

Interlaboratory Study for PFAS Background Extracted from Multiple Pre-cleaned XAD-2 Adsorbent Lots

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Introduction

XAD[®]-2 is a macroporous, nonionic polymeric adsorbent resin composed of polystyrene cross-linked with divinylbenzene, developed in the early 1970s by Rohm and Haas for use in chromatographic and environmental applications. Characterized by a high surface area and a hydrophobic, aromatic matrix, XAD[®]-2 exhibits strong affinity for non-polar to moderately polar organic compounds.

Day 1	Soak with water 5 min, remove fines, soak w/water overnight
Day 2	Hot water, extract 8 hrs
Day 3	Methanol, extract 22 hr
Day 4	Methylene Chloride, extract 22 hr
Day 5	Ammonium hydroxide in methanol, extract 22 hr

Figure 1. Cleaning of XAD[®]-2 resin from OTM-45 Appendix.

There are 2x, 20-40 g cartridges of XAD[®]-2 specified by OTM-45 sampling train (Figure 2), highlighted in green (1) and pink (2).

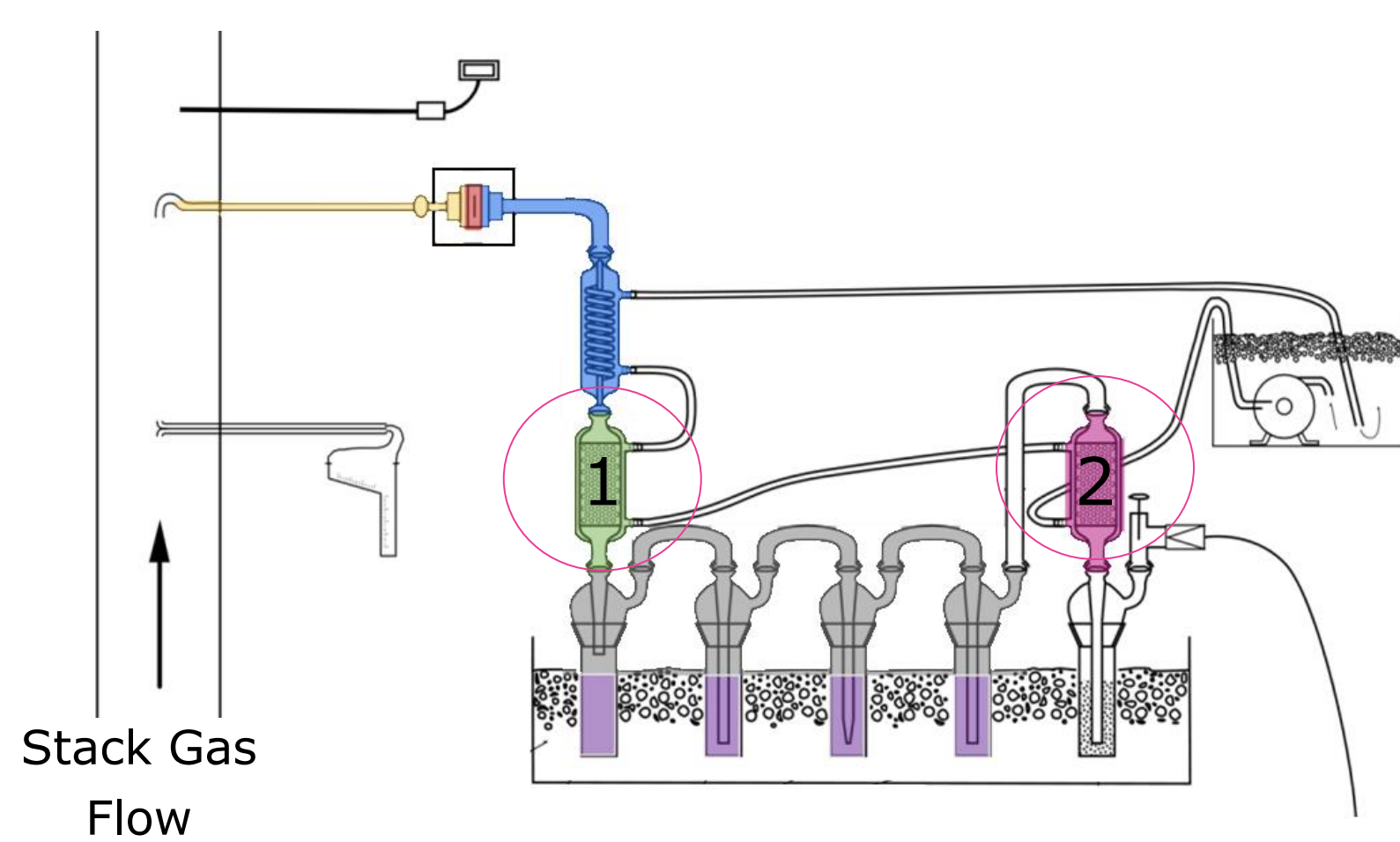


Figure 2. OTM-45 Sampling Train, Measurement of select per- and polyfluorinated alkyl substances from stationary sources.

Supelpak[™]-2SVM is a clean version of XAD[®]-2 ready for analysis of semi-volatiles for various methods including EPA 10 – Semivolatiles in Stationary Source Emissions, EPA TO-13A PAHs in Ambient Air, EPA 23 – Dioxins in Stationary Source Emission, and ASTM D6209 – PAHs in Ambient Air. However, background levels of PFAS contaminations were not low enough to meet the requirements of the environmental labs.

Method

To prepare the XAD[®]-2 resin for use for PFAS analysis, we first pre-cleaned it using a more sustainable process compared to OTM-45 appendix described, and then immediately analyzed the cleaned batches using the extraction procedures outlined in Method OTM-45. Batches that showed low background levels of PFAS were then sent out to multiple certified PFAS-testing laboratories for confirmation of cleanliness.

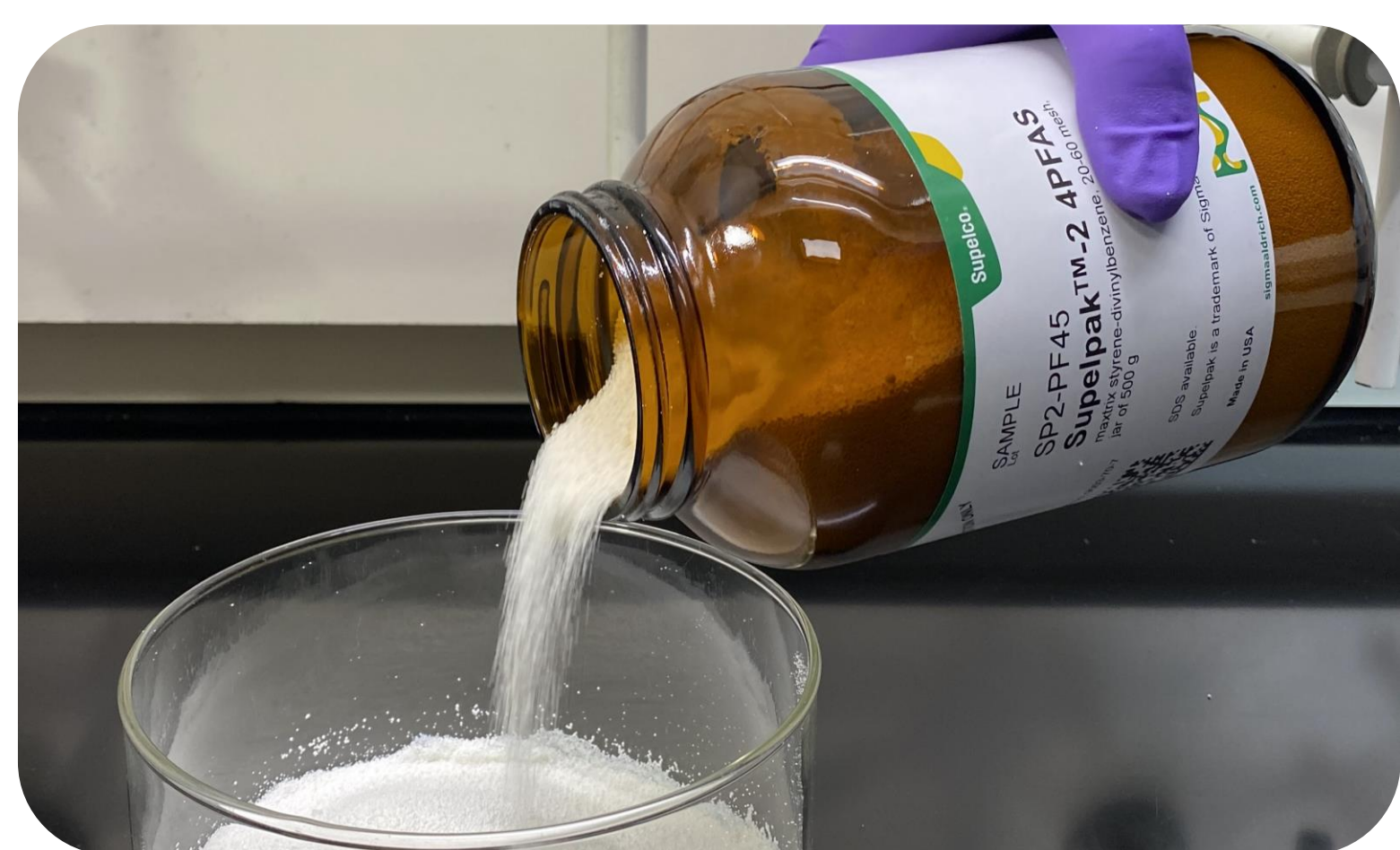


Figure 4. Supelpak[™]-2 4PFAS, a ready-to-use cleaned XAD[®]-2 resin for PFAS analysis

Sample Preparation

The exact method to extract PFAS from XAD[®]-2 was not held constant between laboratories. Each lab did use a verified method. The general method outlined in OTM-45 Rev 1 is listed below (key differences are highlighted in Table 1).

- 2x Extraction of XAD[®]-2 resin with MeOH+NH₄OH (1.5% v/v)
- Combine fractions, use 90 mL of extract, neutralize and bring up to 1 L w/ water
- Concentrate to <10 mL, then <2 mL (not dryness)
- SPE with WAX and GCB
- Analysis by LC-MS/MS



Table 1. Overview of methods utilized by the various laboratories.

	Overview of Laboratories Parameters					
	L1	L2	L3	L4	L5a	L5b
Mass resin (g)	30	20	30	20	20	20
Method	OTM-45 v1	OTM-45 v1	OTM-45 v1	OTM-45 v0	OTM-45 v0	OTM-45 v0
Enrichment	SPE	SPE	SPE	Evaporate	Evaporate	ASE

Table 2. LC-MS conditions utilized by one of the laboratories for analysis.

LC-MS Conditions	
Instrument:	Agilent Infinity Series II & SCIEX Triple Quad 6500+
Column:	Ascentis [®] Express 90 Å PFAS, 2.7 µm, 100 x 2.1 mm I.D.
Delay Column:	Ascentis [®] Express 160 Å PFAS, 2.7 µm, 50 x 3.0 mm I.D.
Detector:	MS/MS, MRM, ESI(-)
Injection:	5 µL
Other parameters available upon request	

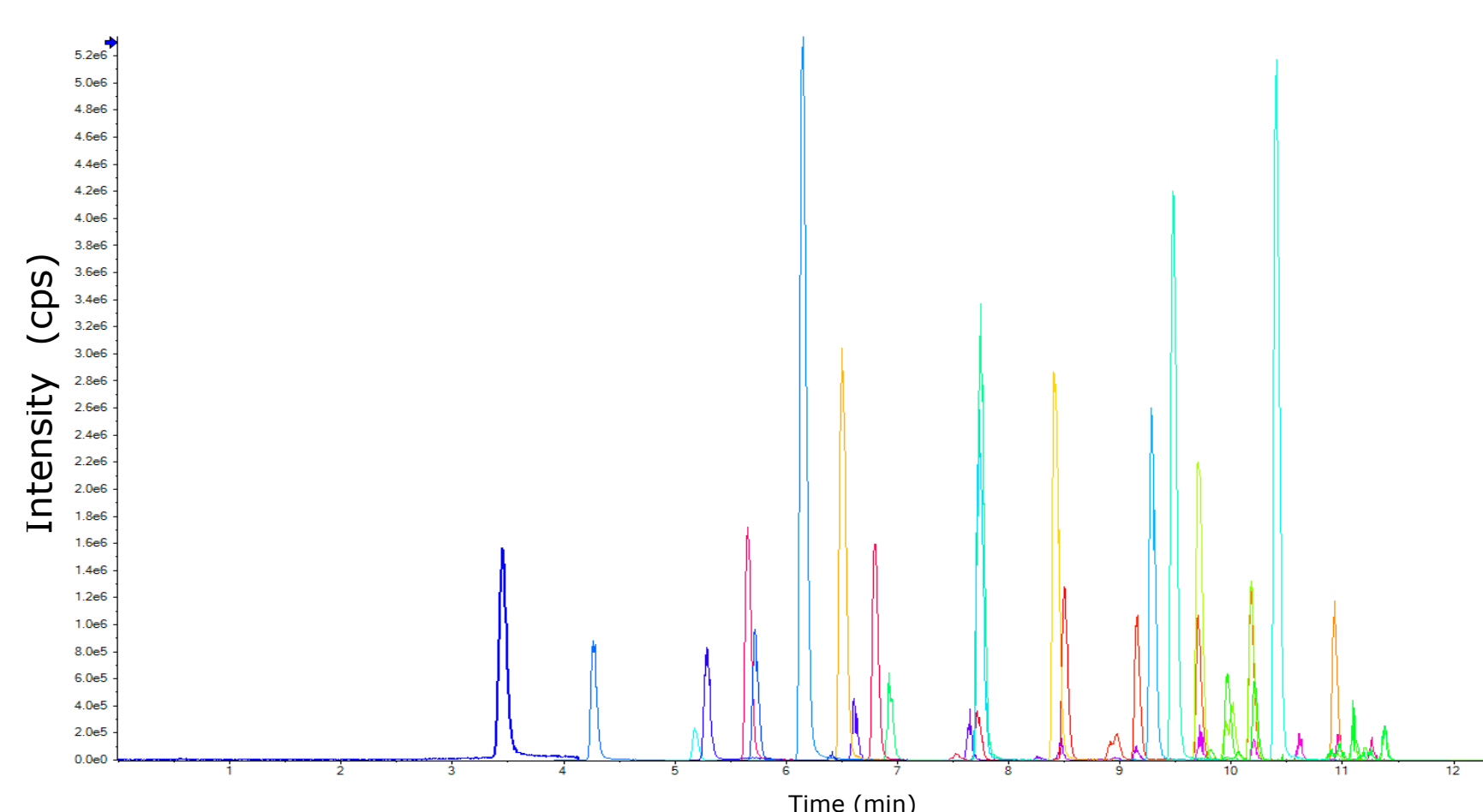


Figure 3. Representative extracted PFAS chromatogram (MRM).

Results

Four of the five laboratories reported non-detect for all the targeted PFAS compounds outlined. One laboratory (Lab 4), detected two analytes slightly above their report limits (Table 3).

Table 3. Highlighted PFAS compounds above reporting limit for Lab 4.

	Concentration (pg/g)		
	Sample 1	Sample 2	Reporting Limit
PFHxA	<RL	26.12	25
PFOA	<RL	27.18	25
All other analytes	<RL	<RL	25

Discussion

Four of the five laboratories that participated in this study reported non-detects for the targeted PFAS analytes specified. While this outcome strongly supports the effectiveness of the pre-cleaning process and indicates a very low background contamination level in the cleaned XAD[®]-2 resin, it's worth noting that the reporting limits (Table 4) as well as isotope standard recoveries (Table 5) varied across laboratories. These differences in detection sensitivity could reflect variations in instrumentation, methodology nuances, or lab-specific protocols.

Table 4. Reporting limits (RL) for various PFAS analytes by lab

Analyte	Reporting Limit (pg/g)					Analyte	Reporting Limit (pg/g)				
	L1	L2	L3	L4	L5		L1	L2	L3	L4	L5
PFBA	333	13.3	4.4	25	200	N-MeFOSAA	33	3.3	1.1	25	300
PFPeA	33	6.7	0.6	25	200	N-EtFOSE	33	3.3	0.6	25	10
PFHxA	100	3.3	1.1	25	200	4:2 FTS	33	13	0.6	25	200
PFHpA	33	3.3	0.6	25	200	6:2 FTS	33	13	2.2	25	200
PFOA	33	3.3	0.6	25	200	8:2 FTS	33	13	1.1	25	200
PFNA	33	3.3	0.6	25	200	10:2 FTS	33	-	1.1	25	-
PFDA	33	3.3	0.6	25	200	ADONA	100	13	0.6	25	200
PFUnA	33	3.3	0.6	25	200	HFPO-DA	166	13	0.6	25	500
PFDoA	33	3.3	1.1	25	300	9CI-PF3ONS	33	13	0.6	25	500
PFTra	33	3.3	1.1	25	200	11Cl-PF3OUdS	33	13	0.6	25	500
PFTeDA	33	3.3	1.1	25	200	NFDHA	33	6.7	0.6	25	3
PFHxDA	33	-	0.6	25	-	PFEESA	33	6.7	0.6	25	3
PFODA	33	-	1.1	25	-	PFMBA	33	6.7	0.6	25	3
PFBS	33	3.3	0.6	25	200	PFMPA	33	6.7	0.6	25	3
PFPeS	33	3.3	0.6	25	200	PFECHS	33	-	0.6	25	-
PFHxS	33	3.3	0.6	25	200	6:2 FTUCA	33	-	2.2	25	-
PFHpS	33	3.3	0.6	25	200	8:2 FTUCA	33	-	2.2	25	-
PFOS	33	3.3	0.6	25	200	10:2 FTUCA	33	-	2.2	25	-
PFNS	33	3.3	0.6	25	300	3:3 FTCA	33	17	2.2	25	3
PFDS	33	3.3	0.6	25	300	5:3 FTCA	33	83	1.1	25	3
PFDoS	33	3.3	1.1	25	300	7:3 FTCA	33	83	1.1	25	3
FOSA	33	3.3	0.6	25	200	6:2 FTCA	33	-	4.4	25	-
N-MeFOSA	33	3.3	1.1	25	20	8:2 FTCA	33	-	4.4	25	-
N-EtFOSA	33	3.3	1.1	25	20	10:2 FTCA	33	-	4.4	25	-

Also considered was the recovery of isotope dilution standards as outlined within OTM-45. Shared below are the averaged recoveries from two samples being sent to each lab.

Table 5. Isotope dilution standards recoveries

Isotope Standard	Recovery %				
	L1	L2	L3	L5A	L5B
¹³ C-Perfluorobutanoic acid	87	92	73	90	73
¹³ C-Perfluoropentanoic acid	85	85	77	86	72
¹³ C-Perfluorohexanoic acid	82	87	69	92	70
¹³ C-Perfluoroheptanoic acid	81	83	68	56	57
¹³ C-Perfluorooctanoic acid	88	87	74	88	67
¹³ C-Perfluorononanoic acid	85	94	78	94	68
¹³ C-Perfluorodecanoic acid	60	72	74	92	69
¹³ C-Perfluoroundecanoic acid	58	59	48	86	72
¹³ C-Perfluorododecanoic acid	116	51	55	84	74
¹³ C-Perfluorotetradecanoic acid	25	56	-	71	65
¹³ C-Perfluorobutanesulfonate	71	86	75	94	57
¹³ C-Perfluorohexanesulfonate	72	87	69	86	72
¹³ C-Perfluorooctanesulfonate	60	67	77	92	75
¹³ C-Hexafluoropropylene oxide	69	88	48	88	60
d ₃ -N-Methyl-PFOS-amidoacetic acid	85	99	-	98	73
d ₅ -N-Ethyl-PFOS-amidoacetic acid	76	102	-	105	66
¹³ C-Perfluorooctanesulfonamide	64	89	-	99	73
d ₂ -N-ethyl PFOS-amide	29	52	-	19	26
d ₃ -N-methyl-PFOS-amido ethanol	39	47	-	12	34
d ₅ -N-ethyl PFOS-amido ethanol	41	65	-	42	35
¹³ C-Perfluorooctanesulfonamide	30	61	-	43	37
¹³ C-4:2 Fluorotelomer sulfonate	116	113	95	103	108
¹³ C-6:2 Fluorotelomer sulfonate	120	120	71	136	96
¹³ C-8:2 Fluorotelomer sulfonate	118	144	78	110	109

Conclusions

Validation of Resin Cleanliness:

Consistent non-detect results across multiple independent labs suggest that the cleaned XAD[®]-2 material is suitable for use in ultra-trace PFAS air sampling applications, without further purification reducing false positives due to background contamination.

The CoA for this cleaned XAD[®]-2 product, Supelpak[™]-2 4PFAS will provide the reporting limits and contaminant level for each PFAS analyte listed within OTM-45.

Enhanced Confidence in Field Deployments:

When validated, such low-background sorbents could streamline sampling logistics by minimizing the need for extensive pre-treatment or post-collection sample screening.

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