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Overview

EPA 533 recommends SPE cartridges containing weak anion exchange, mixed-mode polymeric sorbent (polymeric backbone and a diamino ligand), with a particle size of approximately 33 μm. The Solid Phase Extraction (SPE) sorbent must have a pKa above 8 for optimal extraction. In this application note we compared the recovery of PFAS compounds using a Strata-XL-AW SPE cartridge, a similar functionality but with 100 µm particle size, pore size of 300 Å, and a 500 mg/6 mL format. The EPA 533 method flexibility allows alternative SPE phases to be used. Six sets of extractions were performed on drinking water using a Strata X-AW and a Strata XL-AW SPE cartridge. The extractions performed using the Strata-XL-AW met EPA recovery requirements, while providing the advantage of faster sample loading. This reduces the sample loading time by half for clean samples (like drinking water) compared to traditional extraction methods using smaller pore and particle sizes, while maintaining % recovery, as presented in Table 1.

LC Conditions

Column: Gemini[™] 3 μm C18 **Dimensions:** 50 x 2.0 mm

Part No.: <u>00B-4439-B0</u>

Mobile Phase: A: 20 mM Ammonium Acetate

B: Methanol

| Gradient: | Time (min) | % I |
|------------------|------------|------------|
| | 0 | 5 |
| | 0.5 | 5 |
| | 3 | 40 |
| | 16 | 80 |
| | 18 | 80 |
| | 20 | 95 |
| | 22 | 95 |
| | 25 | 5 |
| | 35 | 5 |

Flow Rate: 0.25 mL/min

Injection Volume: 2 µL

Detection: ESI-MS/MS

Sample Preparation

| Step | Description |
|---------------------------|--|
| Sample Pre- treatment: | 250 mL sample is fortified with isotopically labeled analogues of the method analytes. |
| Load: | 250 mL pre-treated sample through a Strata-XL-AW 500 mg/6 mL (<u>8B-S051-HCH</u>) cartridge or through a Strata-X-AW 500 mg/6 mL (<u>8B-S038-HCH</u>) cartridge. |
| Wash: | Cartridges with aqueous Ammonium Acetate, followed by Methanol. |
| Elute: | Sample with Ammonium Hydroxide in Methanol. |
| Dry Down: | Under a gentle stream of Nitrogen in a heated water bath. |
| Reconstitute: | Sample to a final volume to 1 mL with 20 % Water in Methanol (v/v) before analyzing by LC-MS/MS. |

"The Strata-XL-AW tubes result in a significant time savings during the sample loading step. The Strata-XL-AW tubes take about half the time to load clean samples, and about 20 % of the time required for dirty samples. This reduces the impact of the SPE bottleneck, allowing us to process more samples per shift."

Agustin Pierri, PhD,
 Technical Director, Weck Laboratories

 Table 1. Comparison of % Recovery of PFAS Compounds Using Strata™-XL-AW versus Strata-X-AW SPE Cartridges.

| | Strata XL-AW | | |
|---------------------------------------|-------------------------|------|------|
| Target Analyte | Avg % Recovery (n=6) | STD | %RSD |
| ¹³ C ₄ -PFBA | 89.23 | 2.47 | 3% |
| PFBA | 97.45 | 3.12 | 3% |
| PFMPA | 101.54 | 3.07 | 3% |
| ¹³ C ₅ -PFPeA | 90.55 | 2.88 | 3% |
| PFPeA | 104.59 | 3.97 | 4% |
| ¹³ C ₃ -PFBS | 88.56 | 2.48 | 3% |
| PFBS | 103.54 | 2.87 | 3% |
| PFMBA | 104.9 | 4.68 | 4% |
| PFEESA | 104.71 | 4.06 | 4% |
| NFDHA | 108.74 | 7.44 | 7% |
| ¹³ C ₂ -4:2 FTS | 83.73 | 3.79 | 5% |
| 4:2 FTS | 92.23 | 3.41 | 4% |
| ¹³ C ₅ -PFHxA | 90.99 | 3.43 | 4% |
| PFHxA | 96.42 | 4.63 | 5% |
| PFPeS | 101.69 | 4.70 | 5% |
| ¹³ C ₃ -HFPO-DA | 89.75 | 4.07 | 5% |
| HFPO-DA | 106.86 | 4.11 | 4% |
| ¹³ C ₄ -PFHpA | 90.66 | 4.29 | 5% |
| PFHpA | 99.51 | 3.76 | 4% |
| ¹³ C ₃ -PFHxS | 91.27 | 3.82 | 4% |
| PFHxS | 103.11 | 4.96 | 5% |
| ADONA | 102 | 3.31 | 3% |
| ¹³ C ₂ -6:2 FTS | 86.01 | 5.28 | 6% |
| 6:2 FTS | 93.71 | 4.85 | 5% |
| ¹³ C ₈ -PFOA | 91.4 | 4.12 | 5% |
| PFOA | 102.97 | 3.67 | 4% |
| PFHpS | 105.16 | 4.49 | 4% |
| ¹³ C ₉ -PFNA | 89.91 | 4.04 | 4% |
| PFNA | 102.76 | 1.98 | 2% |
| ¹³ C ₈ -PFOS | 88.85 | 4.72 | 5% |
| PFOS | 105.93 | 4.61 | 4% |
| 9Cl-PF3OUdS | 99.65 | 2.93 | 3% |
| ¹³ C ₂ -8:2 FTS | 85.24 | 4.22 | 5% |
| 8:2 FTS | 100.36 | 3.75 | 4% |
| ¹³ C ₆ -PFDA | 89.83 | 5.43 | 6% |
| PFDA | 100.45 | 4.10 | 4% |
| ¹³ C ₇ -PFUnA | 87.57 | 4.32 | 5% |
| PFUnA | 100.51 | 2.95 | 3% |
| 11Cl-PF3OUdS | 103.01 | 2.32 | 2% |
| ¹³ C ₂ -PFDoA | 83.61 | 4.49 | 5% |
| PFDoA | 103.67 | 4.07 | 4% |

| | Strata-X-AW | | |
|---------------------------------------|-------------------------|------|------|
| Target Analyte | Avg % Recovery (n=6) | STD | %RSD |
| ¹³ C ₄ -PFBA | 91.28 | 3.37 | 4% |
| PFBA | 97.2 | 4.13 | 4% |
| PFMPA | 100.62 | 3.40 | 3% |
| ¹³ C ₅ -PFPeA | 92.54 | 3.15 | 3% |
| PFPeA | 101.93 | 4.51 | 4% |
| ¹³ C ₃ -PFBS | 87.13 | 2.03 | 2% |
| PFBS | 104.89 | 3.93 | 4% |
| PFMBA | 104.09 | 4.66 | 4% |
| PFEESA | 104.92 | 3.91 | 4% |
| NFDHA | 112.38 | 7.87 | 7% |
| ¹³ C ₂ -4:2 FTS | 82.47 | 2.88 | 3% |
| 4:2 FTS | 91.46 | 3.04 | 3% |
| ¹³ C ₅ -PFHxA | 91.35 | 3.67 | 4% |
| PFHxA | 95.57 | 4.76 | 5% |
| PFPeS | 101.71 | 2.16 | 2% |
| ¹³ C ₃ -HFPO-DA | 93.37 | 2.23 | 2% |
| HFPO-DA | 103.16 | 3.27 | 3% |
| ¹³ C ₄ -PFHpA | 90.34 | 3.42 | 4% |
| PFHpA | 99.45 | 4.33 | 4% |
| ¹³ C ₃ -PFHxS | 91.22 | 4.46 | 5% |
| PFHxS | 102.05 | 2.73 | 3% |
| ADONA | 102.8 | 3.80 | 4% |
| ¹³ C ₂ -6:2 FTS | 84.55 | 2.58 | 3% |
| 6:2 FTS | 94.44 | 3.89 | 4% |
| ¹³ C ₈ -PFOA | 91.08 | 3.54 | 4% |
| PFOA | 102.9 | 4.60 | 4% |
| PFHpS | 103.84 | 5.91 | 6% |
| ¹³ C ₉ -PFNA | 89.96 | 3.05 | 3% |
| PFNA | 101.27 | 5.45 | 5% |
| ¹³ C ₈ -PFOS | 88.02 | 3.13 | 4% |
| PFOS | 102.97 | 5.48 | 5% |
| 9CI-PF3OUdS | 95.64 | 3.81 | 4% |
| ¹³ C ₂ -8:2 FTS | 83.6 | 2.37 | 3% |
| 8:2 FTS | 99.39 | 4.86 | 5% |
| ¹³ C ₆ -PFDA | 89.62 | 2.55 | 3% |
| PFDA | 97.12 | 4.22 | 4% |
| ¹³ C ₇ -PFUnA | 84.69 | 3.25 | 4% |
| PFUnA | 99.46 | 4.25 | 4% |
| 11Cl-PF3OUdS | 100.1 | 6.08 | 6% |
| ¹³ C ₂ -PFDoA | 82.21 | 2.36 | 3% |
| PFDoA | 101.45 | 4.30 | 4% |

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