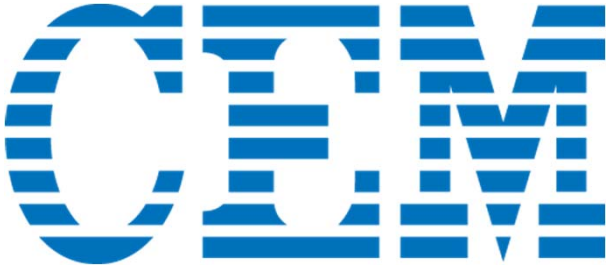

A New Rapid, Simple, and Efficient Extraction Method of PFAS from Soil

Alicia D. Stell, Ph.D.

CEM Corporation

PFAS: Per- and Polyfluoroalkyl Substances










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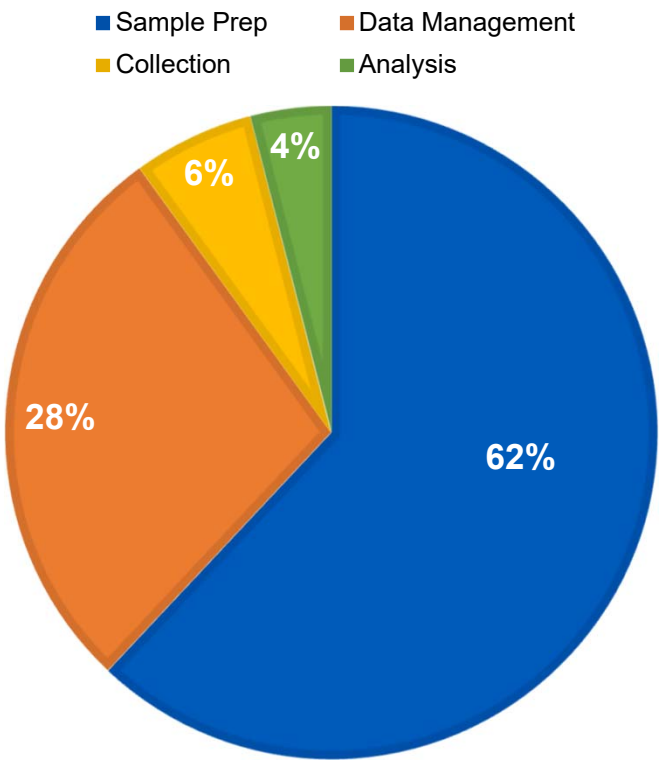
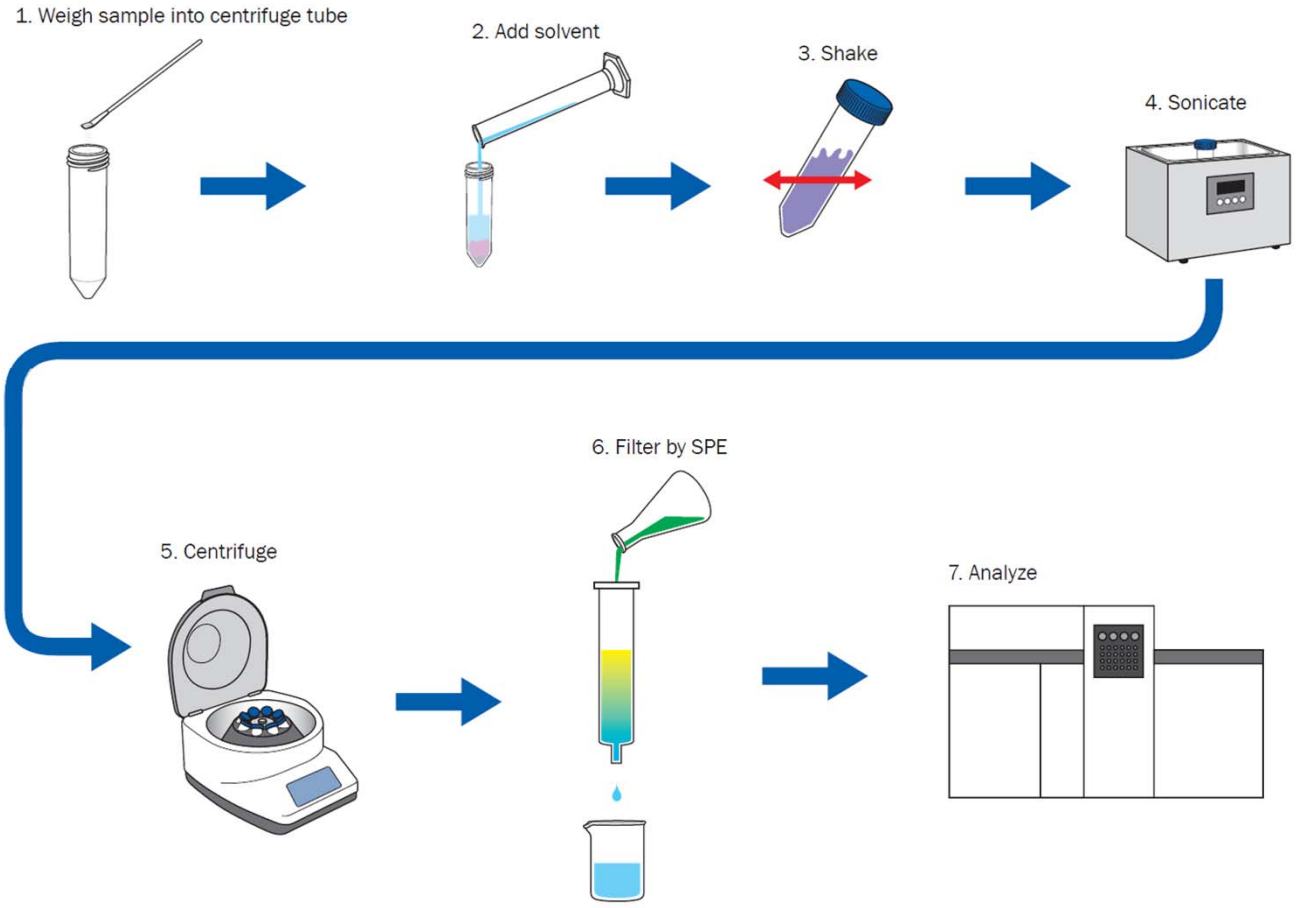
Sample Preparation is the Bottleneck

Time Spent on Typical Analysis

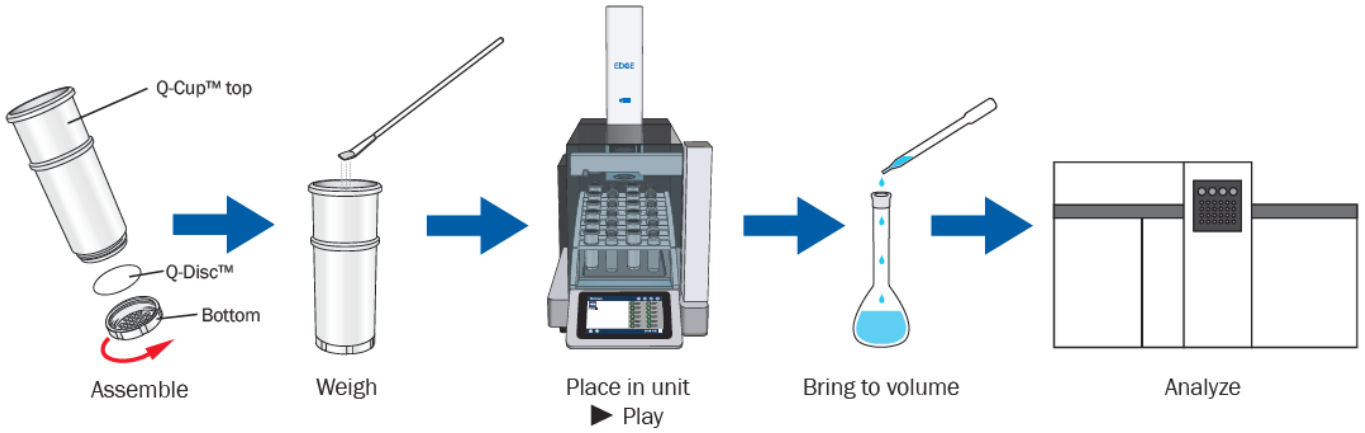


R.E. Majors, LC/GC Magazine

Typical PFAS Extraction from Soil

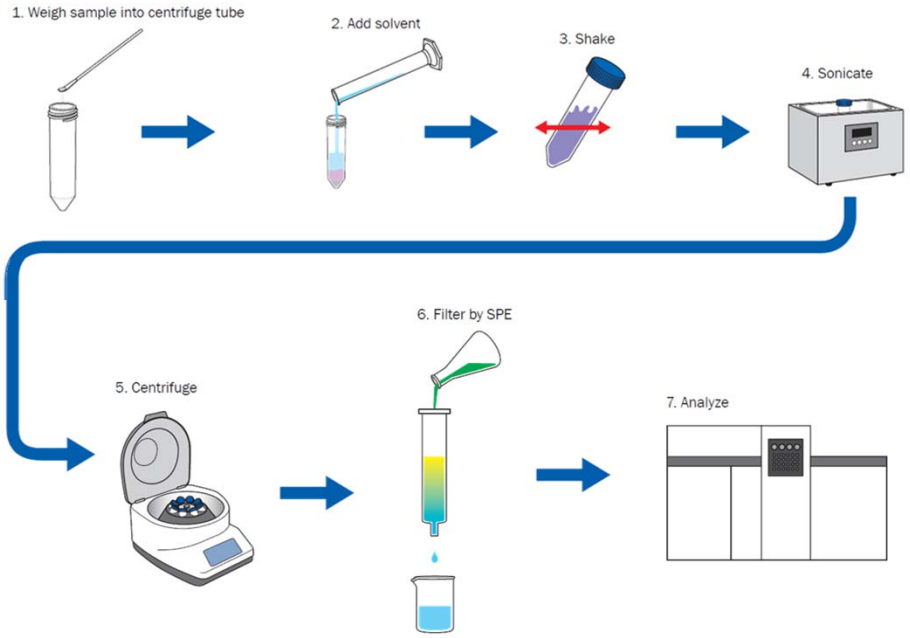


EDGE Extraction

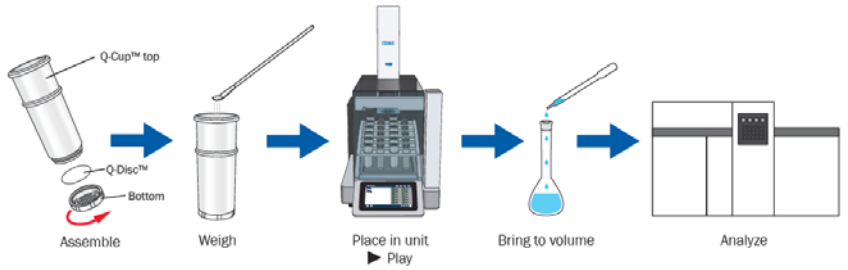


Standard versus EDGE Extraction

Standard



EDGE



EDGE Advantages

- Automation
- In-cell Cleanup
- Simple
- Multi-matrix Multi-residue Method
- PFAS free

PFAS Free Instrumentation

- PEEK and Polypropylene Tubing
- Side Enclosure
- Centrifuge Tube Collection
- Nitrogen Option
- PFAS free consumables

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EDGE Sample Prep

Layered in Q-Cup (sample holder)

EDGE Sample Prep

Layered in Q-Cup (sample holder)

Q-Disc	S1 (C9+G1+C9)

EDGE Sample Prep

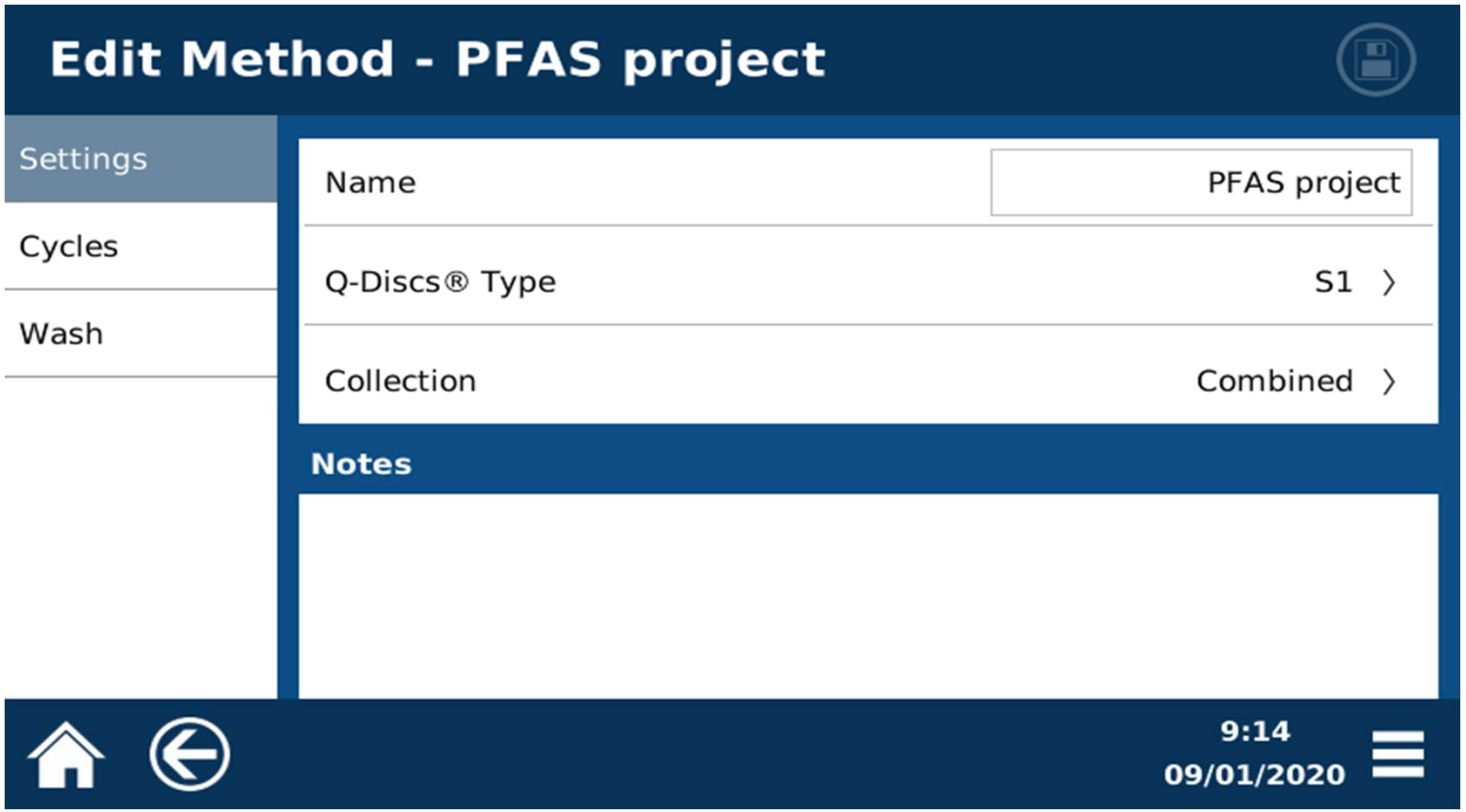
Layered in Q-Cup (sample holder)

Sample	5 g: Soil
Q-Disc	S1 (C9+G1+C9)

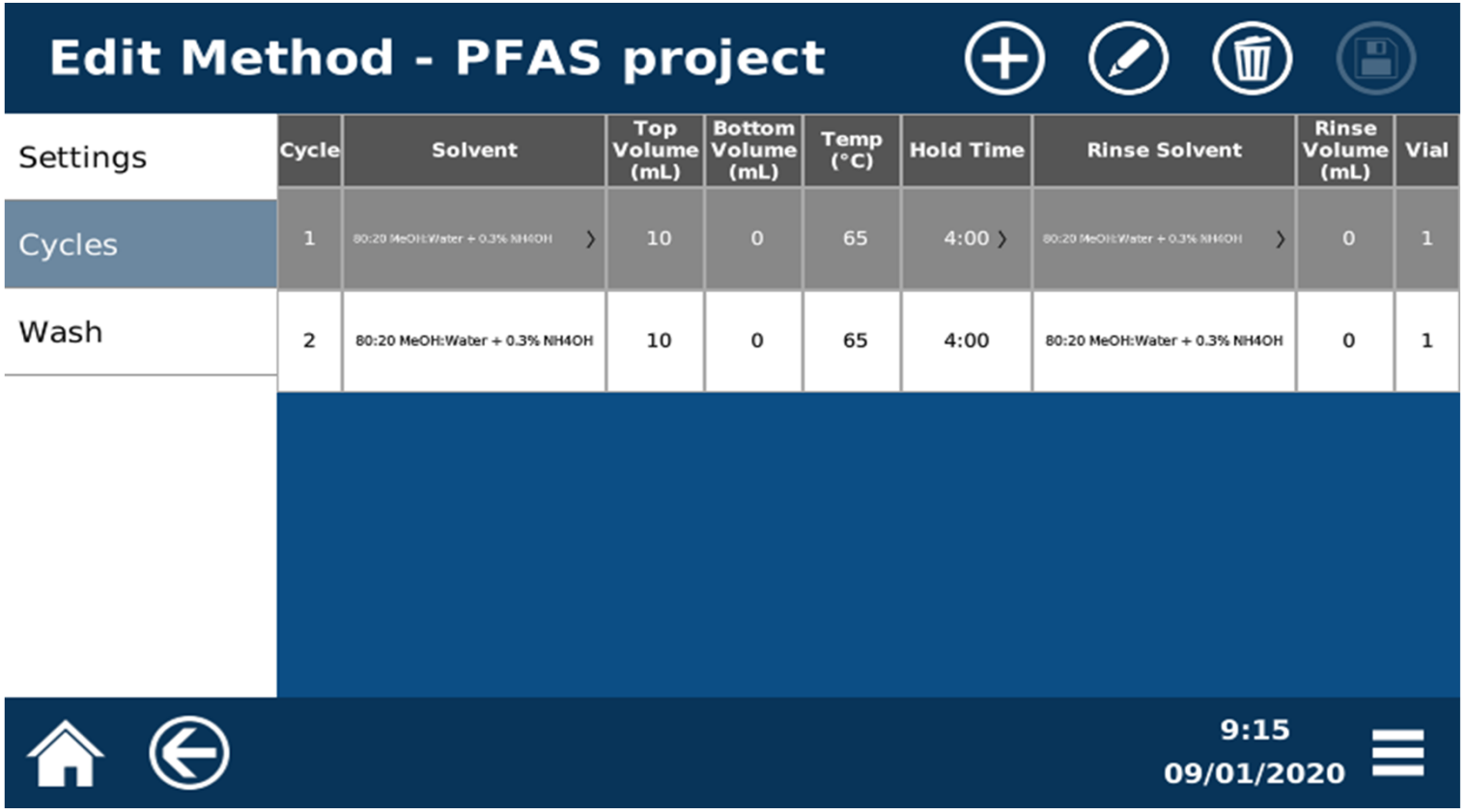
EDGE Sample Prep

Layered in Q-Cup (sample holder)	
Spike	Wellington Laboratories: PFAC30PAR and MPFAC-24ES
Sample	5 g: Soil
Q-Disc	S1 (C9+G1+C9)

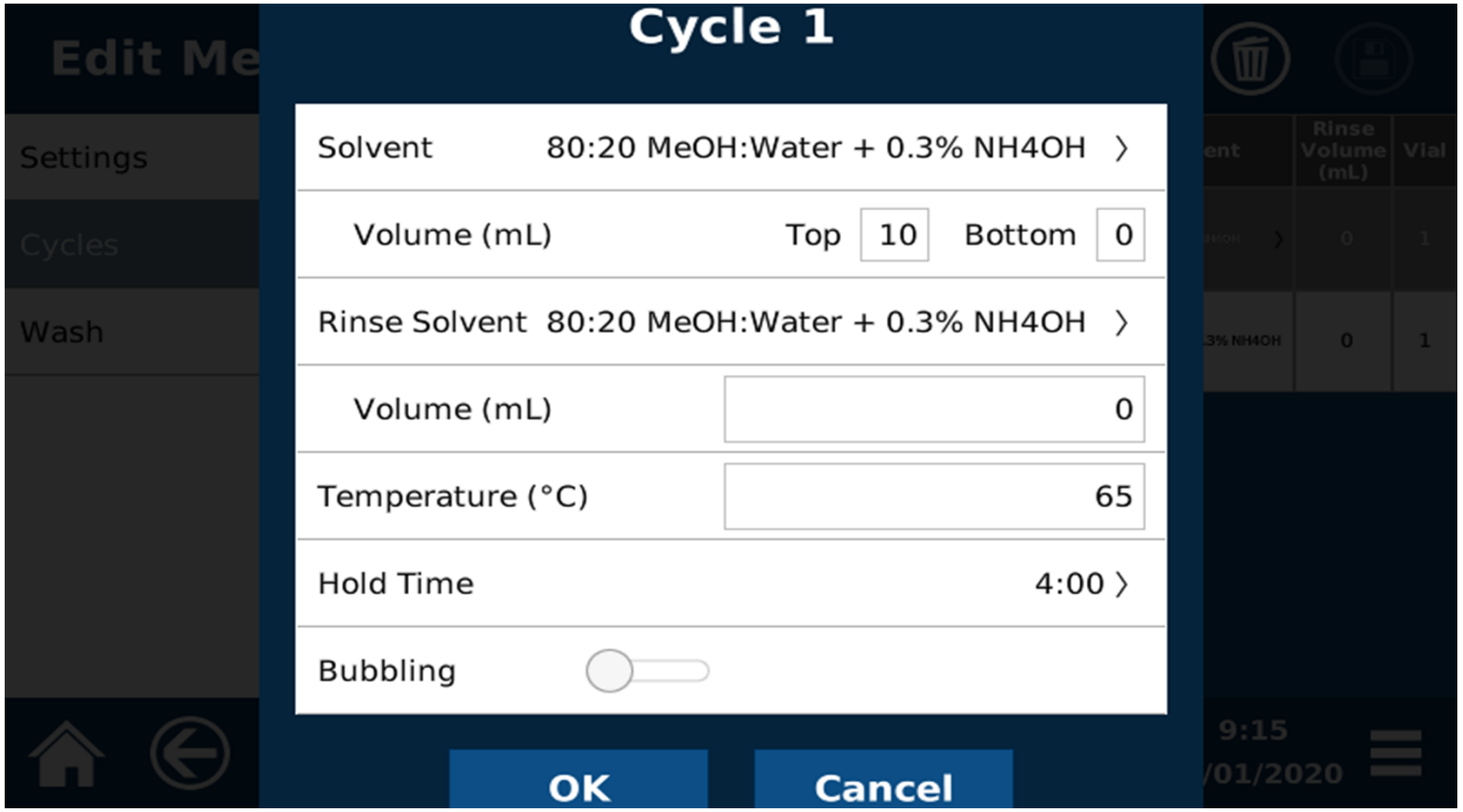
EDGE Method


Edit Method - PFAS project
Settings
Cycles
Wash
Name
PFAS project
Q-Discs® Type
S1
Collection
Combined
Notes
9:14
09/01/2020

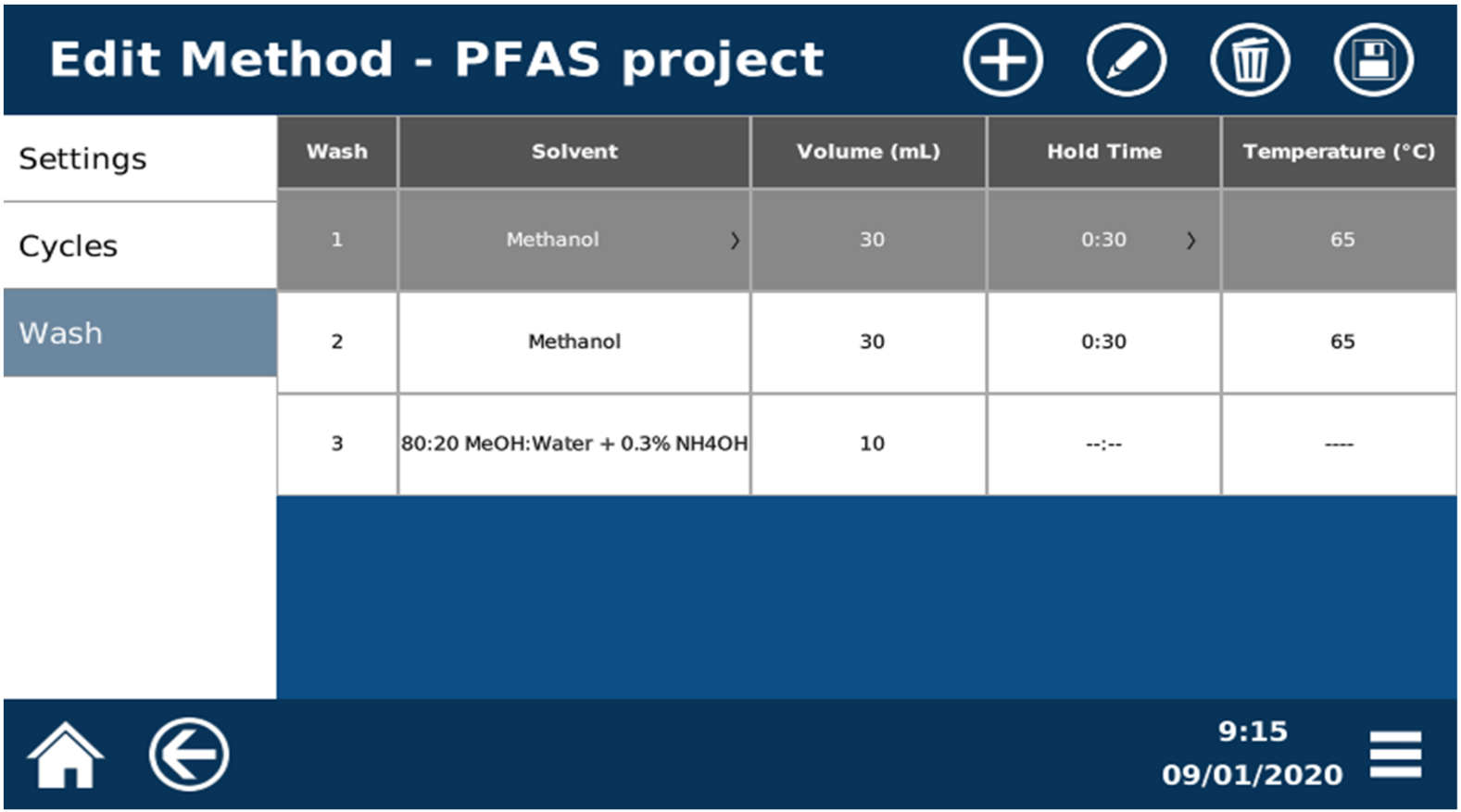
EDGE Method


Edit Method - PFAS project
Settings
Cycles
Wash
Cycle
Solvent
Top Volume (mL)
Bottom Volume (mL)
Temp (°C)
Hold Time
Rinse Solvent
Rinse Volume (mL)
Vial
1
80:20 MeOH:Water + 0.3% NH4OH
10
0
65
4:00
80:20 MeOH:Water + 0.3% NH4OH
0
1
2
80:20 MeOH:Water + 0.3% NH4OH
10
0
65
4:00
80:20 MeOH:Water + 0.3% NH4OH
0
1
9:15
09/01/2020

EDGE Method


Edit Me
Settings
Cycles
Wash
Cycle 1
Solvent 80:20 MeOH:Water + 0.3% NH4OH
Volume (mL) Top 10 Bottom 0
Rinse Solvent 80:20 MeOH:Water + 0.3% NH4OH
Volume (mL) 0
Temperature (°C) 65
Hold Time 4:00
Bubbling
OK
Cancel
Rinse Volume (mL)
Vial
9:15
/01/2020

EDGE Method


Edit Method - PFAS project
Settings
Cycles
Wash
Wash
Solvent
Volume (mL)
Hold Time
Temperature (°C)
1
Methanol
30
0:30
65
2
Methanol
30
0:30
65
3
80:20 MeOH:Water + 0.3% NH4OH
10
--:--

9:15
09/01/2020

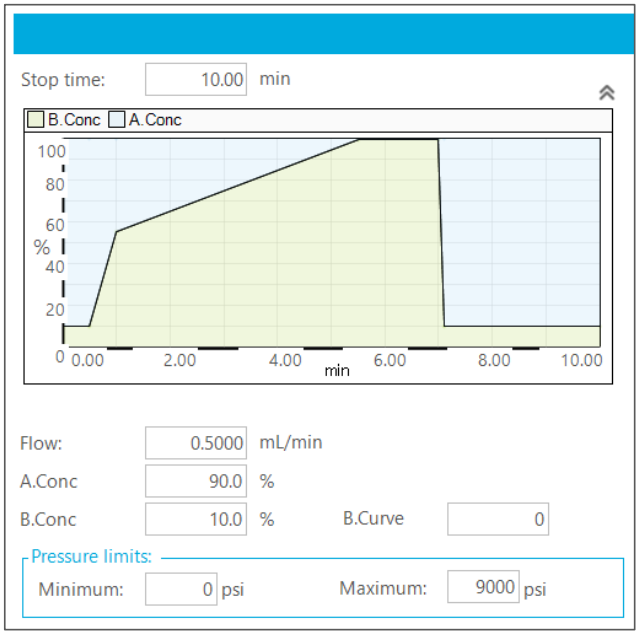
Minimal Post work

Dilute to known volume

Neutralize

Instrument Conditions

Acquisition Parameters	
HPLC System	ExionLC™ HPLC System
MS/MS System	5500+ QTRAP
Ion Source	Turbo V™
Injection Volume	1 µL
Analytical Column	Phenomenex. Gemini C18 3 µm, 3 x 100 mm
Delay Column	Phenomenex. Gemini C18 5 µm, 3 x 50 mm
LC Flow Rate	500 µL/min
Mobile Phases	Water & MeOH (both with 10 mM ammonium acetate)
Source & MS Parameters	TEM = 600 C GS1= 60, GS2 = 60 ISV = -4500 CUR = 35, CAD = 8

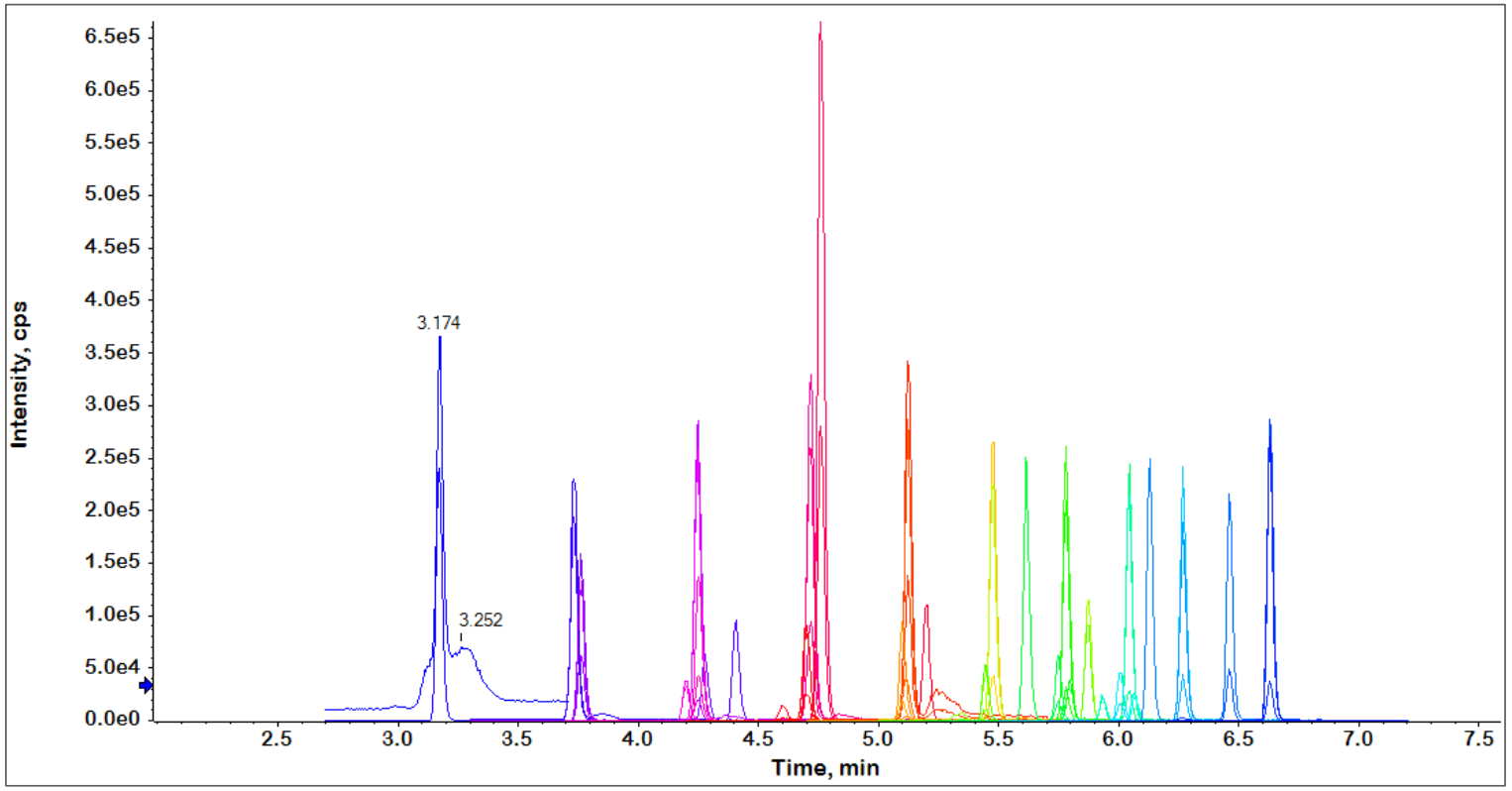


Flow program

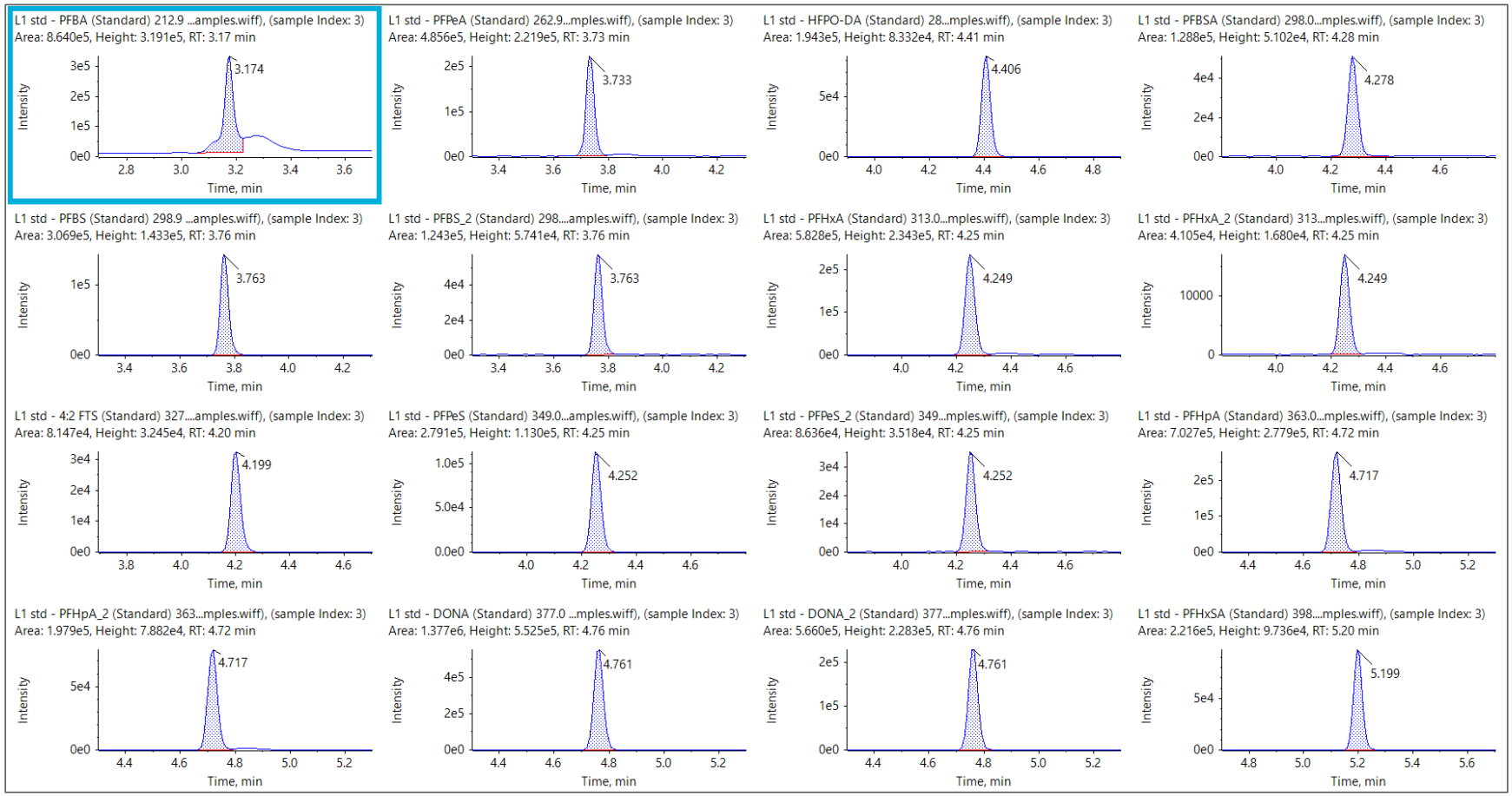
Flow program ○ Simple

	Time	Flow	A.Con	B.Con	B.Curve
1		0.5000	90.0	10.0	0
2	0.50	0.5000	90.0	10.0	0
3	1.00	0.5000	45.0	55.0	0
4	5.50	0.5000	1.0	99.0	0
5	7.00	0.5000	1.0	99.0	0
6	7.10	0.5000	90.0	10.0	0
7	10.00	0.5000	90.0	10.0	0

Example Chromatogram – 2 ng/mL standard



Example Chromatogram – 2 ng/mL standard



Soil Data: High Spike 20 ppb

PFAS	% Recovery	% RSD (n=3)
1H, 1H, 2H, 2H-perfluorodecane sulfonic acid (8:2 FTS)	91	9.7
1H, 1H, 2H, 2H-perfluorooctane sulfonic acid (6:2 FTS)	90	11
1H,1H,2H,2H-perfluorohexane sulfonic acid (4:2 FTS)	101	5.8
N-ethylperfluoro-1-octanesulfonamidoacetic acid (EtFOSAA)	95	12
N-methylperfluoro-1-octanesulfonamidoacetic acid (MeFOSAA)	87	12
perfluoro-1-butanesulfonic acid (PFBS)	97	13
perfluoro-1-decanesulfonic acid (PFDS)	91	14
perfluoro-1-heptanesulfonic acid (PFHpS)	96	12
perfluoro-1-nonanesulfonic acid (PFNS)	91	16
perfluoro-1-octanesulfonamide (PFOSA)	85	20
perfluoro-1-pentanesulfonic acid (PFPeS)	88	11
perfluorohexanesulfonic acid (PFHxS)	97	11

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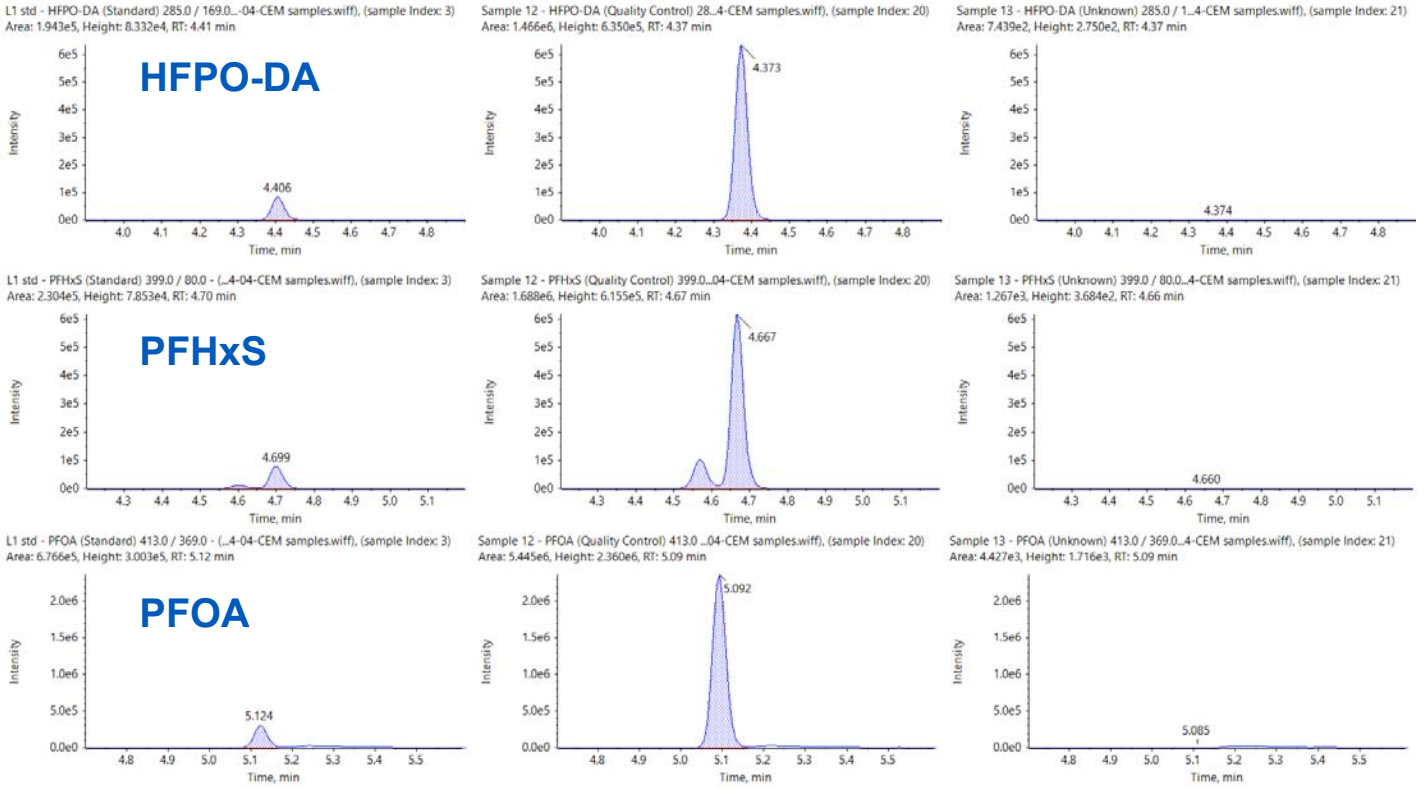
Soil Data: High Spike 20 ppb

PFAS	% Recovery	RSD (n=3)
perfluoro-n-butanoic acid (PFBA)	87	12
perfluoro-n-decanoic acid (PFDA)	89	15
perfluoro-n-dodecanoic acid (PFDoA)	87	10
perfluoro-n-heptanoic acid (PFHpA)	86	10
perfluoro-n-hexanoic acid (PFHxA)	88	11
perfluoro-n-nonanoic acid (PFNA)	88	10
perfluoro-n-octanoic acid (PFOA)	91	10
perfluoro-n-pentanoic acid (PFPeA)	86	9.5
perfluoro-n-tetradecanoic acid (PFTeDA)	91	11
perfluoro-n-tridecanoic acid (PFTrDA)	87	11
perfluoro-n-uoecanoic acid (PFUdA)	87	12
perfluorooctanesulfonic acid (PFOS)	98	8.9

No Carryover – Sample Immediately After High Spike

2 ng/mL std	High Spike	Blank



ERA Soil CRM Data

PFAS	% Recovery	RSD (n=2)
PFBA	88	13
DONA	96	18
4:2 FTS	106	15
HFPO-DA	86	13
PFDoA	92	7.5
PFHpS	99	19
PFHxS	108	20
PFOSA	95	24
PFOS	105	15
PFUnA	98	16

Conclusions

- EDGE instrumentation is PFAS free
- No contamination from sample preparation or consumables
- Good recoveries and RSD values for high spiked and CRM soil samples
- No carryover from high spike sample
- EDGE is a rapid, simple, and efficient technology for the extraction of PFAS from soil

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We Are Where You Are

- 60 associates in the United States and Canada
- 24-hour telephone support

Extraction Team

Alicia Stell, PhD
Lead R&D Scientist

Candice Cashman, PhD
Senior Scientist

Brittany Fessler
Product Specialist

Benedict Liu
Applications Scientist

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Questions

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