

# APPLICATIONS

## Per- and Polyfluoroalkyl Substances (PFAS) Extraction by LC-MS/MS Using Strata® PFAS for a Stacked Solid Phase Extraction (SPE) Solution

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### Overview

PFAS are a class of highly stable synthetic organic compounds used in a wide variety of industrial and commercial applications. They are also highly stable in the environment and strongly bioaccumulate. As a result, they have become ubiquitous throughout the global environment and are often referred to in today's media as "Forever Chemicals". Consequently, PFAS levels need to be tested in drinking water and more recently methods have been developed to measure PFAS in other environmental matrices that require more complex clean-up solutions, such as wastewater, soils and sediments.

The United States Department of Defense (DOD) is dealing with very extensive PFAS contamination owing to the widespread use of PFAS based Aqueous Film Forming Foam (AFFF) used as fire suppression foams at many military installations. As a result, DOD has developed its own PFAS analytical guidelines to deal with the unique environmental monitoring and clean-up challenges found on their installations. These guidelines, contained within the DOD QSM 5.1/5.3 documentation, contains a unique sample clean-up and concentration approach not found in EPA Methods which are designed only for drinking water application.

DOD QSM 5.1/5.3 specifies the use of a polymeric weak anion exchange (WAX) SPE sorbent in combination with graphitized carbon black (GCB) sorbent for the clean-up of solid samples, soils, biota, sediments, or non-drinking water samples. This can be performed by using two individual tubes of WAX and GCB sorbent that are applied sequentially or the use of dispersive SPE (dSPE) utilizing GCB following the WAX SPE tube extraction. Both methods add time to the clean-up procedure and present the opportunity for loss of analytes and introduction of imprecision. In this communication we describe a significant improvement to the guidelines, Strata PFAS SPE, wherein the two sorbents are contained within a single tube, offering the opportunity for decreased sample processing time and increased accuracy and precision. When comparing different recoveries for a small subset of

analytes for a WAX SPE and dSPE GCB method vs Strata PFAS, the recovery is greatly improved using Strata PFAS (**Table 1**).

Strata PFAS is a stacked single cartridge solution with polymeric WAX and GCB sorbents that functions as a traditional Solid Phase Extraction (SPE) cartridge with a built in polishing step to meet the aforementioned DOD guidelines. This SPE product increases lab productivity and reduces the need for multiple extraction tubes when compared to a traditional two tube method.

**Table 2** presents typical analyte recovery data from a routine Laboratory Control Sample (LCS) analyzed by a commercial testing laboratory highly experienced with the performance of DOD QSM 5.1/5.3. The LCS had been spiked with all 32 target analytes at 25 µg/L and was analyzed with a batch of field samples to demonstrate method performance and data acceptability. The recovery data show that all 32 analytes were well within method recovery limits with an average recovery of 98.8% and a mean recovery of 99.0%, thereby demonstrating acceptability of the use of Strata PFAS in the performance DOD QSM 5.1/5.3.

The LCS sample was extracted with Strata PFAS under the conditions shown below and analyzed on a LC-MS/MS system using a [Gemini® 3 µm C18](#) HPLC column under the conditions described in a previously published [GenX Application Note](#).

### SPE Conditions

**Cartridge:** Strata PFAS 200 mg WAX/50 mg GCB/6 mL)

**Part No.:** [CS0-9207](#)

**Condition 1:** 4 mL 0.3 % Ammonium hydroxide

**Condition 2:** 4 mL Methanol

**Equilibrate:** 5 mL Water

**Load:** Add sample at 4 mL/min

**Wash:** 2x 4 mL Water

**Elute:** 2x 4 mL 0.3 % Ammonium hydroxide in Methanol

**Evaporate:** to dryness and reconstitute to 1 mL with Methanol/Water (96:4)

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**Table 1: Recovery Comparisons of WAX SPE and dSPE using GCB vs Strata® PFAS Single Cartridge Method**

Analyte	WAX SPE + dSPE GCB % Recovery	Strata PFAS Stacked Cartridge % Recovery
13C2-PFDoDA	77	84.5
13C2-PFTeDA	62	84.0
PFODA	38	78.3
PFHxDA	63	89.3

**Table 2: Recovery of QSM 5.3 Target Analytes from a Laboratory Control Sample Using Strata PFAS SPE (WAX/GCB)**

Analyte	Actual Concentration	Sample Result	% Recovery	Method Recommendation Limits	Pass/Fail
PFBA	25.600	22.640	88	84-135	Pass
PFPeA	25.600	22.157	87	75-138	Pass
PFBS	22.640	22.300	99	81-133	Pass
4:2-FTS	23.920	22.078	92	64-134	Pass
PFHxA	25.600	24.644	96	80-137	Pass
PFPeS	24.000	21.699	90	82-132	Pass
HFPODA	25.600	26.336	103	70-130	Pass
PFHpA	25.600	27.018	106	80-140	Pass
PFHxS	24.200	24.713	102	71-131	Pass
DONA	24.120	26.083	108	70-130	Pass
6:2-FTS	24.280	24.217	100	51-155	Pass
PFHpS	24.360	23.015	94	80-129	Pass
PFOA	25.600	25.043	98	83-138	Pass
PFOS	24.480	22.492	92	54-139	Pass
PFNA	25.600	25.872	101	73-140	Pass
9CI-PF3ONS	23.840	21.863	92	70-130	Pass
PFNS	24.560	21.993	90	71-121	Pass
PFDA	25.600	25.047	98	78-137	Pass
8:2-FTS	24.520	22.231	91	62-133	Pass
PFOSA	25.600	25.714	100	73-121	Pass
NMEFOSAA	25.600	30.906	121	53-136	Pass
PFDS	24.640	22.873	93	69-124	Pass
PFUnDA	25.600	26.353	103	70-134	Pass
NEtFOSAA	25.600	28.765	112	59-145	Pass
11CI-PF3OUdS	24.120	22.625	94	70-130	Pass
PFDoDA	25.600	27.710	108	75-139	Pass
10:2-FTS	24.680	26.626	108	50-124	Pass
PFDoS	24.800	21.509	87	39-121	Pass
PFTTrDA	25.600	25.814	101	67-144	Pass
PFTeDA	25.600	25.446	99	79-134	Pass
PFHxDA	25.600	29.662	116	36-136	Pass
PFODA	25.600	27.373	107	10-124	Pass

Recovery Range: 87 % - 116 %  
Average Recovery: 98.8 %  
Mean Recovery: 99.0 %

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## Acknowledgements

Special thanks Andrew Patterson, Charles Neslund, Robert Brown and to the team at Eurofins for their help contributing to this product and method.

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