long-term exposure reconstruction without re-sampling. In the future, integrating AI-driven annotation tools into open-access HRMS data platforms will help democratize PFAS research, enabling cross-laboratory harmonization and improving global surveillance capabilities.

4.3. Developing Universal Standards and Certified Reference Materials

One of the most persistent barriers to comparability in PFAS analytics is the lack of certified reference materials (CRMs) for complex matrices. Current CRMs such as NIST SRM 4327 for drinking water do not represent soils, sediments, or biological tissues, where PFAS partitioning and matrix interferences are significant (Sommerfeld et al., 2025).

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Future efforts should prioritize:

1. Matrix-specific CRMs, particularly for high organic matter soils, wastewater sludge, and serum.
2. Stable isotope-labeled standards for short-chain and emerging PFAS, many of which currently lack surrogates for isotope dilution quantification.
3. Globally harmonized QA/QC frameworks, building upon the OECD's 2023 harmonization initiative, to define acceptable precision, recovery, and reporting formats across laboratories.

Without such reference standards, it is impossible to align analytical outcomes across nations and accurately assess environmental trends or health risks. Standardization will therefore underpin the reliability of both regulatory decisions and risk modelling.

4.4. Integrating Green Chemistry and Sustainable Analytical Design

Analytical science must also advance in sustainability. The extensive utilization of fluorinated solvents, single-use cartridges, and substantial amounts of organic solvents contradicts environmental stewardship objectives. To reconcile analytical accuracy with ecological responsibility, PFAS detection must integrate green analytical chemistry principles.

Deep eutectic solvents (DES) and ionic liquids have already demonstrated comparable extraction efficiencies to traditional solvents with drastically reduced toxicity and waste (Fan et al., 2021). Microextraction techniques**,** including dispersive liquid–liquid microextraction (DLLME) and solid-phase microextraction (SPME)**,** minimize solvent consumption while maintaining high enrichment factors.

Future research should advance bio-based sorbents, such as cellulose-modified polymers or plant-derived nanocomposites, to replace fluorinated extraction materials. Furthermore, the analytical community must adopt life-cycle assessment (LCA) frameworks to evaluate the total environmental impact of PFAS detection, encompassing sample preparation, instrument operation, and waste disposal.

4.5. Field-Deployable and Real-Time Monitoring Technologies

While laboratory-based LC–MS/MS and HRMS remain indispensable, they are resource-intensive and inaccessible for many monitoring networks. The next frontier is real-time, on-site PFAS detection using portable, miniaturized sensors.

Surface-enhanced Raman spectroscopy (SERS) and electrochemical biosensors have shown promise for field analysis. Huo et al. (2025) reported sub-ng L^{-1} detection of PFOS using SERS with gold–silver nanostructures, while Rezaei et al. (2024) developed a MOF graphene hybrid electrode capable of simultaneous PFOA and PFOS quantification at 0.05 ng L^{-1} sensitivity.

However, reproducibility remains limited by matrix interferences and substrate degradation. Future innovation should focus on:

- **Standardized SERS substrates** with improved signal stability.
- **Integrated microfluidic–electrochemical devices** combining sample extraction, preconcentration, and detection in a single unit.
- **Wireless, AI-assisted platforms** capable of automated data transmission and spectral classification for real-time decision-making.

Such technologies would revolutionize environmental surveillance, enabling rapid PFAS mapping in water networks, industrial effluents, and contaminated soils without extensive laboratory infrastructure.

4.6. Understanding Transformation Pathways and Total Fluorine Mass Balance

A critical scientific challenge remains in linking precursor PFAS to their transformation products. Studies using GC–MS and HRMS indicate that volatile precursors (FTOHs, FASAs) degrade into persistent perfluoroalkyl acids (PFAAs), yet the kinetics and intermediate steps remain poorly resolved (Fang et al., 2020).

Future work must integrate environmental chemistry and analytical science to trace these transformation pathways comprehensively. Controlled oxidation and biodegradation experiments combined with isotopic labeling and non-target HRMS tracking can reveal degradation routes and intermediate species.

Moreover, incorporating total organofluorine mass balance (EOF/AOF) alongside compound-specific analysis will provide more accurate estimates of total PFAS burdens. A combination of top-down (CIC) and bottom-up (MS-based) approaches will ultimately be required to close the global fluorine budget and fully assess the persistence of these compounds.

4.7. Bridging Analytical Advances with Policy and Public Health

The analytical community plays a central role in informing regulation and human health protection. However, analytical uncertainty often constrains policy decisions. Future PFAS research must therefore move toward translational analytics bridging the gap between instrumental precision and actionable public health data.

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Key priorities include:

- Establishing threshold detection levels relevant to toxicological endpoints, ensuring that analytical advances directly inform exposure limits.
- Developing standardized reporting protocols for PFAS occurrence data, including metadata for matrix type, extraction conditions, and analytical parameters.
- Strengthening communication between analytical chemists, toxicologists, and regulators to align method detection limits (MDLs) with health-based guidance levels (HBGVs).

By harmonizing analytical innovation with policy translation, PFAS science can more effectively contribute to mitigation strategies and risk assessment frameworks at global and regional scales.

4.8. Emerging Interdisciplinary Directions

PFAS research is now inherently interdisciplinary. The future lies in integrating analytical chemistry, data science, toxicology, and environmental modelling into unified frameworks. AI-driven analytical platforms, coupled with predictive environmental fate modelling, can simulate PFAS transport, transformation, and bioaccumulation in silico, reducing dependence on costly field campaigns.

Furthermore, combining omics approaches (metabolomics, proteomics) with PFAS exposure profiling can help elucidate biological pathways of toxicity. Molecular-level analytical data generated by HRMS will thus serve as the foundation for understanding PFAS–biomolecule interactions, enabling mechanistic insights into bioaccumulation and disease associations.

5. CONCLUSION

Emerging frontiers are poised to close many of these gaps. Artificial intelligence and machine learning are already transforming non-targeted workflows, accelerating the identification of unknown PFAS from complex HRMS datasets. Portable sensor technologies, such as surface-enhanced Raman spectroscopy (SERS) and electrochemical nanocomposite sensors, promise to decentralize PFAS monitoring making real-time detection in the field a practical reality. Furthermore, the integration of AI-driven data platforms, open-access spectral repositories, and inter-laboratory standardization initiatives by organizations like the OECD are setting the stage for global harmonization of PFAS analytics.

Looking ahead, the next decade of PFAS research must prioritize interdisciplinary collaboration bridging analytical chemistry, environmental science, toxicology, and data analytics. Analytical advances must translate into environmental and health insights, supporting regulatory frameworks and public policy. Comprehensive PFAS detection cannot be viewed as an isolated technical goal; rather, it must underpin holistic strategies for exposure mitigation, remediation, and chemical substitution.

This review demonstrates that PFAS analytical technologies have reached remarkable levels of precision and sensitivity. Yet, the field stands at a pivotal stage where additional advancement necessitates extensive detection coverage, uniform global methodology, and sustainable analytical processes. The integration of advanced instrumentation with artificial intelligence and green chemistry provides a means for enhanced, eco-friendly, and internationally standardized PFAS monitoring. Achieving this convergence will be extremely important for accurately tracking, regulating, and alleviating the extensive effects of these persistent chemicals across environmental and biological systems.

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