Analysis of Semi-Volatile Organics in Drinking Water with Semi-Automated Solid Phase Extraction Using EPA Method 525.3

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Semi Volatiles

- Found in drinking water
- In US regulated by EPA method 525.3
- Also regulated elsewhere in the world
- Great demand for fast, reliable and reproducible laboratory analysis

Health Effects SVOCs

- Allergic symptoms
- Delayed reproductive development
- Immunotoxicity
- Cancer
- Asthma (in dust)
- Suspected endocrine disruption

Analysis for SVOCs

- Many labs analyze drinking and waste water samples
- Liquid-Liquid Extraction (LLE) or Solid Phase Extraction (SPE) can be used
- In both cases organics are transferred from water sample to an organic solvent
- With SPE compounds are first deposited on cartridge or disk, then eluted

Comparison of LLE/CLE vs SPE Methods (1)

LLE/CLE	Semi-Automated SPE
Open to laboratory background	Closed system
Uses >360mls solvent	Uses <60mls solvent
Shaking / Continuous process	Filtration process
Forms emulsions requiring centrifuging	No emulsions formed
Little Selectivity	Wide Selectivity (adsorbent)
Requires water removal	In-line water removal

Comparison of LLE/CLE vs SPE Methods (2)

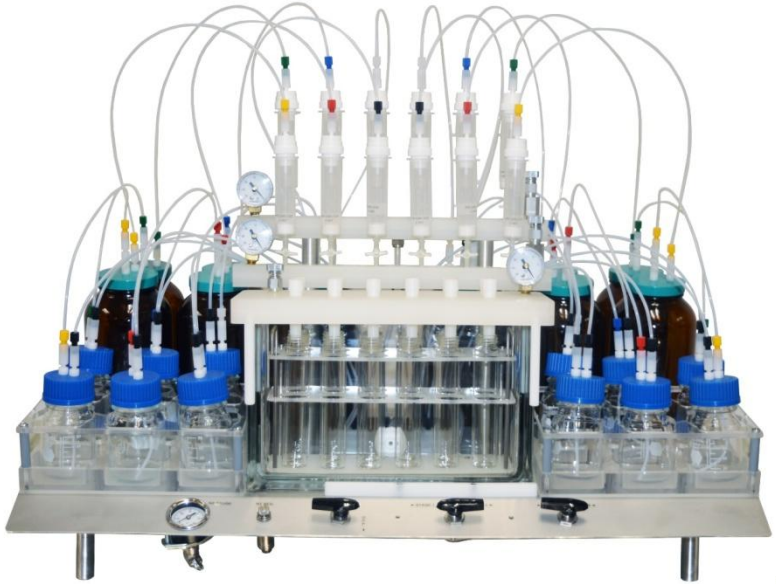
LLE/CLE	Semi-Automated SPE
No Separation of waste Waste	Separates Aqueous and Organic
More volume to evaporate	<60mls solvent to evaporate
High solvent emission	6 times less solvent emission
CLE uses a lot of solvent	Easily Capture Solvent
Requires lots of solvent for cleaning	Lower solvent costs
	Lower Disposal Costs

Reduced Solvent Usage

Semi-Automated SPE

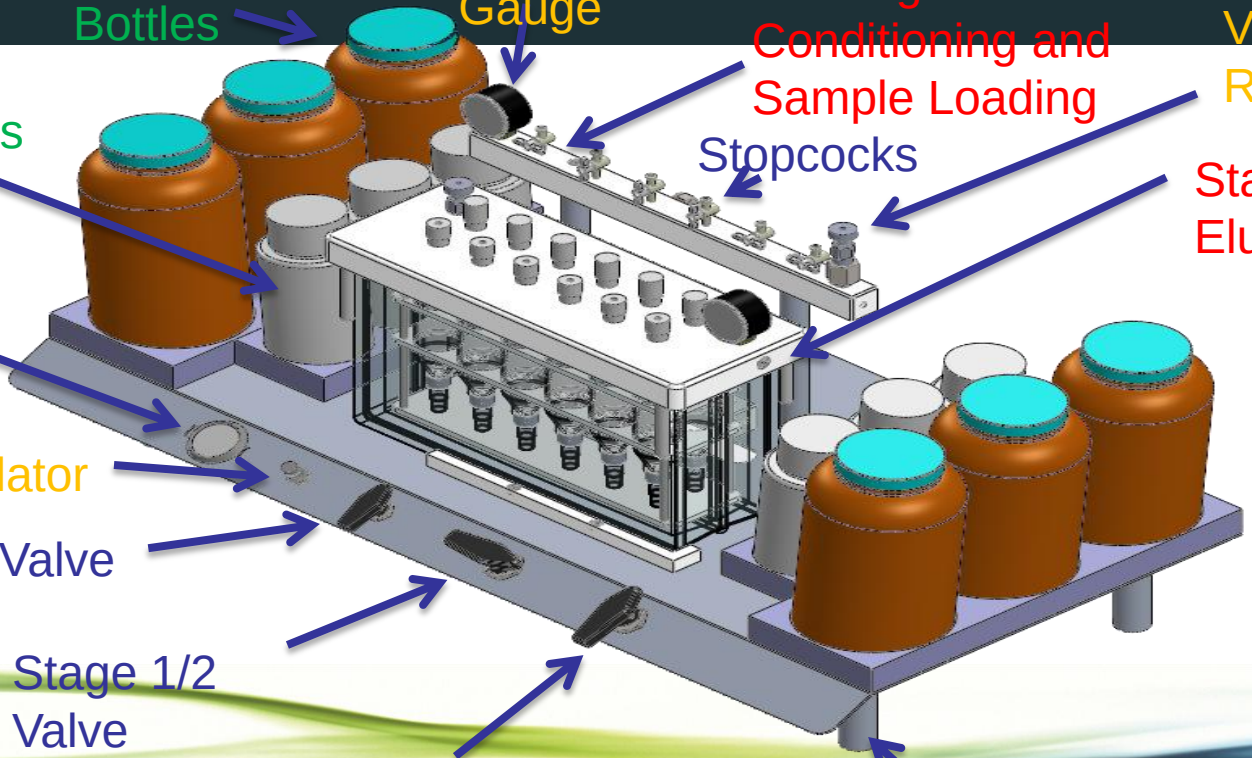
- Semi-automated SPE done by many labs around the world
- Cheaper than fully automated systems
- Important that system is reliable and fast
- Should be able to use variety of cartridges

Semi-Automated FMS System (EZSpe™)

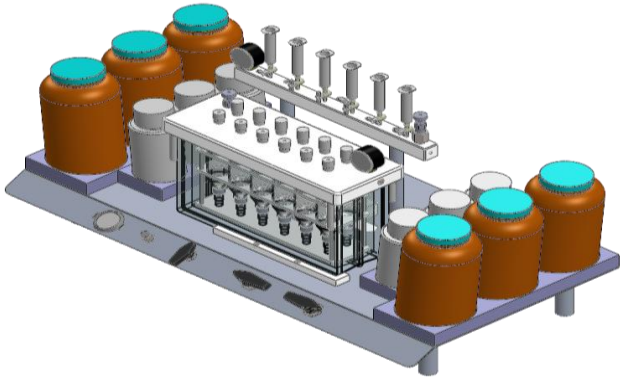


System Layout

FMS
Fluid Management Systems



Cartridge Conditioning (Stage 1, Organic Waste)

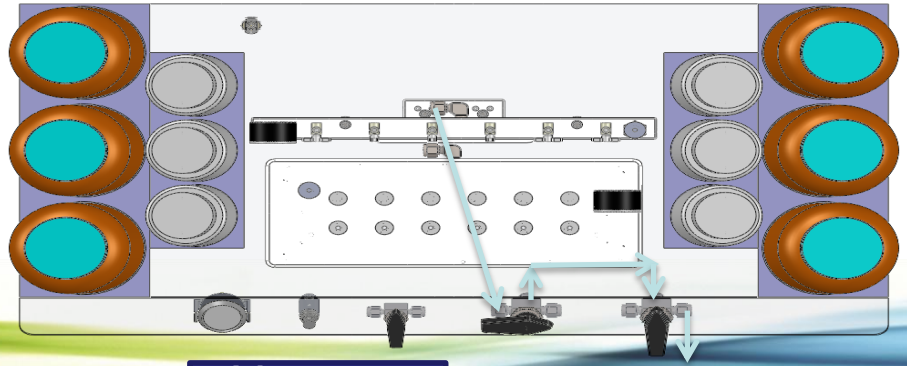


Flow Path

Nitrogen Valve

Stage 1/2 Valve

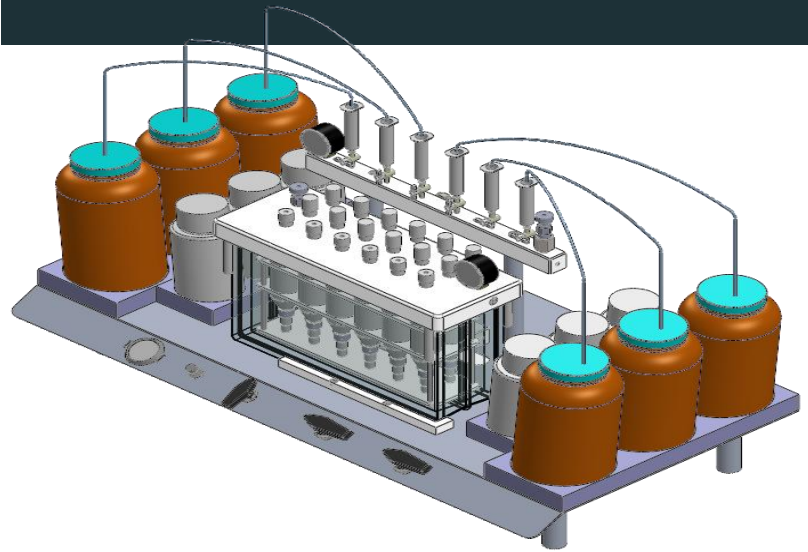
Waste Valve

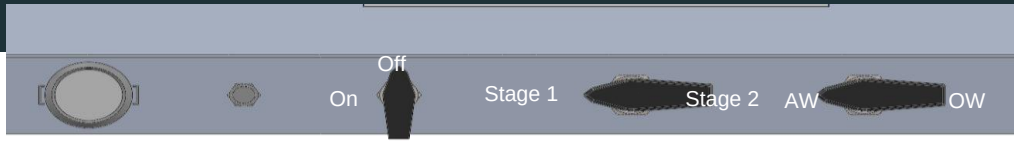


Vacuum Pump

OW

Sample Loading
(Stage 1, Aqueous Waste)

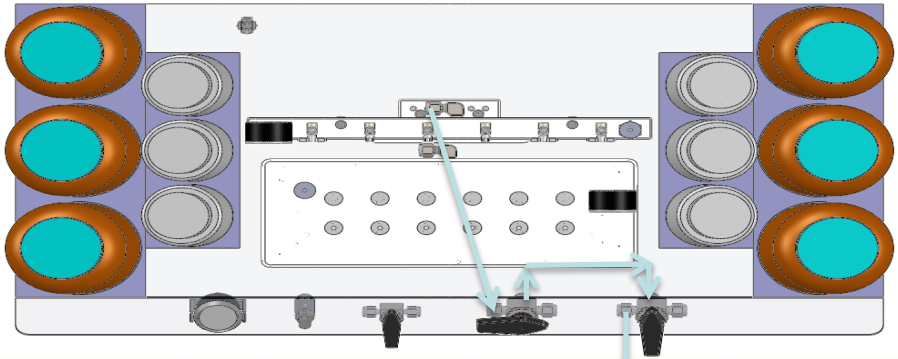




Nitrogen Valve

Stage 1/2 Valve

Waste Valve

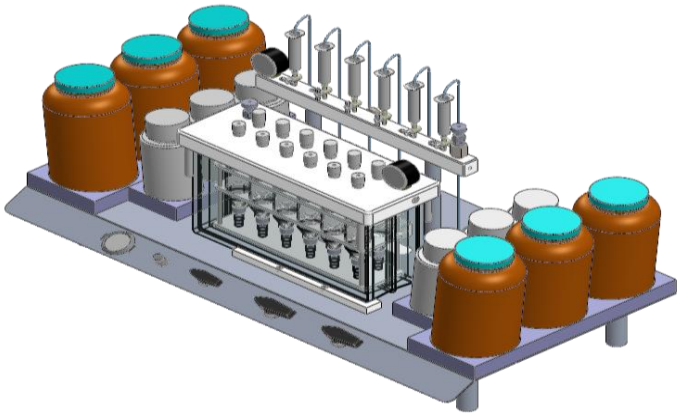


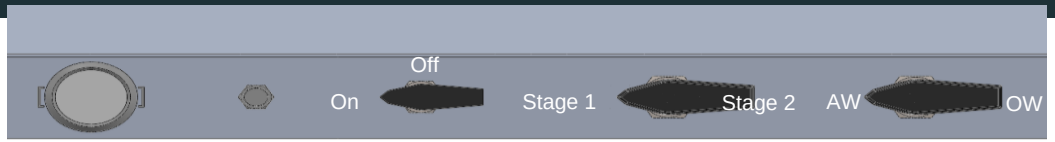
Flow Path

Vacuum Pump

AW

Cartridge Drying- Nitrogen/Vacuum

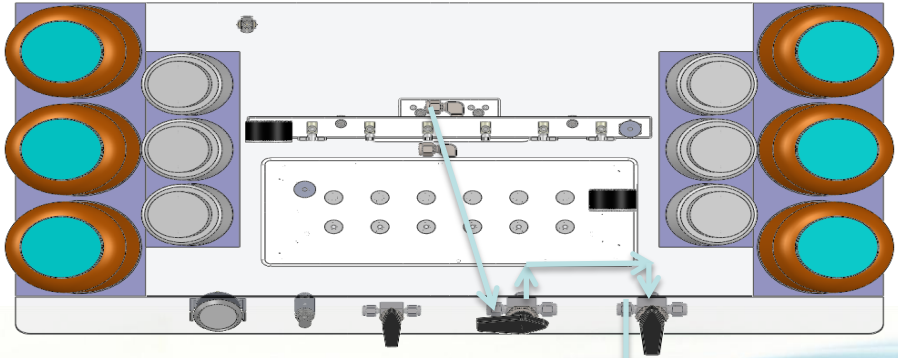




Nitrogen Valve

Stage 1/2 Valve

Waste Valve

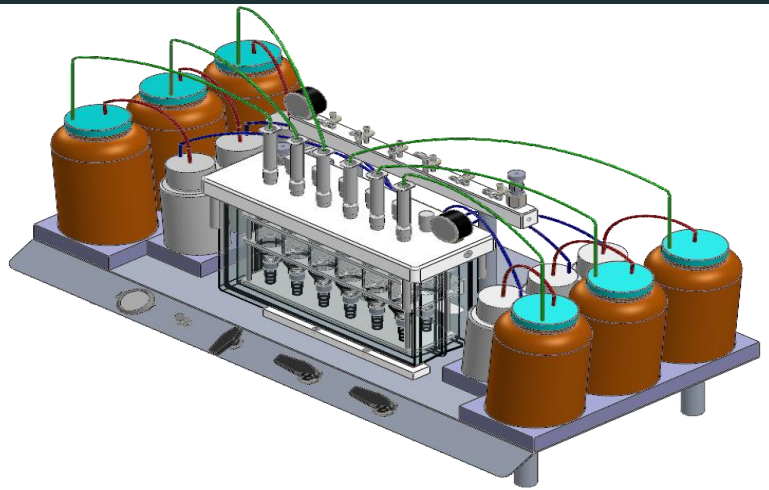


Flow Path

Vacuum Pump

AW

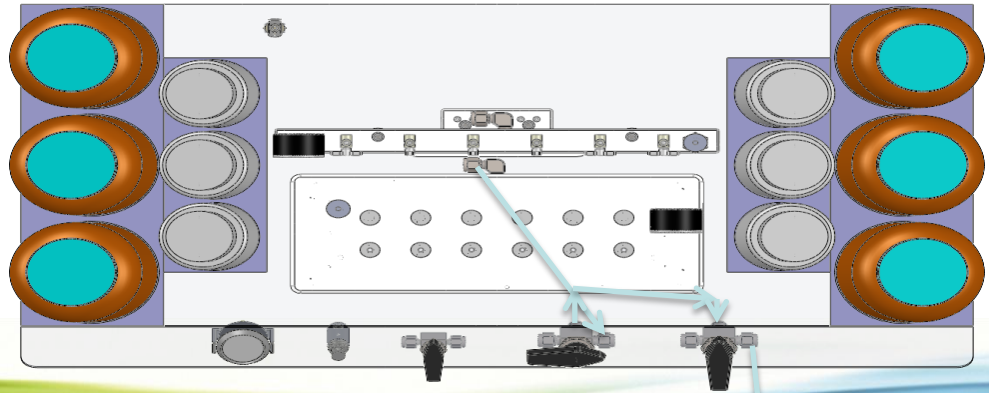
Sample Elution (Stage 2)



Nitrogen Valve

Stage 1/2 Valve

Waste Valve



Flow Path

Vacuum Pump

OW

Attributes EZSpe (1)

- Simple to Operate No Computer or Electronics
- Fast Runs 6 Samples in 20 ~ 50 min (depending on sample size)
- High Throughput Runs 6 Samples in Parallel
- Flexible Uses All SPE Cartridge Sizes
- Semi Automated Vacuum Sample Loading & Valve Selection for Separating Aqueous and Organic Waste

Attributes EZSpe (2)

- Quality Consumables: Guaranteed Certified Cartridges
- Bottle Rinse: Automated Bottle Rinse
- In-line Extract Drying
- Reliable No Maintenance Required
- Zero Cross-Contamination, No Shared Tubing & Fittings

Procedure (1)

- 6 samples (1L water each) are prepared and acidified with 1 mL HCl till pH ~ 2
- Spike with 525.3 standards
- Put sample bottles in place and fill rinse bottles with 10 mL water
- SDVB cartridges are installed in each of the six positions.

Procedure (2)

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 5 mL ethyl acetate, 10 mL methanol (soak 1 min) and 10 mL water (soak 1 min)
- Samples are loaded across cartridges under vacuum
- Cartridges are dried with vacuum for 10 min
- Sample bottles are automatically rinsed from the rinse bottles with 25 mL dichloromethane

Procedure (3)

Stage 2:

- Rinse bottles filled with 5 mL ethyl acetate, rinse sample bottles, load across cartridges, collect
- Same for 5 mL dichloromethane, collect
- In line sodium sulfate drying

12 position evaporator 50 mLs

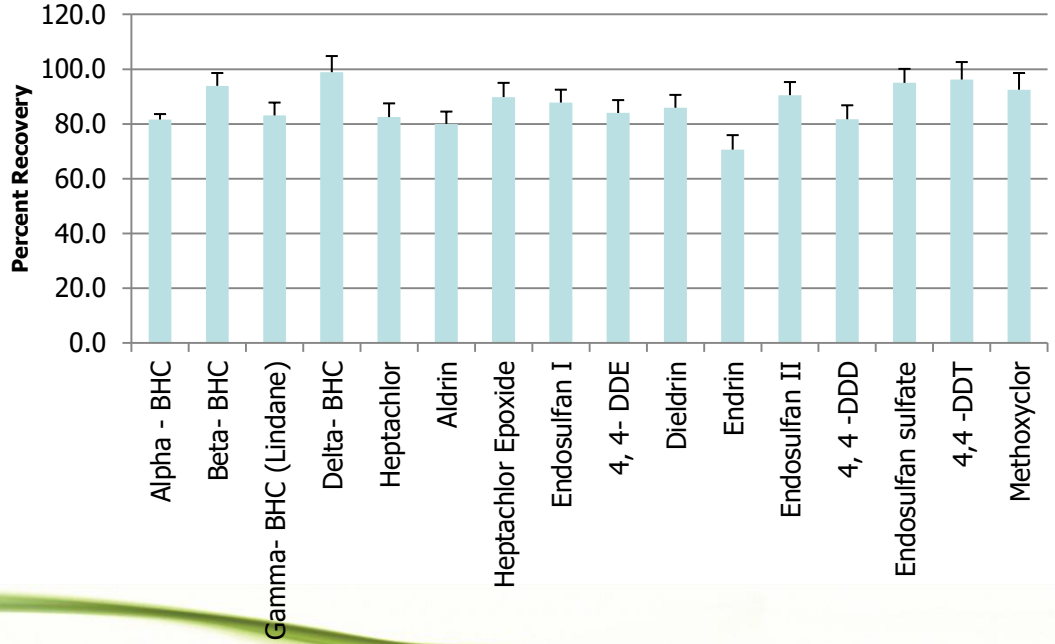
SuperVap Features

- 6 (250mL) and 12 (50mL) position models for extractions, direct-to-vial connections
- Dry bath heating element
- Independent secondary heater for extract nipple (can be disabled)
- Sensor controlled
- Savable temperature log capability.

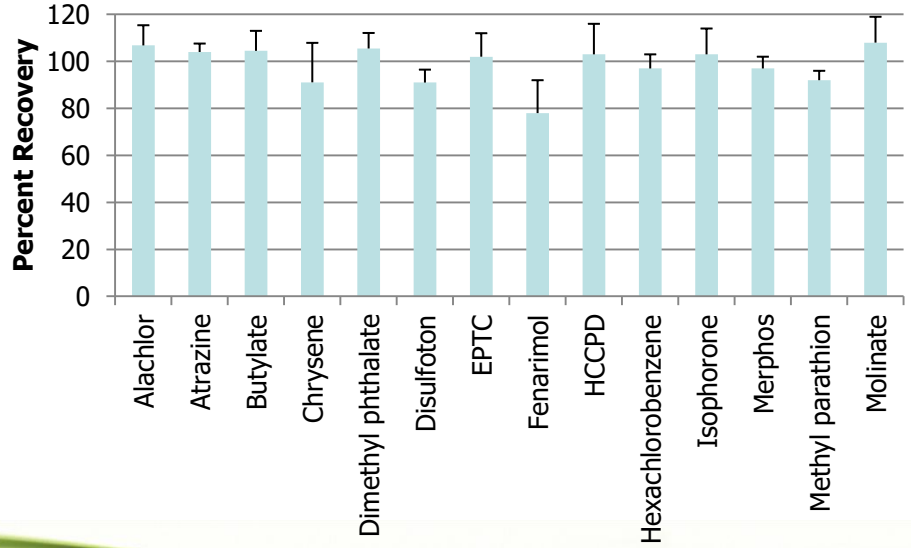
Analysis

- Samples reduced to 1 mL under nitrogen flow
- Samples analyzed in 1 mL DCM
- Semi-Volatiles analyzed with low resolution GC/MS (full scan)

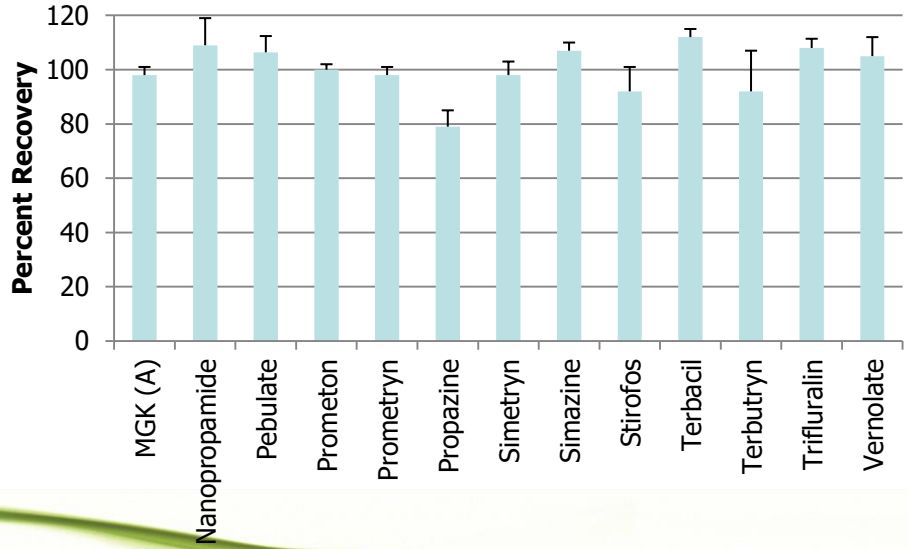
525.3 OCPs (Drinking Water)



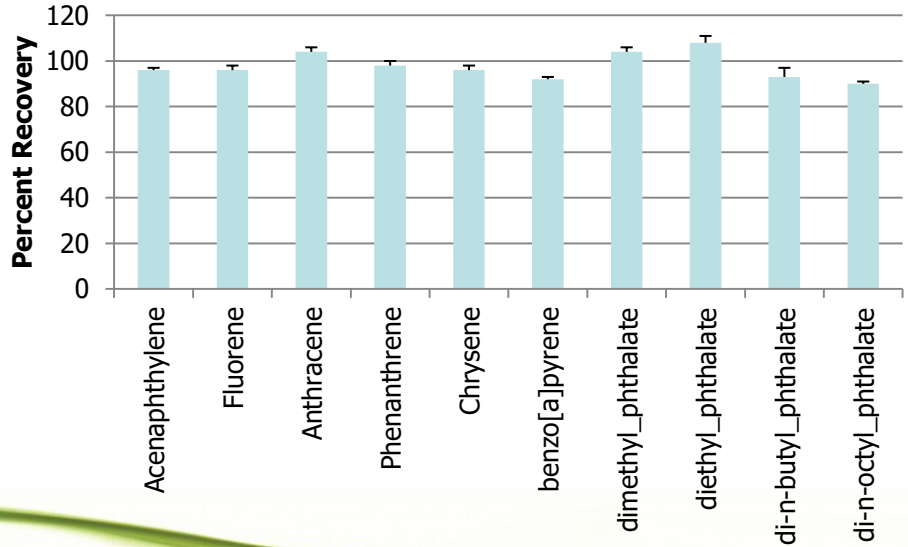
525.3 SVOCs (Drinking Water, 1)



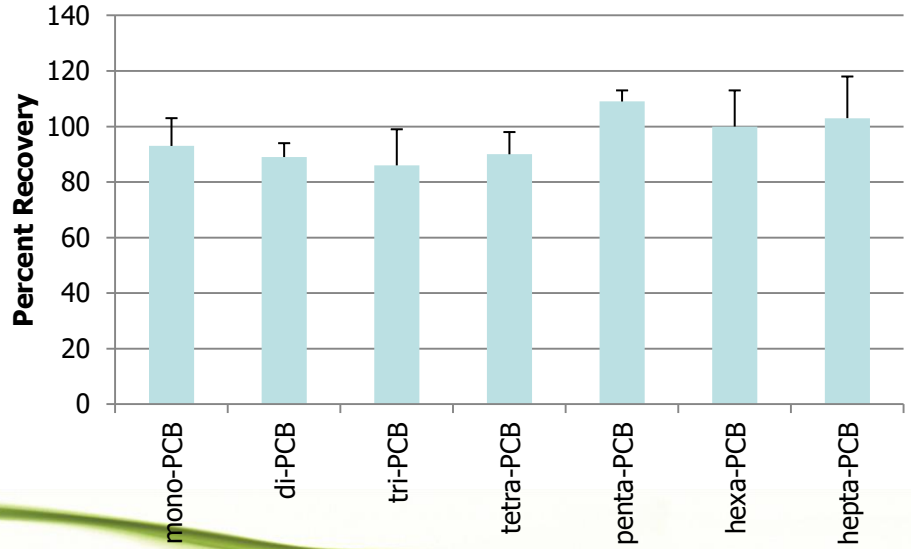
525.3 SVOCs (Drinking Water, 2)



525.3 PAHs and phthalates (Drinking Water)



525.3 PCBs (Drinking Water)



Conclusions

- EZSpe delivers excellent recoveries for 525.3 analytes
- Runs 6 samples in parallel
- Gets data in under 2h
- No maintenance required
- No separate water removal step needed (in-line drying)
- Other applications are beverages, milk and serum

Questions

- Questions?
- See us at booth # 4043.