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Analysis of Organochlorine Pesticides and Semi-Volatile Organics in Water with Semi-Automated Solid Phase Extraction Using EPA Methods 508 and 525.3

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Introduction

- Organochlorine pesticides are man-made organic chemicals with a history of wide spread use around the world
- Persistent to degradation, found in drinking water supplies and sediments
- Some are included in Stockholm Convention (added 2009-2015)
- EPA method 508

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Health risks OCPs

- Organochlorines have a wide range of both acute and chronic health effects
- Cancer
- Neurological effects
- Birth defects
- Many OCPs are also suspected endocrine disruptors

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Semi Volatiles

- Found in drinking water and waste water
- In US regulated by EPA methods 525 and 625
- Also regulated elsewhere in the world
- Great demand for fast, reliable and reproducible laboratory analysis

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Health Effects SVOCs

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

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Analysis for OCPs/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

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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

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Comparison of LLE/CLE vs SPE Methods (2)

LLE/CLE	Semi-Automated SPE
No Separation of waste	Separates Aqueous and Organic Waste
More volume to evaporate	<60mls solvent to evaporate
Massive 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

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Reduced Solvent Usage

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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

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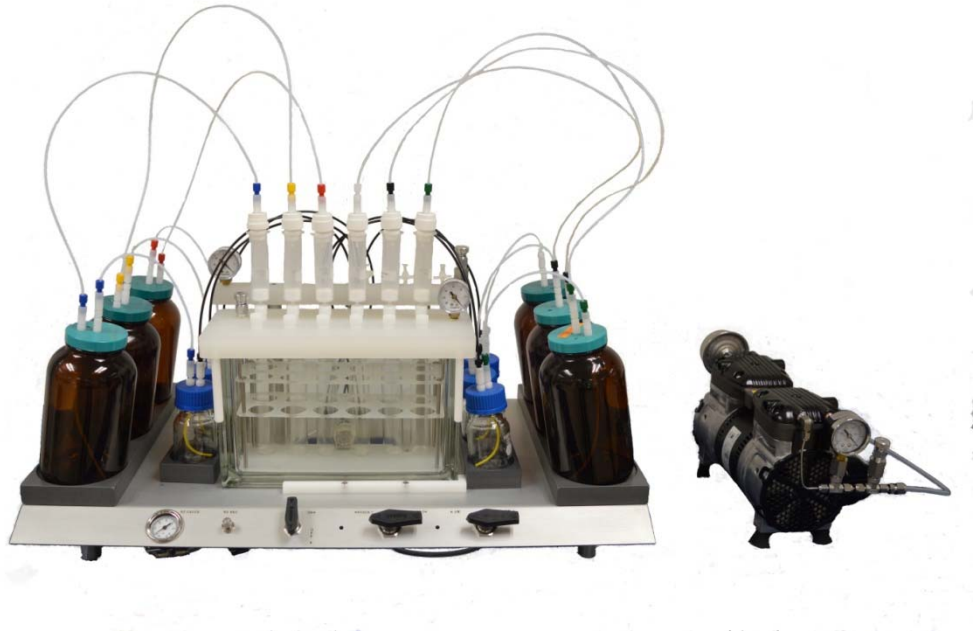
Semi-Automated FMS System (EZSpe™)



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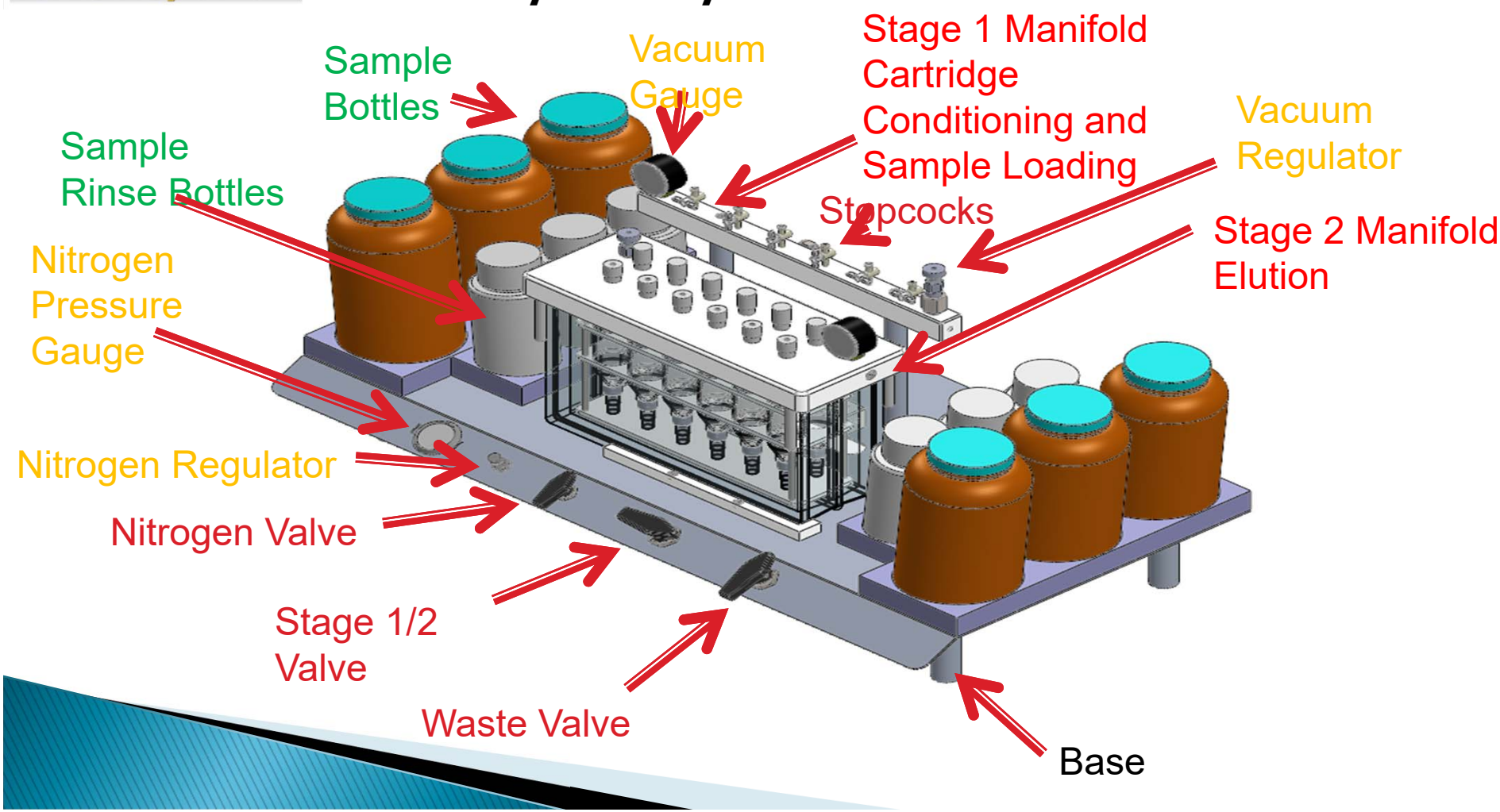
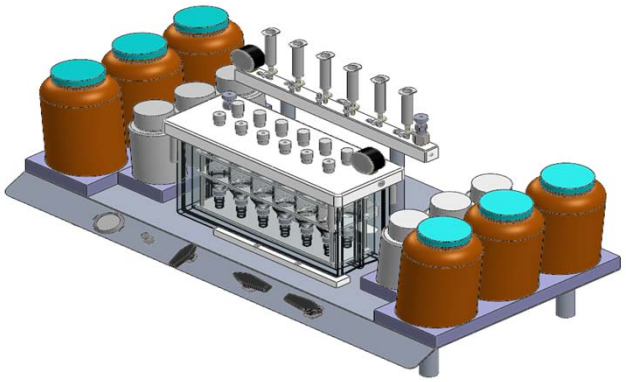
System Layout



Cartridge Conditioning (Stage 1, Organic Waste)

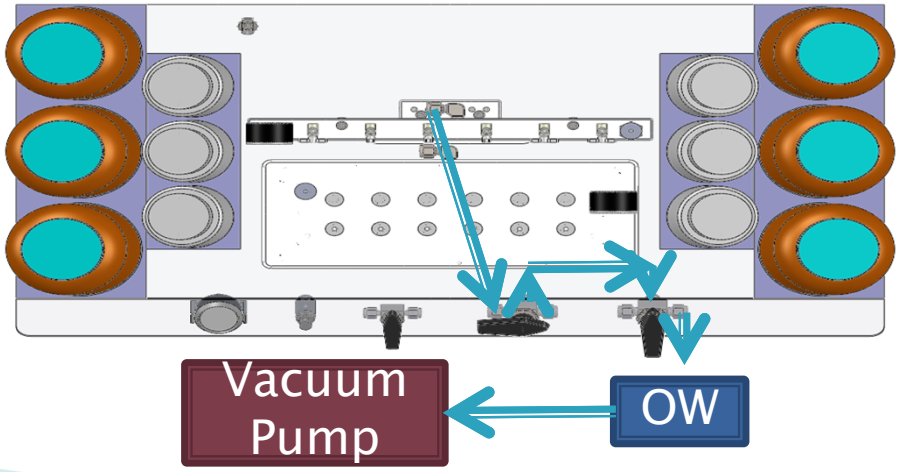


Nitrogen Valve

Stage 1/2 Valve

Waste Valve

Flow Path



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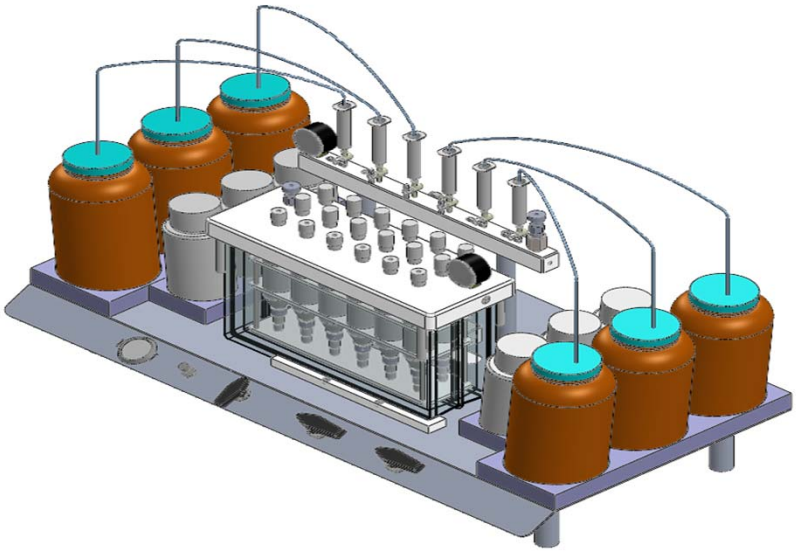
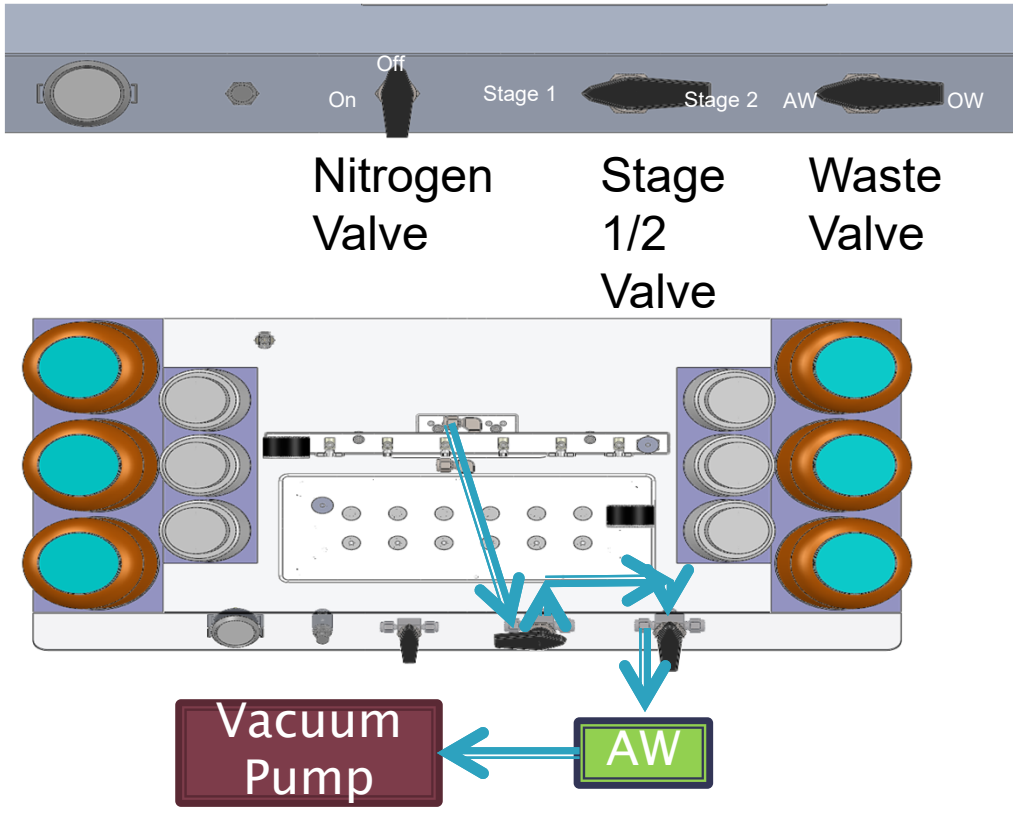
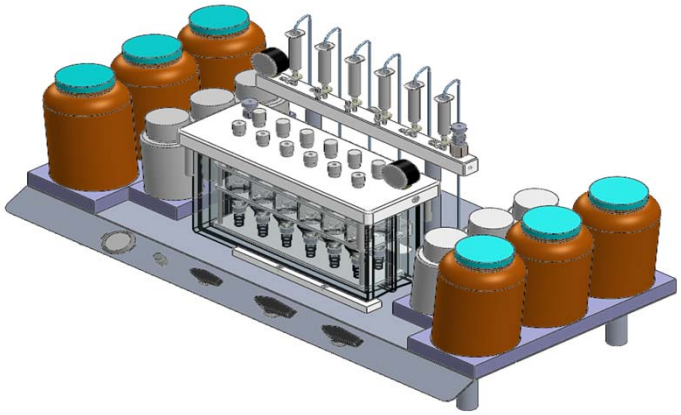
Sample Loading (Stage 1, Aqueous Waste)



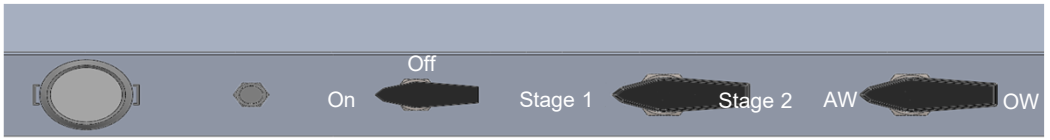
Flow Path



Cartridge Drying- Nitrogen/Vacuum



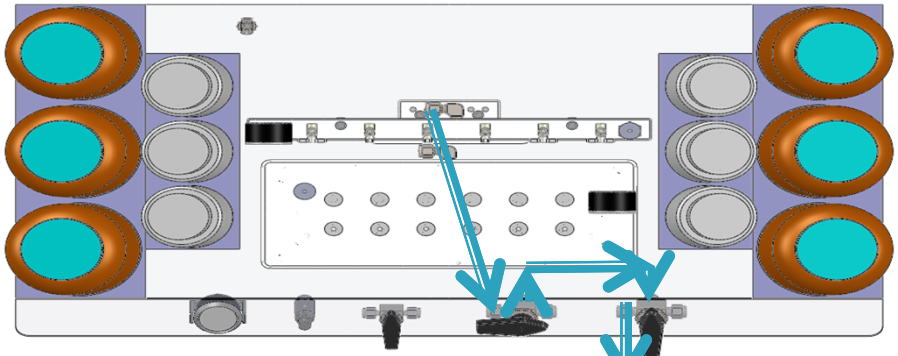
Flow Path



Nitrogen Valve

Stage 1/2 Valve

Waste Valve



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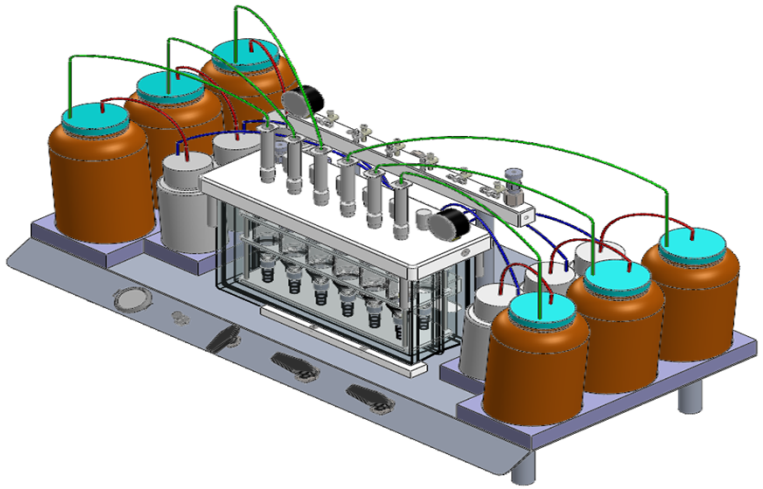
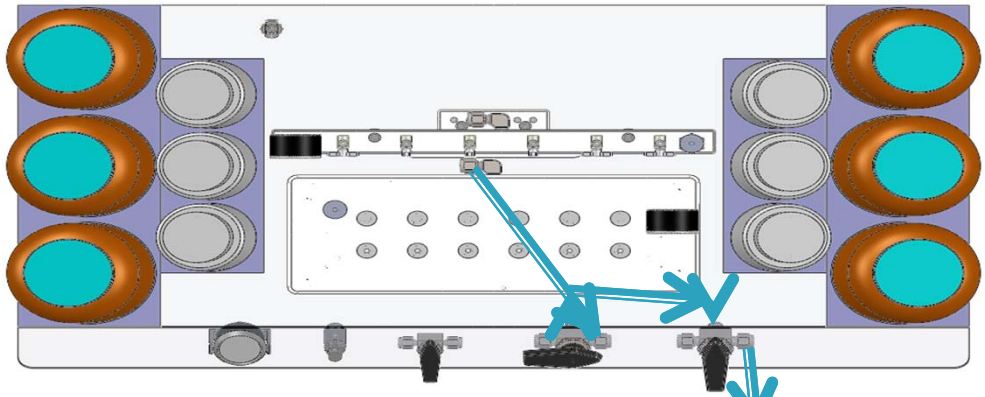
Sample Elution (Stage 2)



Nitrogen Valve

Stage 1/2 Valve

Waste Valve



Flow Path

Vacuum Pump

OW

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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

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Attributes EZSpe (2)

- Quality Consumables Guaranteed Certified Cartridges
- Bottle Rinse Automated Bottle Rinse
- In-Line Drying Elution 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
- Add 5-10 mL methanol and spike with relevant standards
- Put sample bottles in place and fill dichloromethane rinse bottles with 25 mL solvent
- Cartridges are installed in each of the six positions.

Procedure (2)

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 5 mL dichloro-methane, methanol and water
- Samples are loaded across cartridges under vacuum
- Cartridges are dried with nitrogen for 10 min
- Sample bottles are automatically rinsed from the rinse bottles with 25 mL dichloromethane

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Procedure (3)

Stage 2:

- Dichloromethane from sample bottles is loaded across the C18 cartridges and sodium sulfate cartridges
- Eluent is collected for analysis into Direct to GC Vial Collection Vessels

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12 position evaporator 50 mLs

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SuperVap Features

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

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Analysis

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

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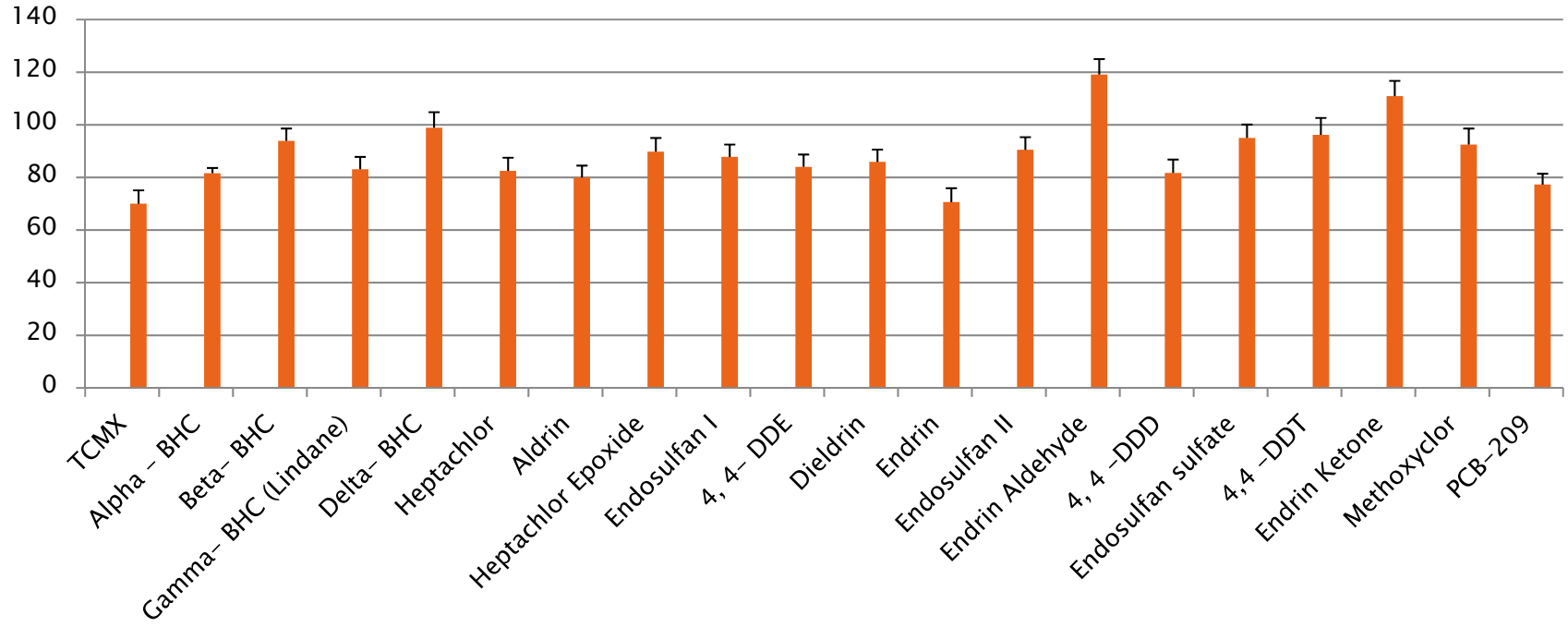
EZSpe data for OCPs (Drinking Water)



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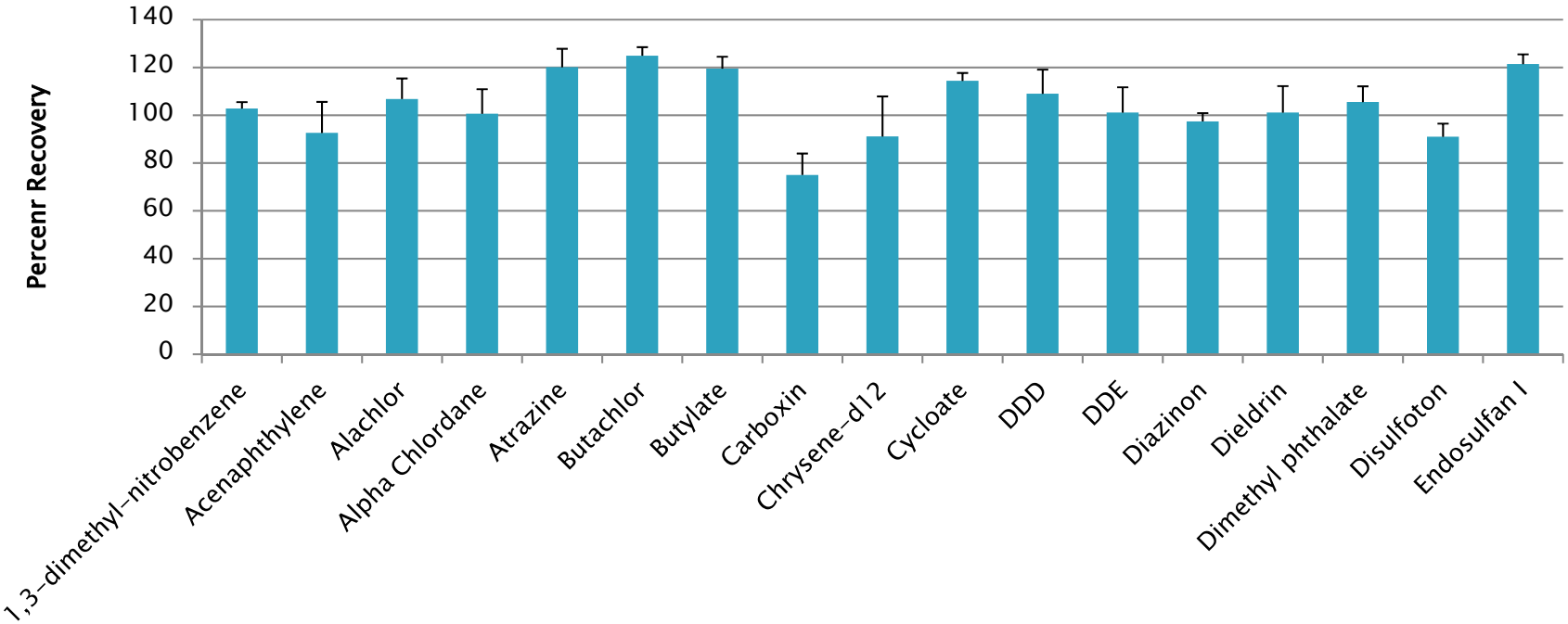
EZSpe data for SVOCs (Drinking Water, 1)



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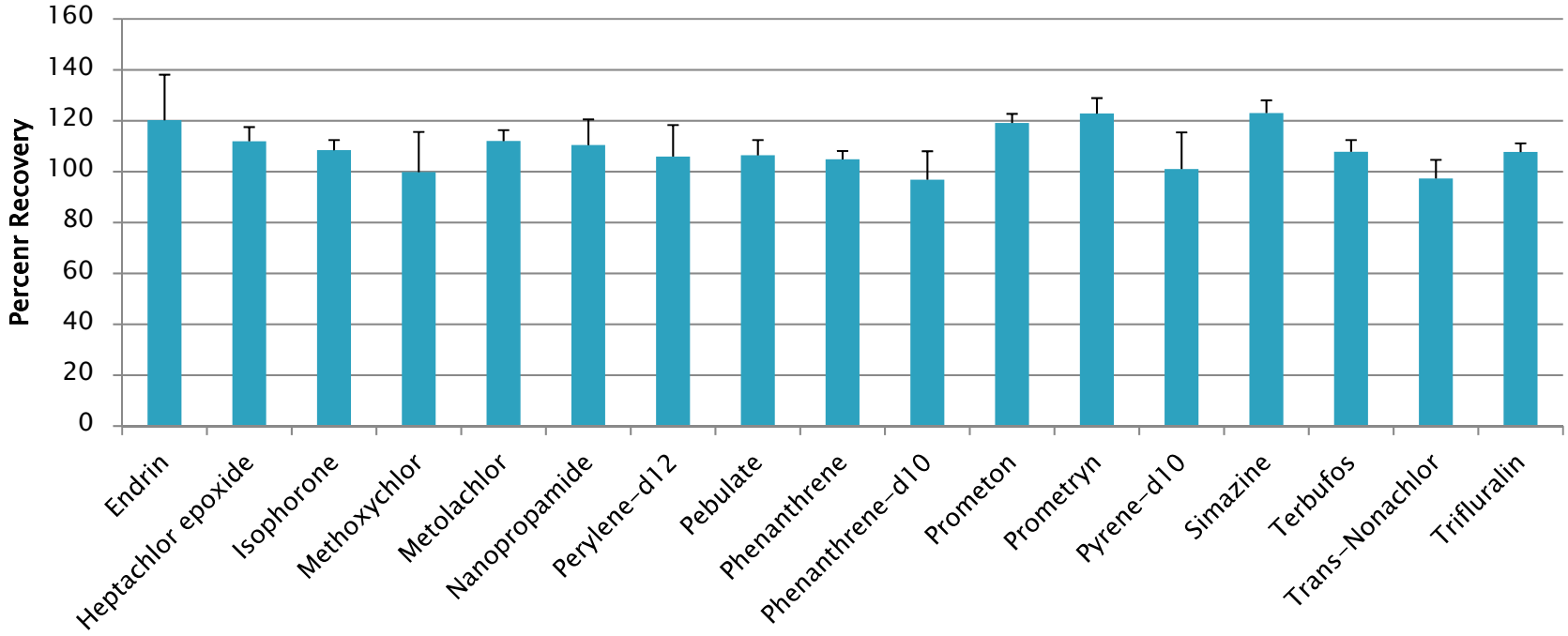
EZSpe data for SVOCs (Drinking Water, 2)



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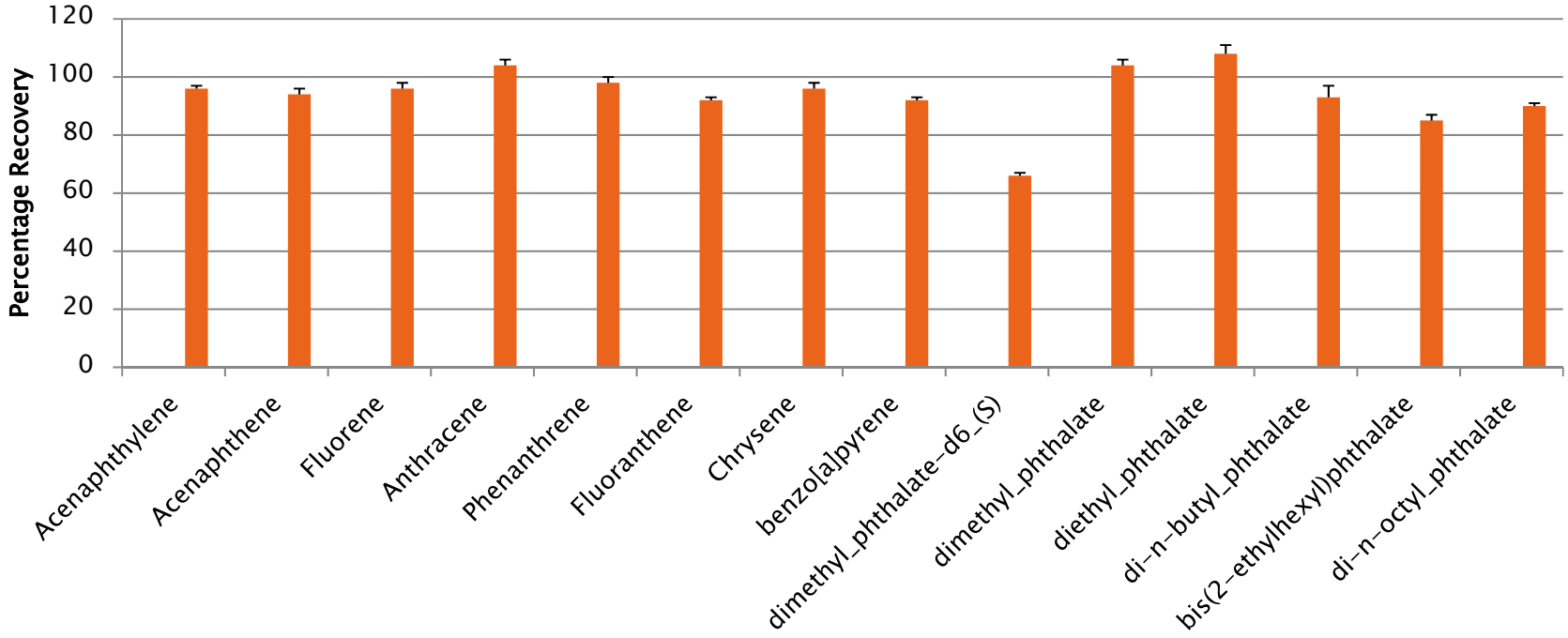
PAHs and phthalates (Waste Water)



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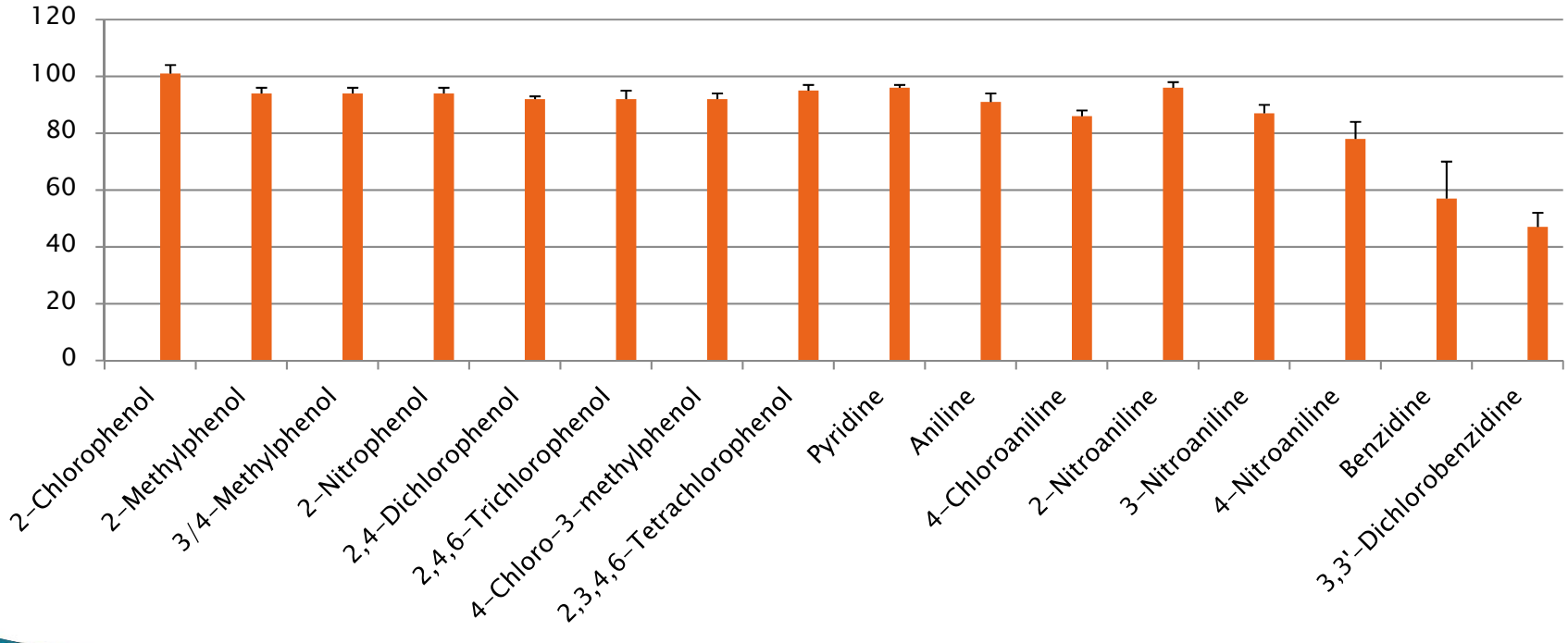
Phenols and Ion Exchangers (Waste Water)



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Conclusions

- EZSpe delivers excellent recoveries for Organochlorine Pesticides and Semi-Volatiles
- 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

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Questions

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