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應用手冊

Repeatability and Reproducibility of the Oasis™ GCB/WAX for PFAS Analysis Cartridges in Soil/Solid Samples for EPA Method 1633

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Abstract

In January 2024, EPA Method 1633 was finalized. EPA Method 1633 is the first method to incorporate the determination of 40 PFAS compounds across many challenging environmental sample matrices outside of drinking water including non-potable waters (ground water, surface water, and wastewater) as well as soil, biosolids, and tissue by LC-MS/MS analysis. EPA Method 1633 requires use of both weak anion exchange (WAX) solid phase extraction with graphitized carbon black (GCB) cleanup and is a performance-based method allowing for modifications as long as acceptance criteria for recoveries and %RSDs are met.

This application note highlights the extraction of soil samples in EPA Method 1633 using the Oasis GCB/WAX for PFAS Analysis, a dual-phase solid-phase extraction cartridge. The results meet the method's acceptance criteria and demonstrate the reproducibility of the cartridges.

Benefits

- Oasis GCB/WAX for PFAS Analysis, a dual-phase cartridge is reproducible and repeatable for EPA Method

1633 soil samples in inter-batch assays

- Reduction of manual steps, overall sample preparation time by use of the dual-phase SPE cartridge
- Acceptance criteria for recoveries and %RSDs are met for EPA Method 1633 for soil samples

Introduction

With EPA Method 1633 finalized, this introduces the first comprehensive US EPA method focused outside of drinking water for PFAS determination.[1] As the method incorporates a wide variety of matrices including ground water, surface water, wastewater, soil, biosolids, and tissue, their sample preparation to ensure reproducibility, sensitivity, and robustness is critical. In EPA Method 1633, the sample preparation incorporates two sorbents, GCB and WAX.

For soil sample analysis, the GCB is packed on top of the WAX sorbent to replicate the EPA Method 1633 where the GCB is used to clean the sample prior to WAX SPE. However, the method is performance-based and gives requirements for establishing equivalency.[1] Oasis GCB/WAX for PFAS Analysis cartridges can alternatively be used and have been tested and shown to meet the acceptance criteria for multiple non-potable water sources as described previously.[2] Oasis GCB/WAX is a dual-phase, or bilayer cartridge which combines both sorbents into a single device benefitting the user by removing total manual steps in the sample preparation workflow and reducing time by up to 20% compared to use of loose GCB and a WAX cartridge. Additionally, Oasis GCB/WAX for PFAS Analysis undergoes a QC-batch release test for common PFAS to ensure cleanliness during the SPE process reducing the risk of false positives.

This application note uses Oasis GCB/WAX for PFAS Analysis on soil samples showing that the acceptance criteria for EPA Method 1633 is met across three separate product lots of GCB/WAX with six replicates within each lot. This demonstrates not only robustness of the SPE product in EPA 1633 workflows, but and the repeatability and reproducibility inter-batch of GCB/WAX cartridges ensures confidence in out-of-the-box performance of the cartridges for use with complex matrices, like those in EPA Method 1633.

Figure 1. Oasis GCB/WAX dual phase cartridge

for PFAS Analysis.

Experimental

LC-MS Analysis

UPLC:	ACQUITY™ UPLC™ I-Class Plus FTN, 50 µL Extension Loop
MPA:	2 mM ammonium acetate in water
MPB:	2 mM ammonium acetate in acetonitrile
Columns:	Analytical column: ACQUITY Premier BEH™ C_{18} 2.1 x 50 mm, 1.7 µm p/n: 186009452 Isolator column: Atlantis™ Premier BEH C18 AX 2.1 x 50 mm, 5.0 µm p/n: 186009407
Column temperature:	35 ˚C
Sample temperature:	8 ˚C
Injection volume:	2 µL
Wash solvent:	50:50 MeOH: H_2O
Purge solvent:	10:90 MeOH: H_2O

MS:	Xevo TQ-XS 01
Capillary voltage:	0.5 kV
Desolvation temperature:	350 °C
Desolvation flow:	900 L/hr
Vials:	700 µL Polypropylene Screw Cap Vials p/n: 186005219

UPLC Gradient Table

Time (min)	Flow (mL/min)	% MPA	% MPB	Curve
0	0.3	95	5	initial
0.5	0.3	75	25	6
3	0.3	50	50	6
6.5	0.3	15	85	6
7	0.3	5	95	6
8.5	0.3	5	95	6
9	0.3	95	5	6
11	0.3	95	5	6

Results and Discussion

High repeatability inter-batch from soil samples using dual-phase Oasis GCB/WAX for PFAS Analysis cartridges. Recoveries and RSD% meet acceptance criteria of EPA Method 1633.

Name	%Rec replicate 1	%Rec replicate 2	%Rec replicate 3	%Rec replicate 4	%Rec replicate 5	%Rec replicate 6	Mean recovery	1633 Recovery acceptance criteria
PFBA	110.3	106.3	131.3	107.5	106.4	103.3	111	70–140
PFPeA	102.6	106.9	127.1	107.8	99.1	104.9	108	70–140
PFHxA	106.3	102	133	100.2	106.1	95.4	107	70–135
PFHpA	99.1	103.6	120.5	103.2	96.1	95.7	104	70–140
PFOA	106.1	106.2	133.2	104.9	98.2	102.3	113	70–140
PFNA	104.4	113	137	118.8	116.5	102.2	115	65–145
PFUnDA	93.8	124.9	145.6	102.4	131.8	131.6	123	70–145
PFDoDA	106.9	97.4	129.9	101.9	103.7	111	108	70–145
PFDA	119.8	113.4	121.1	101.6	119.5	109.9	114	70–145
PFTriDA	100.6	87.7	114.4	101.3	100.6	105.1	102	55–160
PFTreDA	107.6	91.5	162.9	106.8	102.2	98.1	112	70–145
PFBS	104.9	99.6	116.8	97.3	88.2	84.1	98	60–145
PFPeS	102.9	112.6	124.6	100.4	95.5	98.2	99	65–140
PFHxS	97.9	109.3	117.1	94.5	101.3	87.7	101	65–145
PFHpS	116.6	112.4	118.4	90.8	88.8	95.6	104	70–140
PFOS	106.9	110.4	135.8	93.5	109.9	97.6	109	70–135
PFNS	107.5	117.8	111.7	88.9	92.2	93	102	70–140
PFDS	119.3	114.3	120.3	88.6	107.7	97.1	108	50–150
PFDoDS	93.7	87.4	95.8	81	114.3	88.5	89	40–140
GenX	111.4	103.6	144.7	118.6	103.2	111.5	116	70–140
ADONA	115.7	103.5	133.9	116.1	112	108.1	115	70–155
9ClPF3ONS	105.3	104.8	138.5	115.7	112.8	99.2	113	65–135
11ClPF3OUdS	106.7	97.7	123.3	103.7	119.5	100.4	109	50–135
4_2 FTS	105.6	95.8	107.4	94	117.5	94.8	103	70–135
6_2 FTS	132.1	138.2	163.9	133.8	122.9	129.7	137	60–160
8_2 FTS	113.1	92.5	110.5	85.3	100.8	109.8	102	70–140
FOSA	113	103.6	129.2	109.1	104.1	104.6	105	70–140
NMeFOSA	138.5	151.6	171.1	132.4	125.9	130.4	142	65–145
NEtFOSA	138.5	138	181.8	125.2	118.7	139.9	132	70–135
NMeFOSAA	104.4	104	154.6	111.5	98.4	107.7	113	60–145
NEtFOSAA	121.9	110.8	127.1	125.9	96.6	118.8	117	60–150
NMeFOSE	110.8	110.5	144.9	109.9	105.5	115.5	116	70–140
NEtFOSE	119.2	116	159.6	118.4	109.1	121.8	124	70–135
3:3 FTCA	90.1	99.7	117.6	91.5	97.3	92	98	45–155
5:3 FTCA	106.6	99.9	126.7	100.5	103	100.5	100	70–135
7:3 FTCA	103.8	90.1	117.5	96.5	102.7	93.8	90	70–145
PFMPA	106.6	105.6	123.8	101	92.1	99.1	107	70–140
PFMBA	106.8	101.4	129.8	104.4	97.7	99.2	107	55–145
Cholic acid	96.9	116.8	121.7	114.9	109.3	87.7	108	Not listed
PFEESA	100.3	98.8	127.9	101.5	91.9	97.4	103	70–140
NFDHA	103	102.5	129.8	100.6	97	87.2	103	45–145
M4 PFBA	83.1	111.1	85.7	119.4	91.2	106.9	100	8–130
M5_PFPeA	83	112.7	85.3	117.1	96.6	108.4	101	35–130
M5_PFHxA	85.2	118.5	83.7	117.4	103.6	108.8	103	40–130
M4_PFHpA	90.7	114.5	87.5	119	113.3	104.6	107	40–130
M8_PFOA	94.7	121.9	86.1	125	113.3	101.6	107	40–130
M9_PFNA	82.2	109.8	84.1	117.9	108.3	94.1	99	40–130
M6_PFDA	86.2	115.2	90.9	125.8	105.6	95	103	40–130
M7_PFUnDA	80.5	104.4	79	121.9	101.6	88.5	96	40–130
M_PFDoDA	85.4	109	81.1	113.1	112.1	91.6	99	40–130
M2_PFTreDA	71.8	85.8	60.9	94.9	105.6	85.6	84	20–130
M3_PFBS	73.4	110.7	81.5	117.4	90.3	105	99	40–135
M3_PFHxS	81.6	117.4	89.3	126.2	102	105.7	104	40–130
M8_PFOS	83.9	90.7	85	137.2	100.7	115.6	102	40–130
M2_42FTS	84.6	122	106.7	143.1	97.7	114.5	111	40–165
M2_62FTS	91.2	115.5	91.3	127.5	118.1	110.8	109	40–215
M2_82FTS	77.3	119.6	89.2	154.2	108.1	93.1	107	40–275
M8_FOSA	80	92	76.5	112	102.7	106.2	95	40–130
M3_GenX	81.8	112.5	78.1	104.6	96.2	95.9	95	40–130
D3_NMeFOSAA	73.2	82.7	62.9	99.2	95.8	95.3	85	40–135
D5_NEtFOSAA	67.9	80.6	67.1	85.2	90.9	95.2	81	40–150
dNMeFOSA	44.9	42.2	28.9	56.5	56.4	53.8	47	10–130
dNEtFOSA	40	42.6	27.6	60.7	55.4	50.1	46	10–130
d7 NMeFOSE	73.5	74	51.1	94	93.2	91.2	80	20–130
d9 NEtFOSE	68	69.7	46.3	86	86	84.3	73	15–130
M3 PFBA_NIS	77.8	76	68.6	71.8	65.1	73.1	72	50–200
M2 PFHxA_NIS	76.1	72	68.9	73.3	65.3	73.5	72	50–200
M4 PFOA_NIS	73.4	71	69.5	71.4	66.5	76.6	71	50–200
M5 PFNA_NIS	76.9	77	68.3	73.6	69.4	77.2	74	50–200
M2 PFDA_NIS	76	72.5	66.2	68.5	67.9	75.4	71	50–200
18O2 PFHxS_NIS	79.7	72.5	68.7	72.2	68	72.9	72	50–200
M4 PFOS_NIS	73.8	82.9	70.5	68.8	68	64.9	71	50–200

Figure 2. Demonstration of Oasis GCB/WAX for PFAS Analysis performance repeatability requiring no protocol optimization, with recovery

within 1633 acceptance criteria. Green displays values within 1633 acceptance criteria.

**Not listed: EPA 1633 does not provide acceptance criteria for this compound. Note: 2/6 replicates excluded for NEtFOSA (2/3 sample set excluded 140% mean recovery meets specification(70-130%).*

Name	%Rec replicate 1	%Rec replicate 2	%Rec replicate 3	%Rec replicate 4	%Rec replicate 5	%Rec replicate 6	Mean N=6 %RSD total	1633 % RSD acceptance criteria (≤)
PFBA	110.3	106.3	131.3	107.5	106.4	103.3	9	17
PFPeA	102.6	106.9	127.1	107.8	99.1	104.9	9	26
PFHxA	106.3	102	133	100.2	106.1	95.4	12	23
PFHpA	99.1	103.6	120.5	103.2	96.1	95.7	9	21
PFOA	106.1	106.2	133.2	104.9	98.2	102.3	12	23
PFNA	104.4	113	137	118.8	116.5	102.2	11	24
PFUnDA	93.8	124.9	145.6	102.4	131.8	131.6	16	26
PFDoDA	106.9	97.4	129.9	101.9	103.7	111	11	26
PFDA	119.8	113.4	121.1	101.6	119.5	109.9	7	25
PFTriDA	100.6	87.7	114.4	101.3	100.6	105.1	8	26
PFTreDA	107.6	91.5	162.9	106.8	102.2	98.1	23	24
PFBS	104.9	99.6	116.8	97.3	88.2	84.1	12	25
PFPeS	102.9	112.6	124.6	100.4	95.5	98.2	10	29
PFHxS	97.9	109.3	117.1	94.5	101.3	87.7	10	28
PFHpS	116.6	112.4	118.4	90.8	88.8	95.6	13	27
PFOS	106.9	110.4	135.8	93.5	109.9	97.6	14	27
PFNS	107.5	117.8	111.7	88.9	92.2	93	12	27
PFDS	119.3	114.3	120.3	88.6	107.7	97.1	12	31
PFDoDS	93.7	87.4	95.8	81	114.3	88.5	12	40
GenX	111.4	103.6	144.7	118.6	103.2	111.5	13	27
ADONA	115.7	103.5	133.9	116.1	112	108.1	9	50
9ClPF3ONS	105.3	104.8	138.5	115.7	112.8	99.2	12	27
11ClPF3OUdS	106.7	97.7	123.3	103.7	119.5	100.4	10	19
4_2 FTS	105.6	95.8	107.4	94	117.5	94.8	9	26
6_2 FTS	132.1	138.2	163.9	133.8	122.9	129.7	10	19
8_2 FTS	113.1	92.5	110.5	85.3	100.8	109.8	11	26
FOSA	113	103.6	129.2	109.1	104.1	104.6	9	19
NMeFOSA	138.5	151.6	171.1	132.4	125.9	130.4	12	28
NEtFOSA	138.5	138	181.8	125.2	118.7	139.9	16	19
NMeFOSAA	104.4	104	154.6	111.5	98.4	107.7	18	31
NEtFOSAA	121.9	110.8	127.1	125.9	96.6	118.8	10	31
NMeFOSE	110.8	110.5	144.9	109.9	105.5	115.5	12	19
NEtFOSE	119.2	116	159.6	118.4	109.1	121.8	14	17
3:3 FTCA	90.1	99.7	117.6	91.5	97.3	92	10	32
5:3 FTCA	106.6	99.9	126.7	100.5	103	100.5	10	28
7:3 FTCA	103.8	90.1	117.5	96.5	102.7	93.8	9	39
PFMPA	106.6	105.6	123.8	101	92.1	99.1	10	25
PFMBA	106.8	101.4	129.8	104.4	97.7	99.2	11	33
Cholic acid	96.9	116.8	121.7	114.9	109.3	87.7	12	Not listed
PFEESA	100.3	98.8	127.9	101.5	91.9	97.4	12	20
NFDHA	103	102.5	129.8	100.6	97	87.2	14	27
M4 PFBA	83.1	111.1	85.7	119.4	91.2	106.9	15	Not listed
M5_PFPeA	83	112.7	85.3	117.1	96.6	108.4	14	Not listed
M5_PFHxA	85.2	118.5	83.7	117.4	103.6	108.8	15	Not listed
M4_PFHpA	90.7	114.5	87.5	119	113.3	104.6	13	Not listed
M8_PFOA	94.7	121.9	86.1	125	113.3	101.6	14	Not listed
M9_PFNA	82.2	109.8	84.1	117.9	108.3	94.1	15	Not listed
M6_PFDA	86.2	115.2	90.9	125.8	105.6	95	15	Not listed
M7_PFUnDA	80.5	104.4	79	121.9	101.6	88.5	17	Not listed
M_PFDoDA	85.4	109	81.1	113.1	112.1	91.6	15	Not listed
M2_PFTreDA	71.8	85.8	60.9	94.9	105.6	85.6	19	Not listed
M3_PFBS	73.4	110.7	81.5	117.4	90.3	105	18	Not listed
M3_PFHxS	81.6	117.4	89.3	126.2	102	105.7	16	Not listed
M8_PFOS	83.9	90.7	85	137.2	100.7	115.6	20	Not listed
M2_42FTS	84.6	122	106.7	143.1	97.7	114.5	18	Not listed
M2_62FTS	91.2	115.5	91.3	127.5	118.1	110.8	14	Not listed
M2_82FTS	77.3	119.6	89.2	154.2	108.1	93.1	26	Not listed
M8_FOSA	80	92	76.5	112	102.7	106.2	15	Not listed
M3_GenX	81.8	112.5	78.1	104.6	96.2	95.9	14	Not listed
D3_NMeFOSAA	73.2	82.7	62.9	99.2	95.8	95.3	17	Not listed
D5_NEtFOSAA	67.9	80.6	67.1	85.2	90.9	95.2	14	Not listed
dNMeFOSA	44.9	42.2	28.9	56.5	56.4	53.8	23	Not listed
dNEtFOSA	40	42.6	27.6	60.7	55.4	50.1	26	Not listed
d7 NMeFOSE	73.5	74	51.1	94	93.2	91.2	21	Not listed
d9 NEtFOSE	68	69.7	46.3	86	86	84.3	21	Not listed
M3 PFBA_NIS	77.8	76	68.6	71.8	65.1	73.1	7	Not listed
M2 PFHxA_NIS	76.1	72	68.9	73.3	65.3	73.5	5	Not listed
M4 PFOA_NIS	73.4	71	69.5	71.4	66.5	76.6	5	Not listed
M5 PFNA_NIS	76.9	77	68.3	73.6	69.4	77.2	5	Not listed
M2 PFDA_NIS	76	72.5	66.2	68.5	67.9	75.4	6	Not listed
18O2 PFHxS_NIS	79.7	72.5	68.7	72.2	68	72.9	6	Not listed
M4 PFOS_NIS	73.8	82.9	70.5	68.8	68	64.9	9	Not listed

Figure 3. Demonstration of Oasis GCB/WAX for PFAS Analysis performance repeatability requiring no protocol optimization, with %RSD

within 1633 acceptance criteria. Green displays values within 1633 acceptance criteria.

**Not listed: EPA 1633 does not provide% RSD criteria for this compound.*

Conclusion

This study demonstrates the reproducibility of the dual-phase Oasis GCB/WAX for PFAS Analysis cartridges for determination of 40 PFAS and standards using the ACQUITY UPLC I-Class System and Xevo TQ-XS mass spectrometer. The cartridges are suitable for PFAS analysis in accordance with EPA 1633 guidelines for recovery and % RSD. The GCB/WAX cartridges show excellent repeatability across multiple replicates across soil samples. The data demonstrates Oasis GCB/WAX for PFAS Analysis cartridges are ideally suited for PFAS analysis from complex matrices, such as soils and solid samples like those described in EPA Method 1633. Out of the box performance is expected lot to lot and within lot for SPE when using Oasis GCB/WAX for PFAS Analysis.

References

1. Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Soil, Solid, Biosolids, and Tissue Samples by LC-MS/MS, *US Environmental Protection Agency*. Final version. 31 January 2024.
2. Organtini, K.; Rosnack, K.; Plummer, C.; Hancock, P.; Burt, O. Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Accordance With EPA 1633 Part 2: Analysis of Soil Matrices. Waters Application Note. 720008143. 2023.
3. Organtini, K.; Rosnack, K.; Plummer, C.; Hancock, P.; Burt, O. Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Accordance with EPA 1633 Part 3: Analysis of Soil and Tissue. Waters Application Note. 720008230 . 2024.

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