



In-House LC-MS/MS Analysis of PFAS Compounds to Expedite Decision Making

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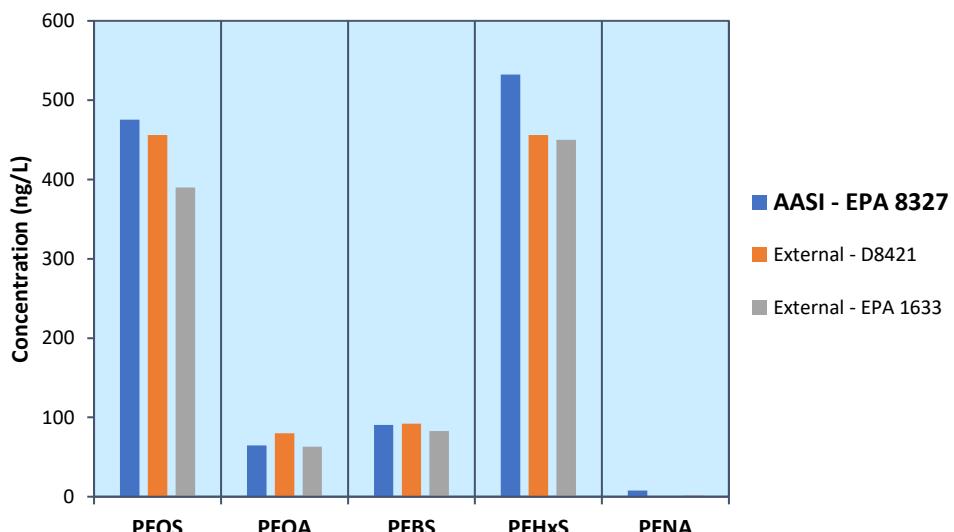
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Executive Summary

The in-house Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS) analysis at Aqua-Aerobic Systems Inc. (AAIS) has been developed and executed for PFAS testing. Given the increasing demand for such analysis and the limited availability of accredited laboratories, turnaround times can reach 6-8 weeks and customers are faced with decision to expedite testing at an additional cost. AAIS has conducted plentiful PFAS analyses over the last year to ensure reliable results that can be compared and aligned with those from accredited laboratories. For example, PFAS pilot study was conducted at Dobbins Air Reserve Base (USA) to evaluate AquaPRS™ PFAS Removal System for groundwater treatment. The results obtained by AAIS for PFAS regulated compounds demonstrated a strong correlation with other external testing methods as illustrated in the chart below.



Keywords:

Per-and Polyfluorinated Alkyl

Surface Water

Groundwater

EPA 8327

Emerging Contaminants

PFAS

AAIS' laboratory testing capability can deliver results within a week and analyze up to 50 samples over that period. Testing 20 samples per analysis is usually performed to ensure high quality outcomes.

Motivation

After several years of research and development, AASI officially launched a novel PFAS treatment system AquaPRS™ in early 2024 which initially focused on highly contaminated groundwater [AASI, 2024]. The subsequent release of the National Primary Drinking Water Regulations (NPDWR) prompted high demand for PFAS laboratory services in the drinking water market, decreasing response times and increasing costs for certified results. Therefore, AASI decided to obtain its own LC-MS/MS instrument to serve its research and development efforts in evaluating and optimizing the AquaPRS™ PFAS Removal System. As well as, facilitate the availability of results to expedite decision making on pilot studies and provide a service to future customers with full-scale AquaPRS™ PFAS removal systems.

Introduction

PFAS, Per- and Polyfluoroalkyl Substances, often referred to as "forever chemicals", are known to pose significant environmental and health risks. As the U.S. Environmental Protection Agency (EPA) moves forward with assessing and regulating PFAS, the water and wastewater industries need laboratories capable of analyzing these compounds. With escalating demand for PFAS analysis and limited number of accredited laboratories, turnaround times can often exceed a month and costs can be very expensive per sample. Further, emerging procedures and target compounds add complexity in identifying the appropriate methods for a given matrix.

Current emphasis on PFAS treatment has focused on municipal drinking water facilities due to the EPA's National Primary Drinking Water Regulations [NPDWR; EPA 2024]. However, increased scrutiny of the relationship between wastewater treatment plant (WWTP) discharges and drinking water intakes has

raised more recent concerns. Ongoing research reveals that PFAS levels in WWTP effluents are higher than expected, potentially due to pharmaceutical residuals. Prescription drugs contribute 75% of the extractable organic fluorine in wastewater, while the six regulated NPDWR PFAS compounds constitute <10% of the total organic fluorine [Ruyle et al., 2025]. Sustainable drinking water management practices must consider the interconnectivity of PFAS with wastewater and stormwater.

LC-MS/MS is a powerful analytical technique that combines the physical separation abilities of liquid chromatography with the mass analysis abilities of mass spectrometry. In this white paper, we will address how AASI is implementing LC-MS/MS for PFAS testing and assess the major findings compared to accredit laboratories' results.

Experimental

■ LC-MS/MS Installation and Running

AASI developed an in-house plan for PFAS testing and integration to efficiently handle high volume of samples and ensure rapid data response times. Activities included defining project goals, creating a budget, identifying test methods, remodeling the onsite laboratory, and training analysts. AASI selected Vanquish Flex tandem TSQ Altis™ Plus Liquid Chromatography-Mass Spectrometry (Figure 1). The manufacturer installed the equipment and formal training for staff was accomplished. Following training, staff developed sampling procedures, cleaning protocols, standard operating procedures for analysis, and report formats.



Figure 1. High Performance Liquid Chromatography-Tandem Mass Spectrometry System

■ PFAS Compounds and Testing Method

AASI implemented the validated testing method of [EPA 8327] that offers the required accuracy with faster response time. **Table 1** lists all 43 PFAS compounds (24 targets and 19 isotopically-labeled surrogates) that can be detected by this direct injection testing method for various sample matrices (Groundwater, Surface water, and Wastewater). Stock standard solution at a concentration of 500 ng/L was used to prepare the calibration curve. The stock solution was diluted using organic diluent of 50% methanol to prepare eight calibration solutions at 300, 200, 100, 50, 25, 10, 5, and 2 ng/L.

Control quality samples (Lower Limits of Quantitation [LLOQs], Laboratory Control Sample [LCS], Matrix Spike/Matrix Spike Duplicate [MS/MSD], Method Blank [MB]) were prepared as indicated by EPA Method 8327. Tested samples (5 mL) were diluted 1:1 with methanol and spiked with 40 µL of surrogate spiking solution and vortexed for 2 minutes. The samples then were filtered and 10 µL acetic acid was added to the filtrate to adjust the pH. Aliquots of 100 µL were transferred to LC-MS vials for injection and analysis by LC-MS/MS.

Table 1. List of PFAS Compounds Analyzed at AASI Lab

PFAS Compound	Abb.	Surrogate	Detection Limit (ng/L)
PERFLUOROALKYLCARBOXYLIC ACIDS			
Perfluorobutanoic acid	PFBA	MPFBA	2
Perfluoropentanoic acid	PPeA	MPFPeA	2
Perfluorohexanoic acid	PFHxA	MPFHxA	2
Perfluoroheptanoic acid	PFHpA	MPFHpA	2
Perfluorooctanoic acid	PFOA	MPFOA	2
Perfluorononanoic acid	PFNA	MPFNA	2
Perfluorodecanoic acid	PFDA	MPFDA	2
Perfluoroundecanoic acid	PFUdA	MPFUdA	2
Perfluorotetradecanoic acid	PFTeDA	MPFTeDA	5
Perfluorododecanoic acid	PFDoA	MPFDoA	5
Perfluorotridecanoic acid	PFTrDA	--	10
PERFLUOROALKYLSULFONATES			
Perfluorobutyl sulfonate	PFBS	MPFBS	2
Perfluorohexyl sulfonate	PFHxS	MPFHxS	2
Perfluoroctyl sulfonate	PFOS	MPFOS	2
Perfluoroheptane sulfonate	PFHpS	--	2
Perfluoropentane sulfonate	PPeS	--	2
Perfluorodecane sulfonate	PFDS	--	2
Perfluorononane sulfonate	PFNS	--	2
FLUORINATED TELOMER SULFONATES			
Sodium 1H,1H,2H,2H-perfluorohexane sulfonate	4:2FTS	M4:2FTS	2
Sodium 1H,1H,2H,2H-perfluorooctane sulfonate	6:2FTS	M6:2FTS	2
Sodium 1H,1H,2H,2H-perfluorodecane sulfonate	8:2FTS	M8:2FTS	2
PERFLUOROOCTANESULFONAMIDE AND PERFLUOROOCTANESULFONAMIDOACETIC ACIDS			
Perfluorooctanesulfonamide	PFOSA	MFOSA	10
2-(N-ethylperfluorooctanesulfonamido) acetic acid	N-EtFOSAA	d5-N-EtFOSAA	2
2-(N-methylperfluorooctanesulfonamido) acetic acid	N-MeFOSAA	d3-N-MeFOSAA	2

Findings

■ LC-MS/MS Quality Performance

The LC-MS/MS system was delivered, installed, and operated successfully. Training sessions were held to focus on the system components such as liquid chromatography, mass spectrometry, user interface, and associated software. AASI was able to obtain a well representative chromatogram for all 43 PFAS compounds. Moreover, AASI was able to establish valid calibration curve which is an essential step in any analytical method. **Figure 2** represents an example for PFAS compounds chromatograms and a calibration curve for PFOA from 2-300 ng/L.

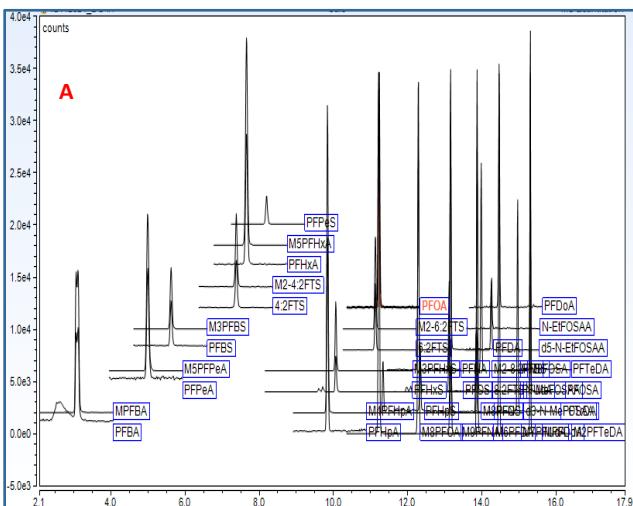


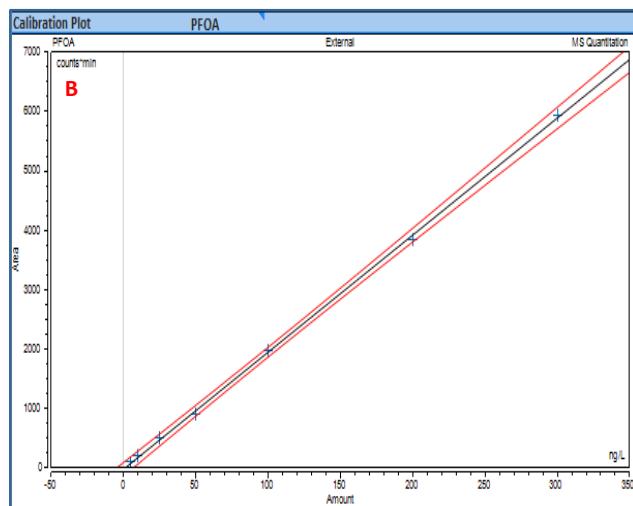
Figure 2. (A) Chromatograms of PFAS Compounds, (B) Calibration Curve of PFOA Showing Valid Alignment

■ AASI Results Vs External Accredited Laboratory

High-quality data remains important, and achieving reliable results was challenging besides learning to operate the LC-MS/MS and interpret data. However, AASI team was able to obtain reliable results so far and conducted many PFAS analyses over the previous months. For instance, a PFAS pilot study was conducted at Dobbins Air Reserve Base (USA) in Fall 2024 to evaluate AquaPRS™ PFAS Removal System for groundwater treatment. Collected samples for the 6-months study were simultaneously analyzed at AASI

lab and external lab to track the reliability of our data. AASI's results using EPA method 8327 were compared to certified contract laboratory's results using EPA method D8421 and EPA method 1633. The comparison showed that AASI's data align with the certified laboratory as indicated in **Figure 3**.

In another study, AquaPRS™ piloting unit is implemented at Victor Gaffney WTP, Gaffney, in South Carolina to treat filtered surface water. The duration of this study is estimated to be approximately 3 months of testing, in which samples are being collected and tested in AASI's lab along with another certified lab to support our data validity.



Recent results have been generated and compared with EPA Method 533 as illustrated in **Figure 4**. The comparison revealed a very positive correlation for analyzed PFAS compounds, especially the EPA regulated compounds of PFOS, PFOA, PFBS, PFNA, and, PFHxS. AASI staff dedicated significant time to reducing contamination and identified sources of concern and made modifications to the analytical process to achieve reliability and promote the LC-MS/MS service for piloting AquaPRS™ PFAS Removal System.

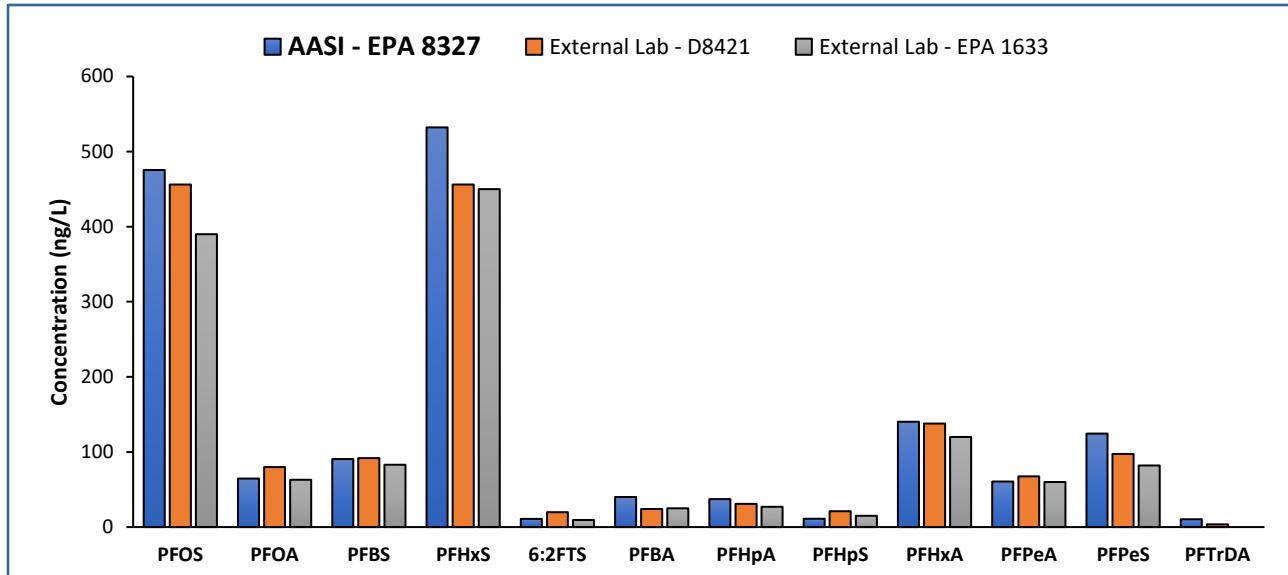


Figure 3. AASI's PFAS Pilot Study Results in Comparison to External Accredited Lab Results Using Methods D8421 and 1633

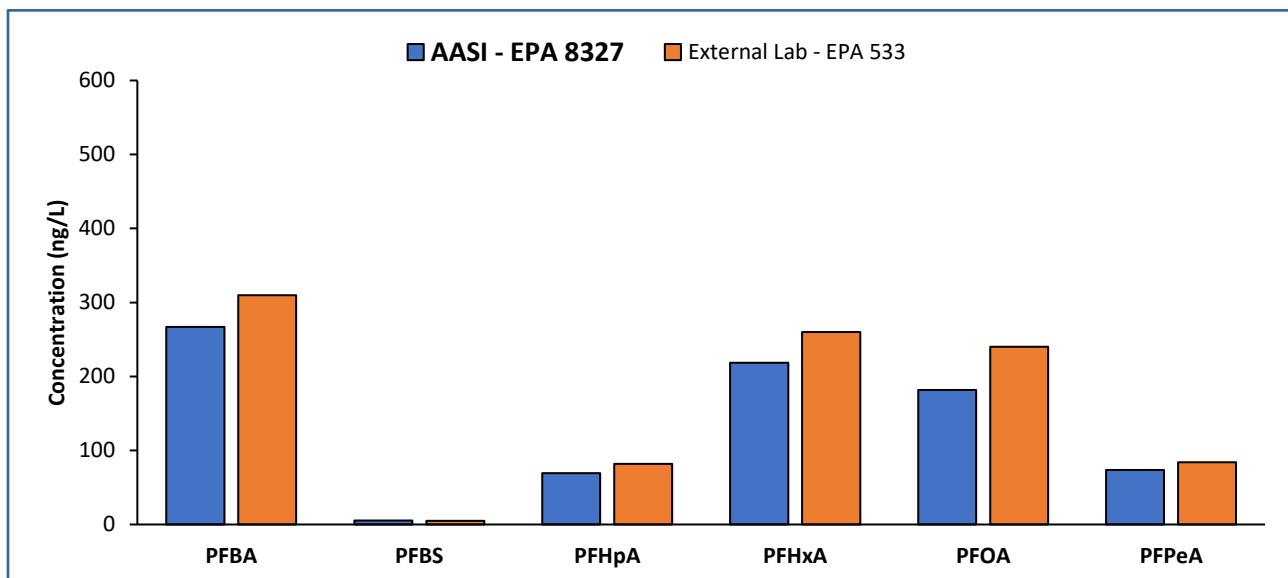


Figure 4. AASI's PFAS Pilot Study Results in Comparison to External Accredited Lab Results using EPA Method 533

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