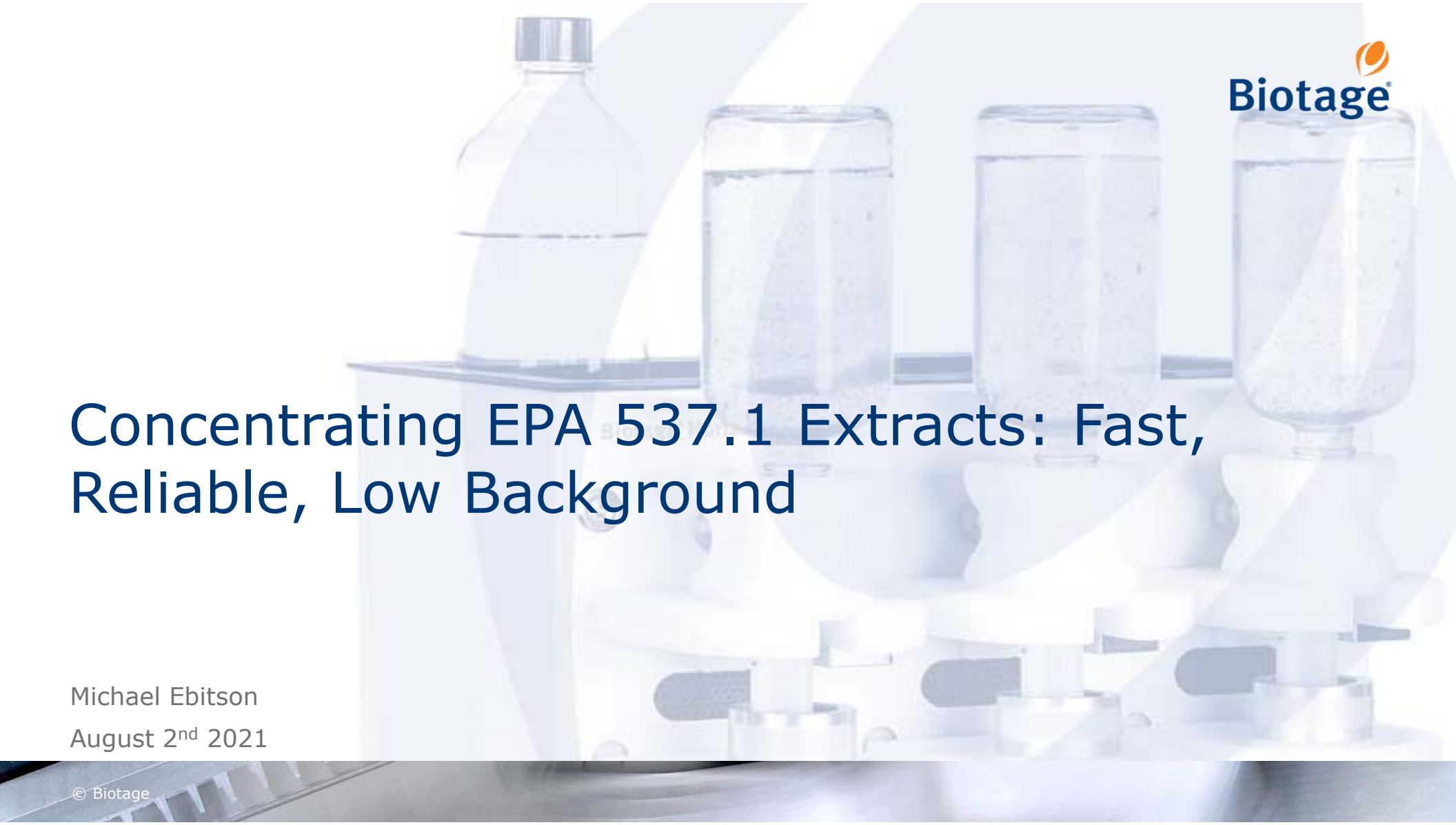

Concentrating EPA 537.1 Extracts: Fast, Reliable, Low Background

Michael Ebitson

August 2nd 2021

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PFAS Methods

- EPA Method 537
  - Drinking Water
  - 14 Compounds
  - 6-mL Format, SDVB Cartridge
- EPA Method 537.1
  - Drinking Water
  - 18 Compounds
  - 6-mL Format, SDVB Cartridge
- EPA Method 533
  - Drinking Water
  - 25 Compounds
  - 6-mL Format, WAX Cartridge (30 um)
- ISO 21675
  - Drinking Water & Non-Potable Water
  - 30 Compounds
  - 6-mL Format, WAX Cartridge
- DoD QSM 5.3
  - Non-Potable Water & Groundwater
  - 25 Compounds
  - 6-mL Format OR Disk Format WAX Cartridge w/GCB Cleanup

Program	Matrix	Format	Sorbent	NEtFOSAA	NMeFOSAA	PFBS	PFDA	PFDoA	PFHpA	PFHxS	PFHxA	PFNA	PFOS	PFOA	PFTA	PFTrDA	PFUnA	11Cl-PF3OUdS	9Cl-PF3ONS	ADONA	HFPO-DA	PFBA	PFPeA	6:2 FTSA	8:2 FTSA	PFDS	Et-FOSA	FOSA	Me-FOSA	PFOcDA	PFHxDA	PFPeS	PFHpS	FHUEA	FOUEA	8:2 diPAP	4:2 FTS	6:2 FTA (FHEA)	8:2 FTA (FOEA)	7:3 FTCA	PFNS	10:2 FTA (FDEA)	PFecHS	NFDHA	PFEESA	PFMPA	PFMBA
UCMR 3	DW	Cart	SDVB			X			X	X		X	X	X																																		
EPA 537	DW	Cart	SDVB	X	X	X	X	X	X	X	X	X	X	X	X	X	X																															
EPA 537.1	DW	Cart	SDVB	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X																												
EPA 533	DW	Cart	WAX			X	X	X	X	X	X	X	X	X			X	X	X	X	X	X	X	X	X							X	X				X						X	X	X	X
ISO 21675	DW/NPW	Cart/Disk	WAX	X	X	X	X	X	X	X	X	X	X	X	X	X	X		X	X	X	X	X	X	X	X	X	X	X	X	X		X		X	X											
DoD QSM 5.3	NPW/GW	Cart/Disk	WAX/Carb	X	X	X	X	X	X	X	X	X	X	X	X	X	X					X	X	X	X	X		X	X			X	X				X				X						

PFAS Methods - Timeline

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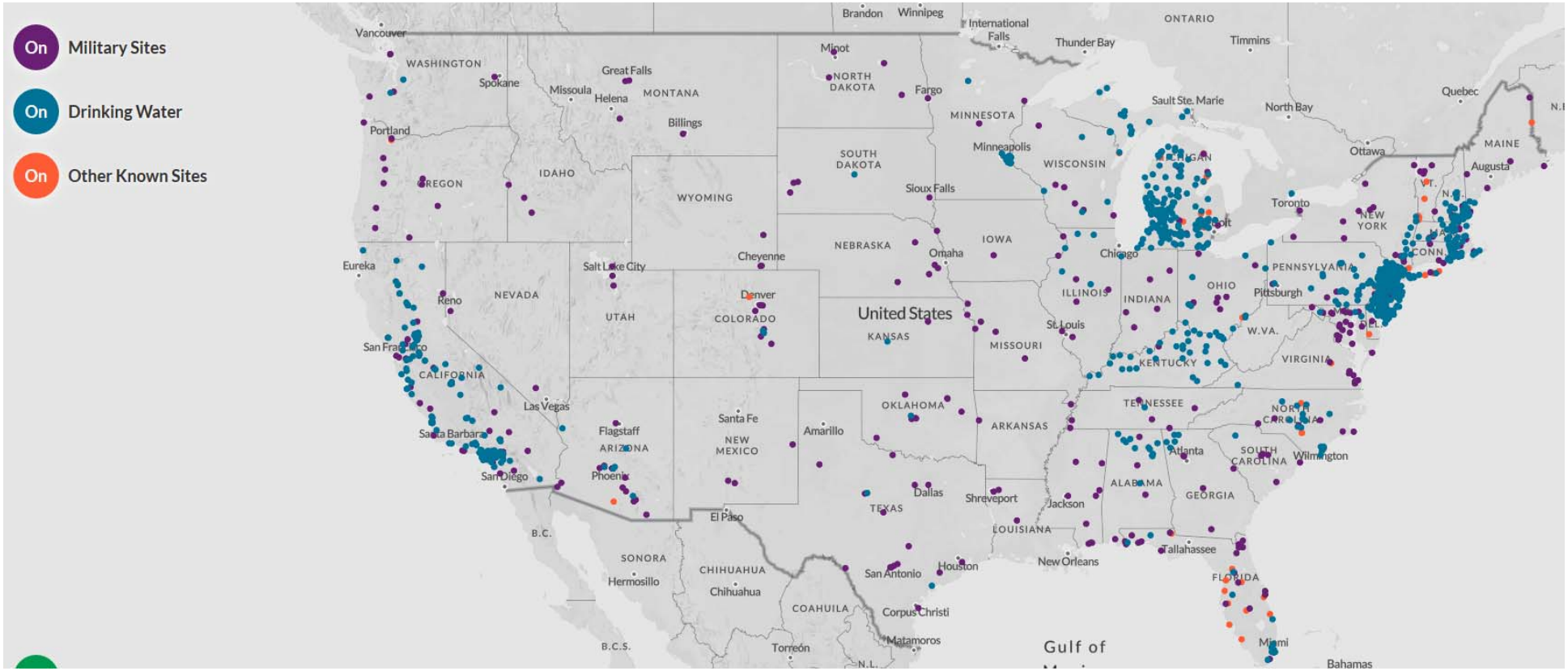
PFAS Contamination in the U.S. (2021)



https://www.ewg.org/interactive-maps/pfas_contamination/map/

Laboratory Goals- What Needs to Be Achieved?

	Solvents	SPE Media	Extraction	Concentration	Sample Prep
Background	✓	✓	✓	✓	✓
Carryover			✓	✓	✓
Lab Environment	✓		✓	✓	✓

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Why Focus On Concentration/Evaporation?

- Last step after timely extraction process
- Can be a source of background
- Can take a long time to evaporate methanol
- Ensure all components are PTFE free
- This process can take a very long time if not properly equipped

Method 537.1 Workflow

Sample → Extraction → Concentration → Analysis

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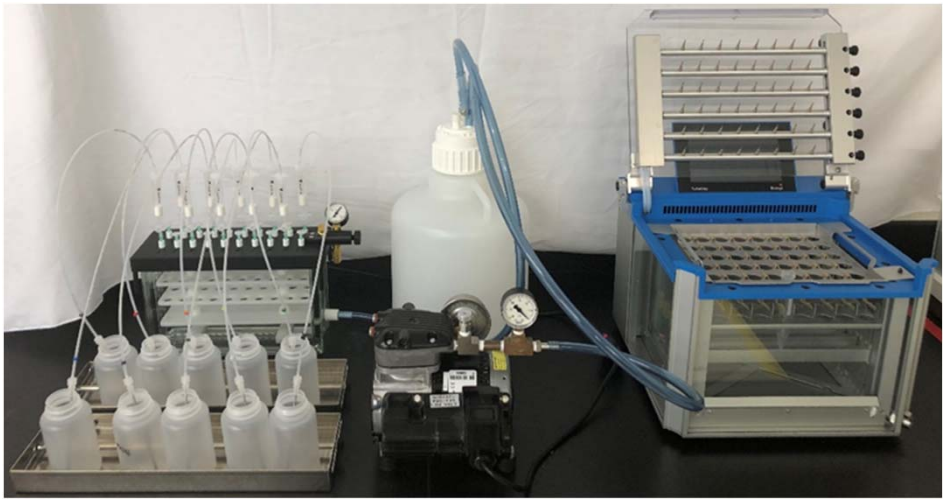
Benchtop Design: A Look at the Workflow



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Summary of SPE Method 537.1

SPE Column Format
ISOLUTE® 101 500 mg/6 mL p/n 101-0050-C

Sample Pre-Treatment
Add 0.94 g of tris(hyroxymethyl)aminomethane and 660 µL of Concentrated HCl to each 250 mL sample, check pH is 7 ± 0.5. Add surrogate standards.

Conditioning
Condition each column with methanol (15 mL)

Equilibration
Equilibrate each column with reagent water (18 mL + 3 mL)

Sample Loading
Load sample (250 mL) at a flow rate of 15 mL/min

Wash
Rinse the column with reagent water
(sample bottle rinsate, 2 x 7.5 mL)

Dry
Dry the column for 5 minutes using a vacuum of -10 to -15 in. Hg.

Elution
Elute the analytes with methanol (sample bottle rinsate 2 x 4 mL)

Post Extraction
Evaporate to dryness and reconstitute in methanol/water (96/4, v/v, 990 µL). Add IS and mix prior to analysis.

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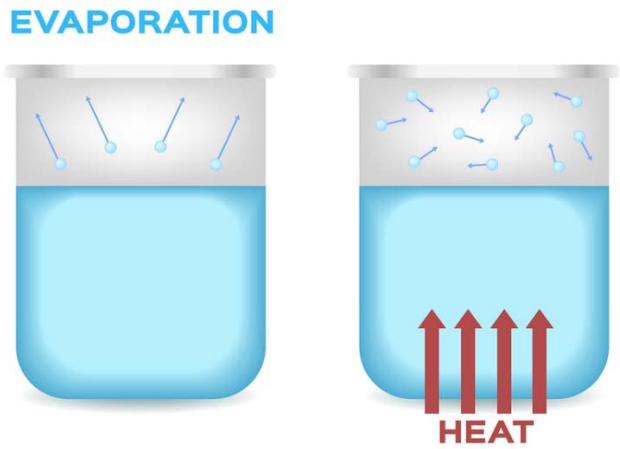
Concentration: Run Conditions

TurboVap® LV Concentration Protocol.

- Bath Temp 60 °C
  - Do not go over 65°C

- Evaporation Mode Method (Ramp Gradient)
  - Step 1 2.5 L/min for 15 min.
  - Step 2 3.0 L/min for 15 min.
  - Step 3 3.5 L/min for 45 min.

- Manifold Setup 48 positions
  - Rack Row Height 120 mm



*The nozzle position was adjusted such that it was as far to the right as possible to give the user a clear view of the vortex within the tube.

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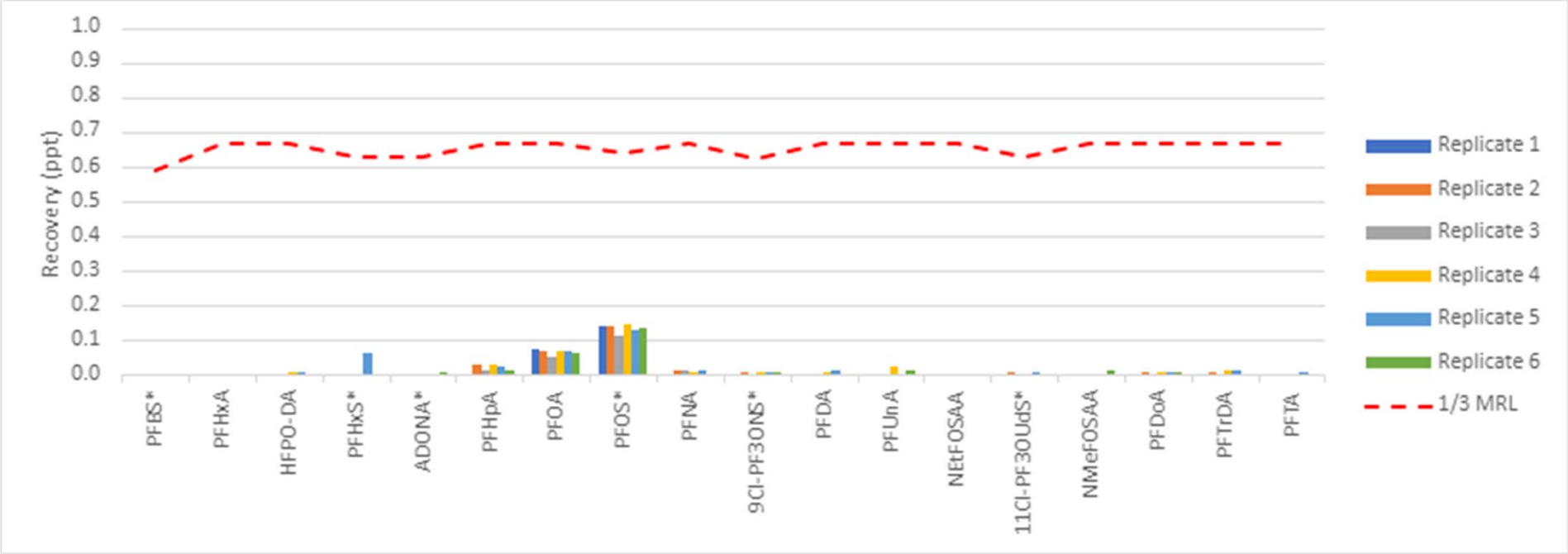
PFAS background of the TurboVap® LV



- Minimum Reporting Level (MRL) of 2 ng/L for all compounds
- This ensures concentration step is clean and no significant background.

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Results of PFAS Evaporation Background Study

	TurboVap® LV					
Replicate	1	2	3	4	5	6
PFBS*	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFHxA	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
HFPO-DA	N.D.	N.D.	N.D.	0.002	0.006	N.D.
PFHxS*	N.D.	N.D.	N.D.	N.D.	0.061	N.D.
ADONA*	N.D.	N.D.	N.D.	N.D.	N.D.	0.004
PFHpA	N.D.	0.030	0.012	0.031	0.024	0.012
PFOA	0.073	0.069	0.050	0.071	0.071	0.063
PFOS*	0.143	0.143	0.115	0.145	0.130	0.137
PFNA	N.D.	0.015	0.013	0.005	0.014	N.D.
9Cl-PF3ONS*	N.D.	0.004	N.D.	0.005	0.002	0.003
PFDA	N.D.	N.D.	N.D.	0.005	0.013	N.D.
PFUnA	N.D.	N.D.	N.D.	0.026	N.D.	0.012
NEtFOSAA	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
11Cl-PF3OUdS*	N.D.	0.006	N.D.	N.D.	0.003	N.D.
NMeFOSAA	N.D.	N.D.	N.D.	N.D.	N.D.	0.013
PFDoA	N.D.	0.004	N.D.	0.007	0.008	0.007
PFTrDA	N.D.	0.003	N.D.	0.013	0.014	N.D.
PFTA	N.D.	N.D.	N.D.	N.D.	0.004	N.D.

- PFOS and PFA were the most consistent
- All analytes were well below the 1/3 MRL limit for EPA 537.1

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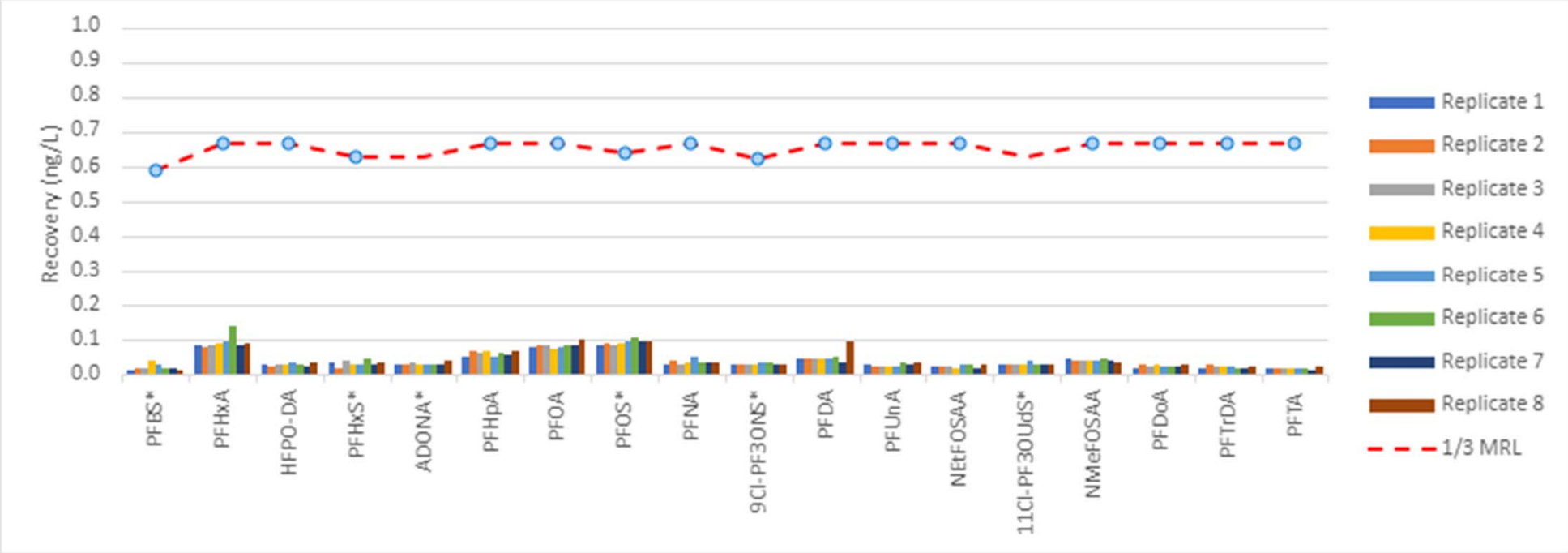
Laboratory Reagent Blanks: PFAS Background



» With Full 537.1 solution including the concentration step

Laboratory Reagent Blanks: PFAS Background

Replicate	Laboratory Reagent Blank							
PFBS*	1	2	3	4	5	6	7	8
PFHxA	0.011	0.020	0.019	0.042	0.033	0.017	0.021	0.011
HFPO-DA	0.084	0.082	0.083	0.089	0.094	0.139	0.087	0.090
PFHxS*	0.032	0.024	0.031	0.028	0.038	0.032	0.026	0.036
ADONA*	0.034	0.019	0.042	0.031	0.032	0.050	0.030	0.034
PFHpA	0.032	0.031	0.034	0.031	0.031	0.032	0.028	0.040
PFOA	0.050	0.069	0.064	0.067	0.054	0.065	0.057	0.068
PFOS*	0.082	0.083	0.085	0.076	0.081	0.088	0.086	0.102
PFNA	0.086	0.091	0.086	0.091	0.097	0.110	0.097	0.095
9Cl-PF3ONS*	0.030	0.039	0.032	0.038	0.052	0.038	0.034	0.037
PFDA	0.029	0.033	0.029	0.031	0.037	0.035	0.033	0.032
PFUnA	0.046	0.050	0.045	0.045	0.048	0.051	0.037	0.097
NEtFOSAA	0.032	0.025	0.027	0.027	0.025	0.034	0.029	0.035
11Cl-PF3OUdS*	0.026	0.025	0.025	0.020	0.029	0.032	0.021	0.032
NMeFOSAA	0.028	0.029	0.028	0.030	0.039	0.033	0.030	0.030
PFDoA	0.048	0.044	0.044	0.041	0.044	0.047	0.039	0.034
PFTrDA	0.022	0.031	0.027	0.028	0.025	0.025	0.027	0.029
PFTA	0.022	0.028	0.025	0.026	0.023	0.021	0.018	0.025

- Sample prep workflow does contribute to PFAS
- Important to know all contributing PFAS sources
- All analytes were well below the 1/3 MRL limit for EPA 537.1

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Turbovap LV Evolution

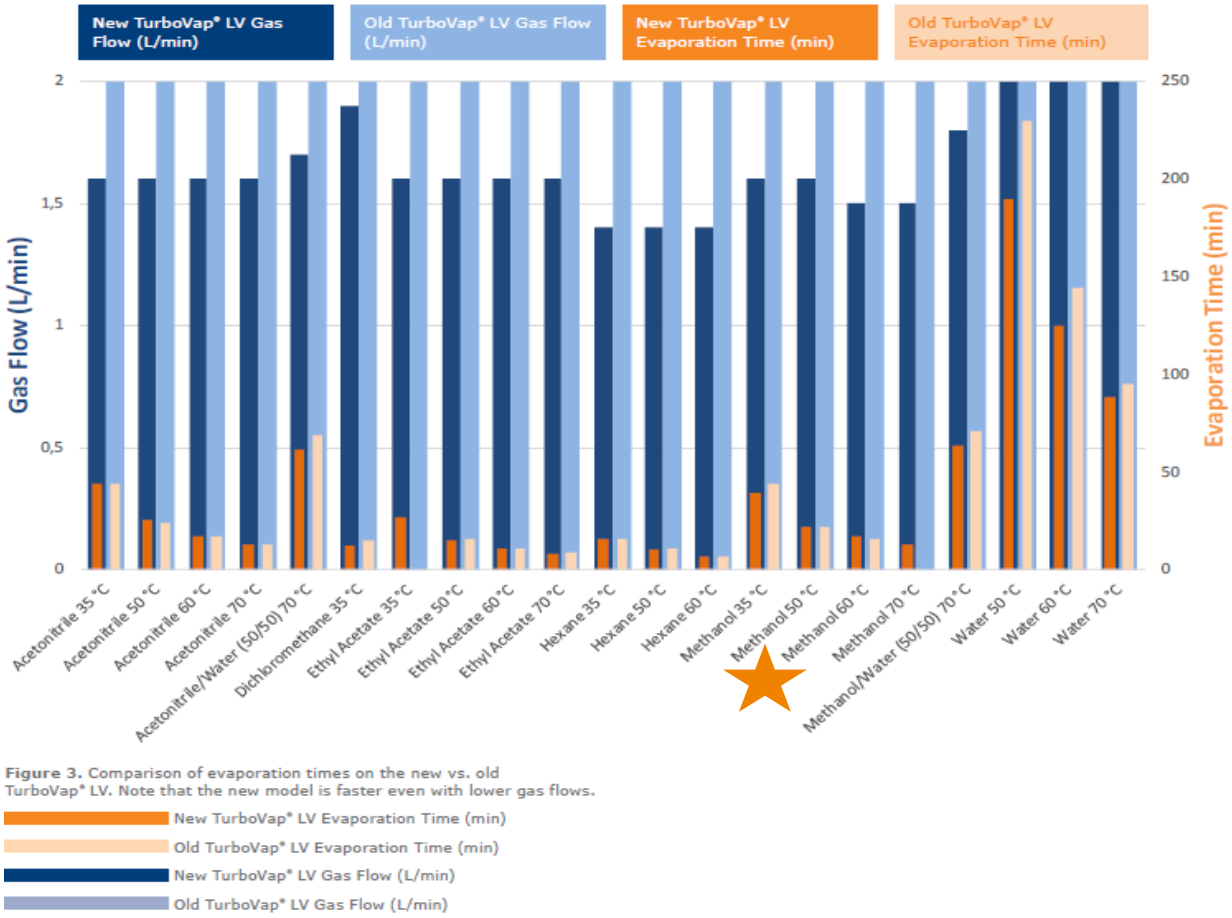

Figure 3. Comparison of evaporation times on the new vs. old TurboVap® LV. Note that the new model is faster even with lower gas flows.

New TurboVap® LV Evaporation Time (min)

Old TurboVap® LV Evaporation Time (min)

New TurboVap® LV Gas Flow (L/min)

Old TurboVap® LV Gas Flow (L/min)

Background Results

- The TurboVap LV is a fast, consistent and reliable instrument to evaporate PFAS analytes
- The instrument does not contribute much to the overall demonstrated PFAS compound background as many analytes were ND.
- Can concentrate up to 48 samples at once with great evaporation precision
    - Will not bottleneck workflows

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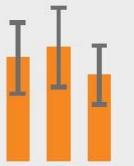
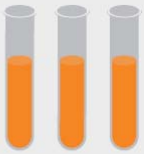
Concentration Workflow



Reduced
RSD

Faster
Evaporation



Improved
Recovery

Questions and Answers

Thank you for attending
For more information, please visit
Biotage.com for more
PFAS Solution!

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