Update on ASTM WK88581 - New Test Method for Determination of Extractable Per- and Polyfluoroalkyl Substances (PFAS) in Solid Matrices by Solvent Extraction followed by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS)

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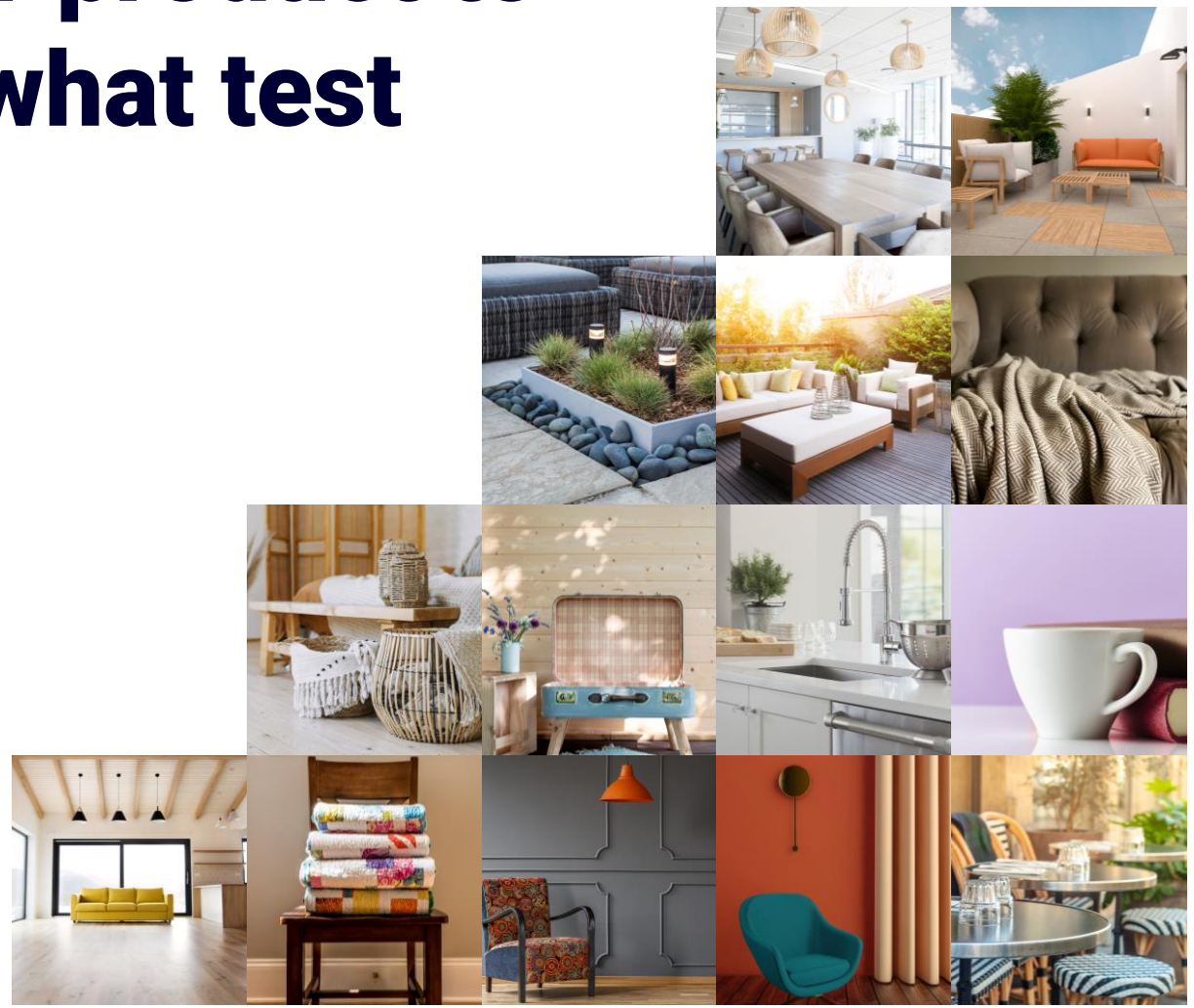
If you had to send your product to a lab for a PFAS test, what test would you get?

- Drinking Water – 533, 537
- Other Water – 1633 or 8421
- Soil – 1633 or 8535



Project Summary

Goal

- Develop a standardized, validated method for testing consumer products for targeted, extractable PFAS analytes.
- Method should be easy for laboratories to implement.
  - Co-solvation and large volume injection eliminates need for SPE or sample cleanup
  - No *a priori* information is required about sample
- Method will include several matrices applicable to a range of consumer products

Sample Preparation

Co-solvation. 0.5 grams of sample is placed in 10 mL basic 50:50 MeOH:Water

Tumble. End-over-end tumbling for 2 hours

Filter. Filter particulate with PP syringe filter, acidify filtrate

Centrifuge. Centrifuge at 3000 rpm at 8 °C for 15 minutes, aliquot into LC vial

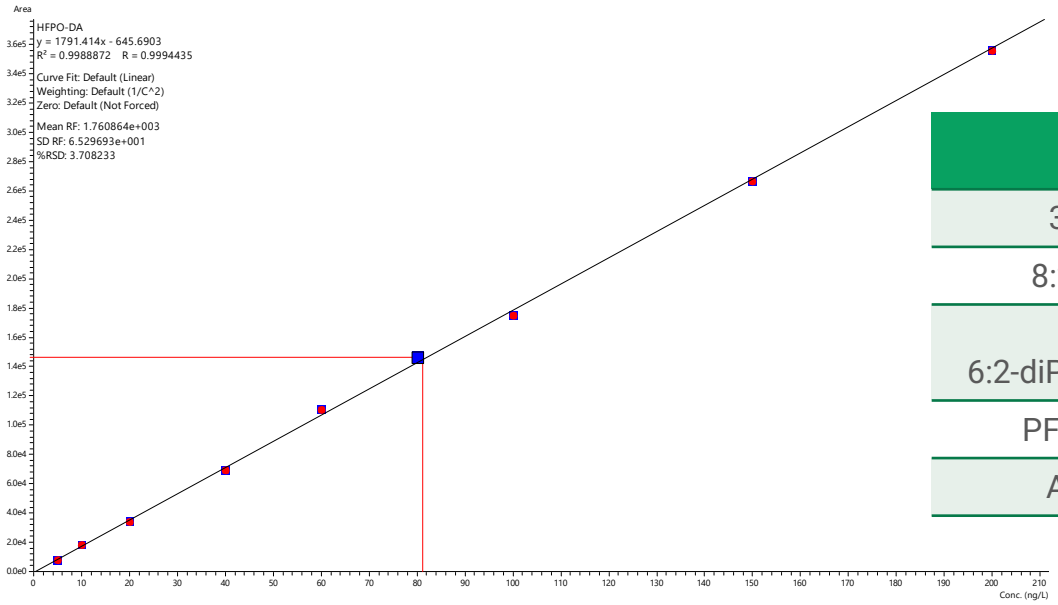
Method Details

- Used ASTM 8535 as initial framework
- External calibration with 46 target analytes and 25 isotopically labeled surrogates
  - 1633 list plus 6:2 dipap, 8:2 dipap, PFPrA, HQ-115, FHUEA and FOUEA.
- Large volume injection for detection limits as low as 100ng/kg for most analytes
  - Coinjection helps improve peak shape for large volume injections

$$C_s\left(\frac{ng}{kg}\right) = \frac{\left[C_i\left(\frac{ng}{L}\right)\right] \times [V_s\,(L)]}{[W_d\,(kg)]}$$

where:

C_s = concentration of target analyte in sample,
C_i = concentration of target analyte in sample from instrument,
V_s = volume of sample, and
W_d = dry weight of sample.

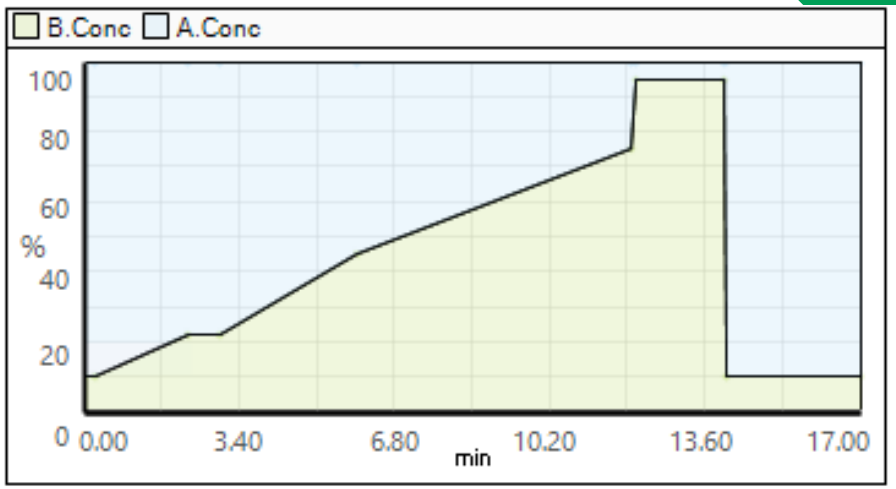


Analyte	Cal 1 (ng/L)	Cal 2 (ng/L)	Cal 3 (ng/L)	Cal 4 (ng/L)	Cal 5 (ng/L)	Cal 6 (ng/L)	Cal 7 (ng/L)	Cal 8 (ng/L)	Cal 9 (ng/L)
3:3 FTCA	--	--	20	40	60	80	100	150	200
8:2, 4:2 FTS	--	10	20	40	60	80	100	150	200
PFPeA, 6:2-diPAP, 8:2-diPAP	25	50	100	200	300	400	500	750	1000
PFPrA, PFBA	--	50	100	200	300	400	500	750	1000
All others	5	10	20	40	60	80	100	150	200

Analytical Details

Shimadzu Nexera LC With 8060NX MS

- Delay Column: C18 - 5um 50mm
- Analytical Column: C18 - 120 3um x 100mm
- 40 uL injection
- Flow Rate: 0.45 mL/min
- Coinjection – 25 uL 0.1% acetic acid, 20uL sample, 25 uL 0.1% acetic acid, 20uL sample
- Mobile phases:
- A – 95%/5% Water / Acetonitrile, 2 mM Ammonium Acetate
- B – Acetonitrile



Method Is Instrument Agnostic

Two Phased Approach

Phase 1

Spiked articles from six matrices with surrogates and measured recovery.

Matrices tested:

1. Paper
    1. Cardboard
    2. Non-cardboard
2. Fabric
3. Plastic
4. Cosmetics
5. Metal
6. Other

Phase 2

Spiked one article from each of six matrices with native PFAS analytes in triplicate at low, medium, and high concentrations and measured recovery.

Matrices tested:

1. Cosmetic
2. Non-Stick Foil
3. Carboard
4. Plastic
5. Athletic Tape
6. Dress Shirt

*n=3 for all tests

Low=600ng/kg

med=1800 ng/kg

high=3500 ng/kg

Phase 1 – Surrogate Spiking of Matrices

Matrices	Samples
Paper n=14	
Cardboard n=6	Pizza box, water cone cup, to-go box (x2), leak proof bowl, shipping box
Non-Cardboard n=8	Coffee cup, popcorn bag, waterproof labels (x2), post-it note, compostable bowl, bath tissue, parchment paper
Fabric n=5	Men's dress shirts(x2), carpet remnants, cotton pad, bed mat
Plastic n=4	Pet food bag, gas can, straw, food wrapper
Cosmetics n=3	Drawing pen, powder, eye liner
Metal n=2	Coated pan, non-stick foil
Other n=6	Dental floss(x2), athletic tape, contact lens, pet food, AFFF

Phase 1 – Surrogate Spiking

All recoveries are for samples spiked with surrogates only.

	Plastic n=4		Fabric n=5		Cosmetics n=3	
Analytes	**Average**	**RSD**	**Average**	**RSD**	**Average**	**RSD**
13C4-PFBA	114%	13%	126%	8%	109%	4%
13C5-PFPeA	112%	8%	123%	6%	109%	5%
13C5-PFHxA	104%	7%	119%	3%	100%	12%
13C4-PFHpA	103%	8%	126%	5%	103%	9%
13C8-PFOA	109%	14%	120%	10%	128%	10%
13C9-PFNA	108%	3%	131%	13%	109%	23%
13C6-PFDA	109%	2%	133%	12%	109%	14%
13C7-PFUnA	104%	5%	130%	7%	99%	26%
13C2-PFDoA	121%	13%	162%	10%	104%	18%
13C2-PFTreA	127%	17%	139%	13%	70%	35%
13C8-PFOSA	100%	5%	110%	23%	92%	25%
D3-NMeFOSAA	108%	6%	168%	10%	102%	36%
D5-NEtFOSAA	113%	5%	196%	38%	105%	27%
D3-NMeFOSA	95%	8%	87%	25%	55%	58%
D5-NEtFOSA	89%	11%	87%	28%	47%	73%
D7-NMeFOSE	93%	8%	61%	49%	45%	34%
D9-NEtFOSE	80%	17%	53%	66%	34%	51%
13C3-HFPO-DA	110%	14%	112%	6%	104%	14%
13C2-4:2FTS	106%	6%	141%	25%	96%	10%
13C2-6:2FTS	108%	8%	206%	51%	96%	19%
13C2-8:2FTS	93%	10%	189%	53%	112%	9%
13C8-PFOS	103%	6%	102%	18%	96%	8%
13C3-PFBS	116%	3%	116%	8%	110%	6%
13C3-PFHxS	102%	7%	103%	12%	93%	7%

Phase 1 – Surrogate Spiking

All recoveries are for samples spiked with surrogates only.

	Metal n=2		Other n=6	
Analytes	**Average**	**RSD**	**Average**	**RSD**
13C4-PFBA	109%	18%	108%	21%
13C5-PFPeA	108%	14%	103%	14%
13C5-PFHxA	98%	8%	99%	18%
13C4-PFHpA	97%	16%	91%	27%
13C8-PFOA	103%	14%	101%	16%
13C9-PFNA	93%	10%	110%	36%
13C6-PFDA	99%	15%	105%	22%
13C7-PFUnA	92%	2%	87%	42%
13C2-PFDoA	93%	8%	80%	49%
13C2-PFTreA	83%	22%	48%	89%
13C8-PFOSA	92%	11%	100%	17%
D3-NMeFOSAA	101%	10%	110%	39%
D5-NEtFOSAA	103%	8%	143%	65%
D3-NMeFOSA	99%	5%	76%	32%
D5-NEtFOSA	94%	5%	63%	38%
D7-NMeFOSE	87%	2%	46%	84%
D9-NEtFOSE	79%	7%	38%	105%
13C3-HFPO-DA	102%	8%	91%	24%
13C2-4:2FTS	86%	21%	104%	12%
13C2-6:2FTS	96%	27%	102%	29%
13C2-8:2FTS	87%	22%	90%	18%
13C8-PFOS	101%	4%	83%	35%
13C3-PFBS	100%	2%	97%	14%
13C3-PFHxS	91%	8%	96%	14%

Phase 1 – Surrogate Spiking: Paper

All recoveries are for samples spiked with surrogates only.

	Paper (all) n=14		Paper (non-cardboard) n=8		Paper (cardboard) n=6	
Analytes	**Average**	**RSD**	**Average**	**RSD**	**Average**	**RSD**
13C4-PFBA	105%	31%	96%	41%	117%	11%
13C5-PFPeA	111%	19%	106%	40%	119%	10%
13C5-PFHxA	102%	22%	102%	43%	102%	7%
13C4-PFHpA	110%	25%	110%	45%	109%	21%
13C8-PFOA	109%	19%	108%	41%	110%	18%
13C9-PFNA	105%	22%	97%	43%	115%	7%
13C6-PFDA	118%	27%	107%	46%	133%	14%
13C7-PFUnA	114%	33%	97%	52%	136%	11%
13C2-PFDoA	102%	39%	89%	59%	120%	23%
13C2-PFTreA	72%	53%	52%	63%	98%	29%
13C8-PFOSA	104%	28%	95%	48%	115%	6%
D3-NMeFOSAA	133%	33%	117%	56%	155%	13%
D5-NEtFOSAA	172%	43%	124%	52%	235%	20%
D3-NMeFOSA	60%	54%	61%	67%	59%	18%
D5-NEtFOSA	75%	40%	69%	58%	83%	12%
D7-NMeFOSE	53%	53%	50%	69%	56%	29%
D9-NEtFOSE	41%	67%	39%	84%	44%	38%
13C3-HFPO-DA	89%	34%	97%	37%	80%	52%
13C2-4:2FTS	239%	52%	192%	66%	301%	35%
13C2-6:2FTS	293%	53%	254%	79%	346%	42%
13C2-8:2FTS	179%	59%	121%	57%	255%	40%
13C8-PFOS	91%	27%	86%	44%	98%	13%
13C3-PFBS	87%	25%	88%	42%	87%	17%
13C3-PFHxS	91%	22%	92%	41%	89%	16%

Duplicates

All recoveries are for samples spiked with surrogates only.

	Pizza Box	To Go Box	Coffee Cup	Popcorn Bag	Compostable Bowl
Analytes	**RSD**	**RSD**	**RSD**	**RSD**	**RSD**
13C4-PFBA	3%	0%	10%	7%	3%
13C5-PFPeA	0%	1%	3%	1%	2%
13C5-PFHxA	4%	8%	8%	4%	7%
13C4-PFHpA	2%	5%	2%	3%	4%
13C8-PFOA	2%	4%	10%	2%	3%
13C9-PFNA	2%	7%	10%	3%	5%
13C6-PFDA	1%	1%	5%	1%	4%
13C7-PFUnA	3%	1%	2%	1%	3%
13C2-PFDoA	1%	1%	4%	1%	4%
13C2-PFTreA	3%	4%	6%	0%	9%
13C8-PFOSA	2%	1%	2%	8%	5%
D3-NMeFOSAA	1%	2%	3%	4%	8%
D5-NEtFOSAA	5%	1%	5%	2%	1%
D3-NMeFOSA	2%	3%	22%	2%	11%
D5-NEtFOSA	6%	3%	5%	2%	0%
D7-NMeFOSE	3%	3%	11%	6%	3%
D9-NEtFOSE	0%	5%	1%	5%	1%
13C3-HFPO-DA	6%	4%	9%	8%	6%
13C2-4:2FTS	3%	5%	2%	2%	10%
13C2-6:2FTS	2%	4%	10%	10%	2%
13C2-8:2FTS	4%	4%	1%	1%	22%
13C8-PFOS	4%	4%	10%	2%	8%
13C3-PFBS	4%	1%	11%	8%	5%
13C3-PFHxS	3%	2%	5%	3%	13%

Two Phased Approach

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3. Plastic
4. Cosmetics
5. Metal
6. Other

Phase 2

Spiked one article from each of six matrices with native PFAS analytes in triplicate at low, medium, and high concentrations and measured recovery.

Matrices tested:

1. Cosmetic
2. Non-Stick Foil
3. Carboard
4. Plastic
5. Athletic Tape
6. Dress Shirt

*n=3 for all tests

Low=600ng/kg

med=1800 ng/kg

high=3500 ng/kg

Phase 2 – Native Spiking: Nonstick Foil

Low Range Spike

Analyte	Average Recovery	%RSD
PFTeDA	114%	12%
PFTrDA	117%	8%
PFDoA	149%	3%
PFUnA	111%	16%
PFDA	118%	11%
PFNA	101%	19%
PFOA	86%	20%
PFHpA	118%	11%
PFHxA	136%	9%
PFPeA	108%	6%
PFBA	103%	8%
PFDS	124%	7%
PFNS	113%	6%
PFOS	104%	12%
PFHpS	121%	22%
PFHxS	108%	17%
PFPeS	125%	2%
PFBS	130%	7%
PFOSA	104%	13%
8:2 FTS	114%	16%
6:2 FTS	96%	10%
4:2 FTS	117%	5%
NEtFOSAA	116%	25%

Analyte	Average Recovery	%RSD
NMeFOSAA	124%	13%
PFDoS	115%	2%
NMeFOSA	118%	5%
NEtFOSA	108%	7%
NMeFOSE	101%	4%
NEtFOSE	115%	4%
HFPO-DA	120%	3%
ADONA	127%	2%
9Cl-PF3ONS	113%	5%
11Cl-PF3OUdS	112%	5%
PFPrA	141%	9%
NFDHA	126%	3%
PFEESA	123%	5%
PFMPA	134%	4%
PFMBA	126%	6%
3:3 FTCA	--	--
5:3 FTCA	111%	12%
7:3 FTCA	93%	12%
FHUEA	118%	5%
FOUEA	127%	6%
HQ-115	137%	3%
6:2 diPAP	108%	11%
8:2 diPAP	178%	49%

Mid Range Spike

Analyte	Average Recovery	%RSD
PFTeDA	111%	8%
PFTrDA	101%	8%
PFDoA	120%	1%
PFUnA	101%	6%
PFDA	94%	9%
PFNA	92%	7%
PFOA	90%	3%
PFHpA	103%	2%
PFHxA	103%	3%
PFPeA	101%	2%
PFBA	99%	1%
PFDS	99%	3%
PFNS	104%	2%
PFOS	98%	3%
PFHpS	107%	3%
PFHxS	99%	6%
PFPeS	100%	3%
PFBS	103%	1%
PFOSA	99%	4%
8:2 FTS	92%	14%
6:2 FTS	85%	4%
4:2 FTS	92%	5%
NEtFOSAA	97%	9%

Analyte	Average Recovery	%RSD
NMeFOSAA	108%	1%
PFDoS	104%	2%
NMeFOSA	95%	2%
NEtFOSA	93%	3%
NMeFOSE	91%	1%
NEtFOSE	92%	1%
HFPO-DA	104%	2%
ADONA	103%	2%
9Cl-PF3ONS	96%	1%
11Cl-PF3OUdS	102%	2%
PFPrA	117%	5%
NFDHA	107%	2%
PFEESA	95%	4%
PFMPA	89%	2%
PFMBA	105%	2%
3:3 FTCA	97%	4%
5:3 FTCA	109%	1%
7:3 FTCA	87%	11%
FHUEA	103%	1%
FOUEA	94%	3%
HQ-115	106%	3%
6:2 diPAP	110%	11%
8:2 diPAP	364%	48%

Phase 2 – Native Spiking: Nonstick Foil

High Range Spike

Analyte	Average Recovery	%RSD
PFTeDA	93%	2%
PFTrDA	101%	4%
PFDoA	116%	2%
PFUnA	100%	2%
PFDA	107%	4%
PFNA	82%	17%
PFOA	93%	4%
PFHpA	99%	1%
PFHxA	97%	1%
PFPeA	98%	2%
PFBA	96%	3%
PFDS	96%	2%
PFNS	95%	3%
PFOS	98%	2%
PFHpS	105%	18%
PFHxS	94%	10%
PFPeS	96%	2%
PFBS	99%	1%
PFOSA	95%	2%
8:2 FTS	104%	7%
6:2 FTS	97%	4%
4:2 FTS	97%	2%
NEtFOSAA	87%	10%

Analyte	Average Recovery	%RSD
NMeFOSAA	108%	15%
PFDoS	100%	0%
NMeFOSA	92%	3%
NEtFOSA	91%	2%
NMeFOSE	91%	3%
NEtFOSE	83%	3%
HFPO-DA	93%	5%
ADONA	95%	2%
9Cl-PF3ONS	90%	3%
11Cl-PF3OUdS	95%	1%
PFPrA	103%	4%
NFDHA	98%	2%
PFEESA	95%	2%
PFMPA	98%	3%
PFMBA	96%	1%
3:3 FTCA	95%	2%
5:3 FTCA	98%	3%
7:3 FTCA	92%	19%
FHUEA	101%	2%
FOUEA	91%	6%
HQ-115	97%	1%
6:2 diPAP	103%	12%
8:2 diPAP	227%	47%

Surrogate Spike n=9

Analyte	Average Recovery	%RSD
13C4-PFBA	105%	8%
13C5-PFPeA	104%	4%
13C5-PFHxA	100%	3%
13C4-PFHpA	98%	6%
13C8-PFOA	97%	5%
13C9-PFNA	91%	13%
13C6-PFDA	101%	6%
13C7-PFUnA	101%	3%
13C2-PFDoA	111%	6%
13C2-PFTeDA	104%	7%
13C8-PFOSA	91%	4%
D3-NMeFOSAA	103%	11%
D5-NEtFOSAA	91%	9%
D3-NMeFOSA	95%	4%
D5-NEtFOSA	90%	4%
D7-NMeFOSE	89%	5%
D9-NEtFOSE	91%	3%
13C3-HFPO-DA	103%	3%
13C2-4:2FTS	97%	9%
13C2-6:2FTS	92%	10%
13C2-8:2FTS	92%	13%
13C8-PFOS	100%	9%
13C3-PFBS	104%	9%
13C3-PFHxS	98%	8%
M4-6:2 diPAP	105%	10%

Phase 2 – Native Spiking: HDPE Coupons

Low Range Spike

Analyte	Average Recovery	%RSD
PFTeDA	135%	10%
PFTrDA	114%	9%
PFDoA	118%	12%
PFUnA	103%	9%
PFDA	133%	17%
PFNA	117%	28%
PFOA	102%	8%
PFHpA	106%	5%
PFHxA	123%	7%
PFPeA	103%	0%
PFBA	94%	2%
PFDS	128%	14%
PFNS	113%	10%
PFOS	99%	3%
PFHpS	115%	27%
PFHxS	96%	14%
PFPeS	120%	7%
PFBS	116%	10%
PFOSA	102%	10%
8:2 FTS	118%	7%
6:2 FTS	95%	9%
4:2 FTS	117%	13%
NEtFOSAA	125%	36%

Analyte	Average Recovery	%RSD
NMeFOSAA	131%	20%
PFDoS	113%	12%
NMeFOSA	105%	10%
NEtFOSA	99%	14%
NMeFOSE	95%	7%
NEtFOSE	109%	8%
HFPO-DA	107%	12%
ADONA	95%	12%
9Cl-PF3ONS	102%	11%
11Cl-PF3OUdS	100%	14%
PFPrA	116%	7%
NFDHA	106%	12%
PFEESA	105%	15%
PFMPA	108%	10%
PFMBA	119%	18%
3:3 FTCA	--	--
5:3 FTCA	95%	10%
7:3 FTCA	81%	26%
FHUEA	101%	17%
FOUEA	109%	23%
HQ-115	121%	15%
6:2 diPAP	140%	6%
8:2 diPAP	593%	48%

Mid Range Spike

Analyte	Average Recovery	%RSD
PFTeDA	121%	3%
PFTrDA	105%	3%
PFDoA	111%	0%
PFUnA	98%	2%
PFDA	105%	5%
PFNA	98%	2%
PFOA	97%	3%
PFHpA	100%	6%
PFHxA	107%	4%
PFPeA	103%	2%
PFBA	101%	1%
PFDS	96%	3%
PFNS	106%	2%
PFOS	99%	3%
PFHpS	96%	12%
PFHxS	95%	7%
PFPeS	103%	4%
PFBS	101%	1%
PFOSA	94%	5%
8:2 FTS	92%	11%
6:2 FTS	80%	2%
4:2 FTS	97%	3%
NEtFOSAA	105%	6%

Analyte	Average Recovery	%RSD
NMeFOSAA	99%	14%
PFDoS	112%	3%
NMeFOSA	100%	1%
NEtFOSA	90%	2%
NMeFOSE	89%	2%
NEtFOSE	95%	3%
HFPO-DA	110%	5%
ADONA	109%	3%
9Cl-PF3ONS	96%	2%
11Cl-PF3OUdS	106%	2%
PFPrA	113%	4%
NFDHA	108%	4%
PFEESA	103%	3%
PFMPA	103%	3%
PFMBA	119%	6%
3:3 FTCA	107%	8%
5:3 FTCA	115%	4%
7:3 FTCA	99%	8%
FHUEA	105%	5%
FOUEA	102%	9%
HQ-115	118%	5%
6:2 diPAP	122%	3%
8:2 diPAP	796%	24%

Phase 2 – Native Spiking: HDPE Coupons

High Range Spike

Analyte	Average Recovery	%RSD
PFTeDA	104%	1%
PFTrDA	102%	0%
PFDoA	117%	1%
PFUnA	94%	1%
PFDA	93%	16%
PFNA	91%	15%
PFOA	96%	3%
PFHpA	95%	5%
PFHxA	98%	1%
PFPeA	101%	1%
PFBA	98%	0%
PFDS	95%	2%
PFNS	94%	1%
PFOS	95%	6%
PFHpS	99%	5%
PFHxS	95%	7%
PFPeS	98%	2%
PFBS	96%	2%
PFOSA	92%	1%
8:2 FTS	104%	7%
6:2 FTS	83%	1%
4:2 FTS	97%	4%
NEtFOSAA	100%	4%

Analyte	Average Recovery	%RSD
NMeFOSAA	102%	7%
PFDoS	104%	1%
NMeFOSA	93%	2%
NEtFOSA	89%	1%
NMeFOSE	89%	1%
NEtFOSE	86%	1%
HFPO-DA	100%	2%
ADONA	95%	2%
9Cl-PF3ONS	92%	1%
11Cl-PF3OUdS	96%	3%
PFPrA	100%	1%
NFDHA	102%	2%
PFEESA	96%	3%
PFMPA	99%	2%
PFMBA	103%	1%
3:3 FTCA	95%	1%
5:3 FTCA	103%	6%
7:3 FTCA	104%	8%
FHUEA	102%	1%
FOUEA	94%	3%
HQ-115	104%	1%
6:2 diPAP	103%	2%
8:2 diPAP	551%	11%

Surrogate Spike n=9

Analyte	Average Recovery	%RSD
13C4-PFBA	102%	3%
13C5-PFPeA	103%	4%
13C5-PFHxA	97%	4%
13C4-PFHpA	93%	9%
13C8-PFOA	90%	9%
13C9-PFNA	93%	9%
13C6-PFDA	102%	6%
13C7-PFUnA	94%	6%
13C2-PFDoA	108%	11%
13C2-PFTeDA	116%	4%
13C8-PFOSA	89%	5%
D3-NMeFOSAA	96%	11%
D5-NEtFOSAA	97%	6%
D3-NMeFOSA	98%	4%
D5-NEtFOSA	90%	8%
D7-NMeFOSE	92%	6%
D9-NEtFOSE	94%	6%
13C3-HFPO-DA	104%	3%
13C2-4:2FTS	100%	8%
13C2-6:2FTS	81%	16%
13C2-8:2FTS	90%	15%
13C8-PFOS	99%	5%
13C3-PFBS	104%	5%
13C3-PFHxS	94%	8%
M4-6:2 diPAP	119%	7%

Phase 2 – Native Spiking: Non-Spiked

Analyte	Cosmetic	Nonstick Foil	Pizza Box	HDPE Coupons	Athletic Tape	Dress Shirt
PFTeDA					7	
PFTrDA					13	
PFDoA					42	6
PFUnA					60	
PFDA			5		113	8
PFNA					179	
PFOA	6	6			201	13
PFHpA					26	112
PFHxA			15		135	13677
PFPeA					16	442
PFBA						507
PFDS						
PFNS						
PFOS			12			
PFHpS						
PFHxS			7			
PFPeS						
PFBS						
PFOSA						
8:2 FTS						
6:2 FTS			6			15
4:2 FTS						
NEtFOSAA						

Analyte	Cosmetic	Nonstick Foil	Pizza Box	HDPE Coupons	Athletic Tape	Dress Shirt
NMeFOSAA						
PFDoS						
NMeFOSA						
NEtFOSA						
NMeFOSE						
NEtFOSE			6			
HFPO-DA						
ADONA						
9Cl-PF3ONS						
11Cl-PF3OUdS						
PFPrA	153				60	146
NFDHA						
PFEESA						
PFMPA						
PFMBA						
3:3 FTCA						
5:3 FTCA						
7:3 FTCA						
FHUEA			10			121
FOUEA			2		13	
HQ-115			15			
6:2 diPAP		54	657	34	276	530
8:2 diPAP			243		31	

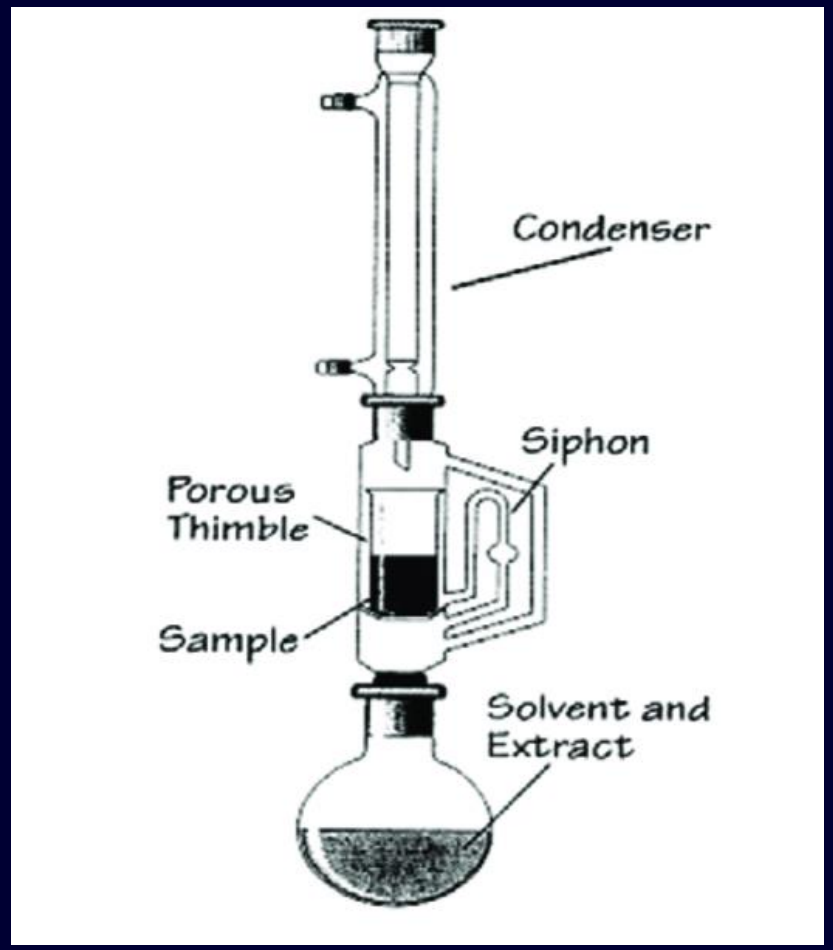
Note: All values are in vial and in units of ng/L

Phase 2 – Surrogate Summary

	Cosmetic		Nonstick Foil		Pizza Box		HDPE Coupons		Athletic Tape		Dress Shirt	
Analytes	**Average**	**RSD**	**Average**	**RSD**	**Average**	**RSD**	**Average**	**RSD**	**Average**	**RSD**	**Average**	**RSD**
13C4-PFBA	102%	9%	105%	8%	121%	5%	102%	3%	114%	4%	128%	4%
13C5-PFPeA	138%	7%	104%	4%	110%	2%	103%	4%	107%	1%	118%	4%
13C5-PFHxA	99%	5%	100%	3%	104%	3%	97%	4%	104%	2%	94%	4%
13C4-PFHpA	101%	3%	98%	6%	106%	4%	93%	9%	98%	6%	111%	8%
13C8-PFOA	100%	6%	97%	5%	94%	7%	90%	9%	94%	8%	101%	9%
13C9-PFNA	170%	9%	91%	13%	87%	8%	93%	9%	101%	8%	115%	11%
13C6-PFDA	117%	7%	101%	6%	115%	11%	102%	6%	110%	3%	117%	7%
13C7-PFUnA	112%	6%	101%	3%	123%	7%	94%	6%	103%	9%	113%	13%
13C2-PFDoA	108%	7%	111%	6%	111%	6%	108%	11%	131%	13%	143%	15%
13C2-PFTreA	150%	4%	104%	7%	114%	13%	116%	4%	66%	12%	144%	8%
13C8-PFOSA	112%	5%	91%	4%	97%	7%	89%	5%	90%	9%	65%	8%
D3-NMeFOSAA	117%	10%	103%	11%	138%	9%	96%	11%	117%	7%	122%	9%
D5-NEtFOSAA	119%	7%	91%	9%	165%	8%	97%	6%	147%	9%	128%	5%
D3-NMeFOSA	93%	4%	95%	4%	57%	9%	98%	4%	75%	8%	45%	6%
D5-NEtFOSA	84%	7%	90%	4%	75%	7%	90%	8%	68%	6%	47%	5%
D7-NMeFOSE	43%	10%	89%	5%	49%	10%	92%	6%	90%	6%	33%	15%
D9-NEtFOSE	42%	7%	91%	3%	51%	3%	94%	6%	81%	4%	22%	13%
13C3-HFPO-DA	101%	7%	103%	3%	82%	6%	104%	3%	107%	3%	120%	8%
13C2-4:2FTS	100%	8%	97%	9%	391%	24%	100%	8%	99%	8%	145%	14%
13C2-6:2FTS	91%	11%	92%	10%	289%	31%	81%	16%	91%	15%	128%	19%
13C2-8:2FTS	157%	25%	92%	13%	345%	37%	90%	15%	90%	19%	117%	17%
13C8-PFOS	99%	6%	100%	9%	91%	8%	99%	5%	92%	9%	89%	8%
13C3-PFBS	102%	10%	104%	9%	88%	7%	104%	5%	108%	3%	85%	8%
13C3-PFHxS	99%	10%	98%	8%	77%	14%	94%	8%	99%	12%	97%	8%
M4-6:2 diPAP	148%	9%	105%	10%	636%	11%	119%	7%	327%	9%	659%	8%

n=9 for all samples

Comparison Tests



Soxhlet

An expensive, timely, and aggressive extraction

- A Soxhlet extraction was used for several articles to compare the proposed extraction process to a more laborious extraction method.
- Results are analyte dependent, but in general:
  - Slightly more native PFAS are found using the Soxhlet extraction than the proposed extraction
  - No **Different** PFAS are detected in the Soxhlet extraction

Comparison: Soxhlet Extraction

Dress Shirt

Analyte	Soxhlet	Solvent
PFTeDA		
PFTrDA		
PFDoA	732	113
PFUnA		
PFDA	451	153
PFNA		
PFOA	476	162
PFHpA	1,428	2,242
PFHxA	167,970	273,531
PFPeA	10,026	8,835
PFBA	12,695	10,327
PFDS		
PFNS		
PFOS		
PFHpS		
PFHxS		
PFPeS		
PFBS		
PFOSA		
8:2 FTS		
6:2 FTS	475	300
4:2 FTS		
NEtFOSAA		

Analyte	Soxhlet	Solvent
NMeFOSAA		
PFDoS		
NMeFOSA		
NEtFOSA		
NMeFOSE		
NEtFOSE		
HFPO-DA		
ADONA		
9Cl-PF3ONS		
11Cl-PF3OUdS		
PFPrA		2,881
NFDHA		
PFEESA		
PFMPA		126
PFMBA		
3:3 FTCA		
5:3 FTCA		
7:3 FTCA		
FHUEA	6,202	2,428
FOUEA		
HQ-115		
6:2 diPAP	9,517	10,597
8:2 diPAP	1,632	299

Note: All values account for dilutions and in units of ng/L

Carpet Remnant

Analyte	Soxhlet	Solvent
PFTeDA	430	125
PFTrDA		
PFDoA	925	160
PFUnA		
PFDA	777	259
PFNA		189
PFOA	387	470
PFHpA	241	229
PFHxA	1,090	465
PFPeA	243	114
PFBA		
PFDS		
PFNS		
PFOS	495	331
PFHpS		
PFHxS		
PFPeS		
PFBS		
PFOSA		
8:2 FTS	101	949
6:2 FTS	348	1,779
4:2 FTS		
NEtFOSAA		

Analyte	Soxhlet	Solvent
NMeFOSAA		
PFDoS		
NMeFOSA		
NEtFOSA		
NMeFOSE		
NEtFOSE		
HFPO-DA		
ADONA		
9Cl-PF3ONS		
11Cl-PF3OUdS		
PFPrA		
NFDHA		
PFEESA		
PFMPA		
PFMBA		
3:3 FTCA		
5:3 FTCA		
7:3 FTCA		
FHUEA		
FOUEA		
HQ-115		
6:2 diPAP	78,119	NA
8:2 diPAP	22,412	NA

Note: NA indicates the solvent extraction was performed before target analyte was added to method.

Comparison: Soxhlet Extraction

Pipette Tip

Analyte	Soxhlet	Solvent
PFTeDA	319	
PFTrDA	339	
PFDoA	372	122
PFUnA	722	508
PFDA	4,921	3,419
PFNA	10,953	9,860
PFOA	49,680	46,807
PFHpA	44,241	30,371
PFHxA	68,043	32,546
PFPeA	122,054	57,182
PFBA	148,498	66,145
PFDS		
PFNS		
PFOS		
PFHpS		
PFHxS		
PFPeS		
PFBS		
PFOSA		
8:2 FTS		
6:2 FTS		
4:2 FTS		
NEtFOSAA		

Analyte	Soxhlet	Solvent
NMeFOSAA		
PFDoS		
NMeFOSA		
NEtFOSA		
NMeFOSE		
NEtFOSE		
HFPO-DA		
ADONA		
9Cl-PF3ONS		
11Cl-PF3OUdS		
PFPrA	141,472	43,388
NFDHA		
PFEESA		
PFMPA		115
PFMBA		
3:3 FTCA		
5:3 FTCA	247	288
7:3 FTCA	662	703
FHUEA	257	217
FOUEA	13,175	10,374
HQ-115		
6:2 diPAP	800	NA
8:2 diPAP	1,495	NA

Note: All values are in vial and in units of ng/L

Pop Corn Bag

Analyte	Soxhlet	Solvent
PFTeDA		
PFTrDA		
PFDoA		
PFUnA		
PFDA		
PFNA		
PFOA		
PFHpA		
PFHxA	188	180
PFPeA		
PFBA		
PFDS		
PFNS		
PFOS		
PFHpS		
PFHxS		
PFPeS		
PFBS		
PFOSA		
8:2 FTS		
6:2 FTS		213
4:2 FTS		
NEtFOSAA		

Analyte	Soxhlet	Solvent
NMeFOSAA		
PFDoS		
NMeFOSA		
NEtFOSA		
NMeFOSE		
NEtFOSE		
HFPO-DA		
ADONA		
9Cl-PF3ONS		
11Cl-PF3OUdS		
PFPrA	2,249	
NFDHA		
PFEESA		
PFMPA	711	
PFMBA		
3:3 FTCA		1,600
5:3 FTCA		
7:3 FTCA		124
FHUEA	282	
FOUEA		
HQ-115		
6:2 diPAP	9,085	NA
8:2 diPAP	1,393	NA

Note: NA indicates the solvent extraction was performed before target analyte was added to method.

Comparison Tests

Cryomilling

Better than coupons?

- One sample (Dress Shirt) was cryomilled to compare proposed method of cutting coupons to a more aggressive preparation technique.
  - Slightly more native PFAS is found using the cryomilling preparation than the proposed preparation.
  - No **Different** PFAS are detected in the Soxhlet extraction

Comparison: CryoMilling

Dress Shirt

Analyte	Cryomill	Coupon
PFTeDA		
PFTrDA		
PFDoA	11	6
PFUnA		
PFDA	16	8
PFNA	5	
PFOA	14	8
PFHpA	308	112
PFHxA	15250	13677
PFPeA	734	442
PFBA	671	516
PFDS		
PFNS		
PFOS		
PFHpS		
PFHxS		
PFPeS		
PFBS		
PFOSA		
8:2 FTS		
6:2 FTS	51	15
4:2 FTS		
NEtFOSAA		

Analyte	Cryomill	Coupon
NMeFOSAA		
PFDoS		
NMeFOSA		
NEtFOSA		
NMeFOSE		
NEtFOSE		
HFPO-DA		
ADONA		
9Cl-PF3ONS		
11Cl-PF3OUdS		
PFPrA	216	144
NFDHA		
PFEESA		
PFMPA		
PFMBA		
3:3 FTCA		
5:3 FTCA		
7:3 FTCA		
FHUEA	2239	121
FOUEA		
HQ-115		
6:2 diPAP	815	530
8:2 diPAP	63	15

Note: All values are in vial and in units of ng/L

Comparison Tests

Total Fluoride and Extracted Fluoride

- Several articles were tested for fluoride content via bomb combustion and Ion Chromatography analysis.
- Extracts and As-received samples were combusted.
  - Detection limits of approximately 20 mg/kg.
- As-received data can be seen in the table.
- All extractions analyzed were non-detects.

Sample	F^- (mg/kg)
Dress shirt	3060
Pop Corn Bag	554
Cosmetic – pressed powder	596
Athletic tape	104
Dental Floss	533000
Carpet remnant	51.4
Pipette Tip	1930

Final Thoughts

Imagine This

You do not include PFAS in your product. You have it tested at a lab and find little to no PFAS.

Another party tests you product and claims it has PFAS in it.

What do you do? How do you compare these tests? What if they used different methods?

Summary

- One standardized and validated method will harmonize product testing across laboratories.
- Extraction methodology is simple, robust, and effective.
- Target analytes are quantitated via external calibration with detection limits ranging from 100 ng/kg to 4,000 ng/kg.
- Single lab validation on a wide range of matrices will make the final method applicable to many industries.
- Anticipated first ballot by end of year.

Thank you!

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