

High Throughput Semi Automated Solid Phase Extraction and Analysis of Waste Water using EPA 8270D

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Introduction Semi Volatiles

- Found in drinking water and waste water
- In US regulated by EPA methods 625.1/8270D
- 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

Extraction/Analysis of 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

Open to laboratory background

Uses >360mls solvent

Shaking / Continuous process

Forms emulsions requiring centrifuging

Little Selectivity

Requires water removal

Semi-Automated SPE

Closed system

Uses <60mls solvent

Filtration process

No emulsions formed

Wide Selectivity (adsorbent)

In-line water removal

Comparison of LLE/CLE vs SPE Methods (2)

LLE/CLE

No Separation of waste

More volume to evaporate

Massive solvent emission

CLE uses a lot of solvent

Requires lots of solvent for cleaning

Semi-Automated SPE

Separates Aqueous and Organic Waste

<60mls solvent to evaporate

6 times less solvent emission

Easily Capture Solvent


Lower solvent costs

Lower Disposal Costs

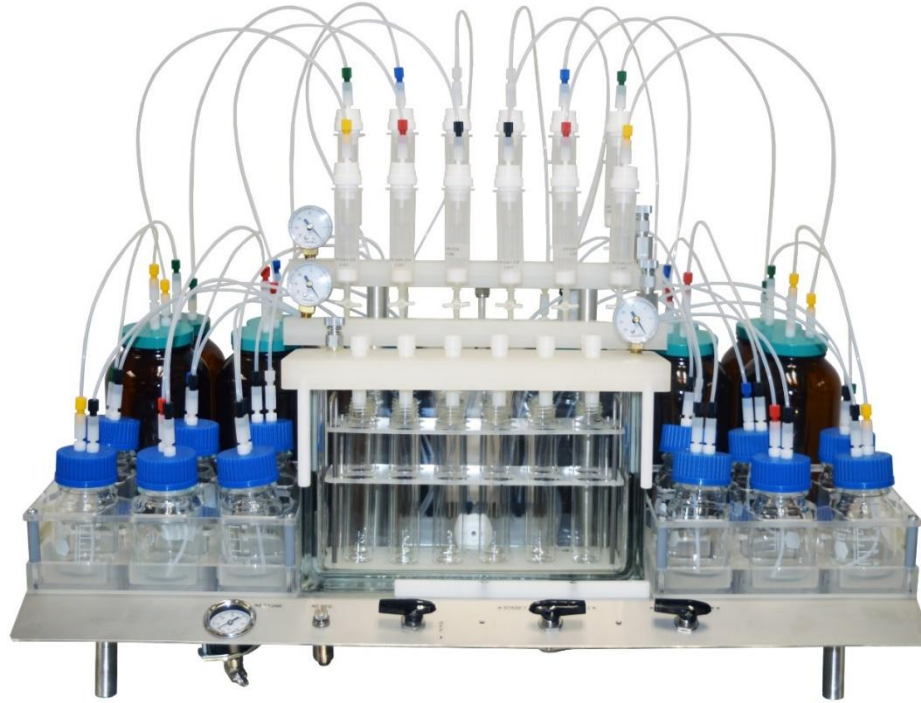
Reduced Solvent Usage



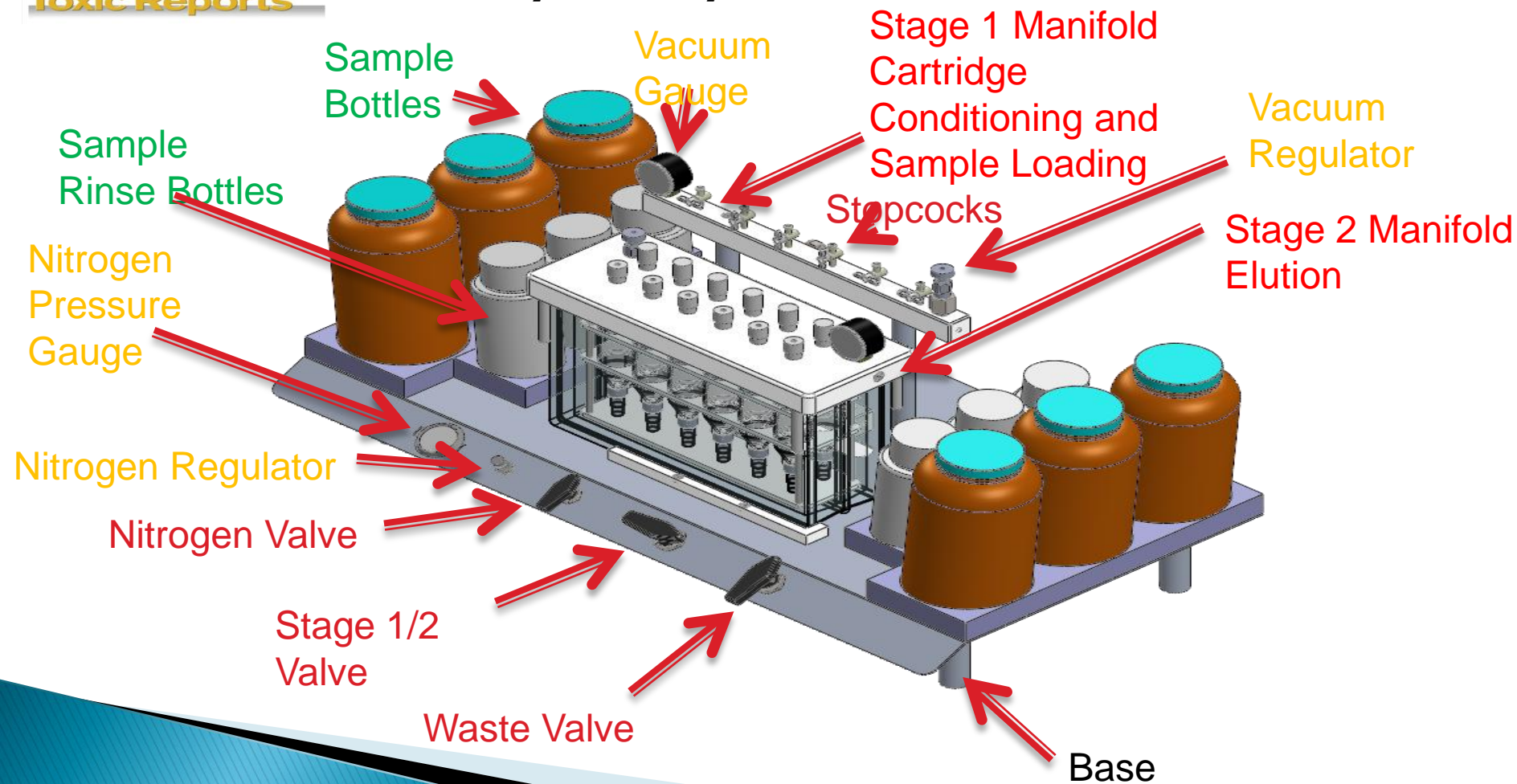
Semi-Automated SPE

- ▶ 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
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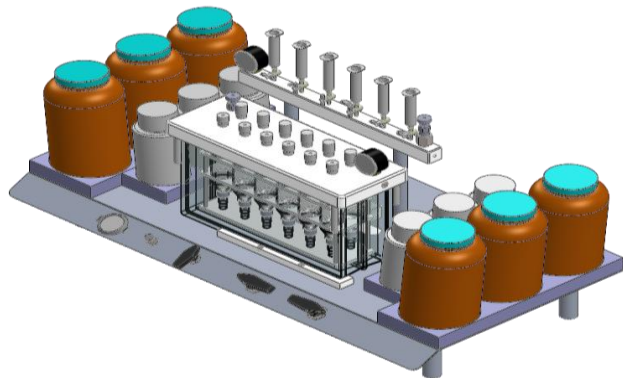
Semi-Automated FMS System (EZSpe®)



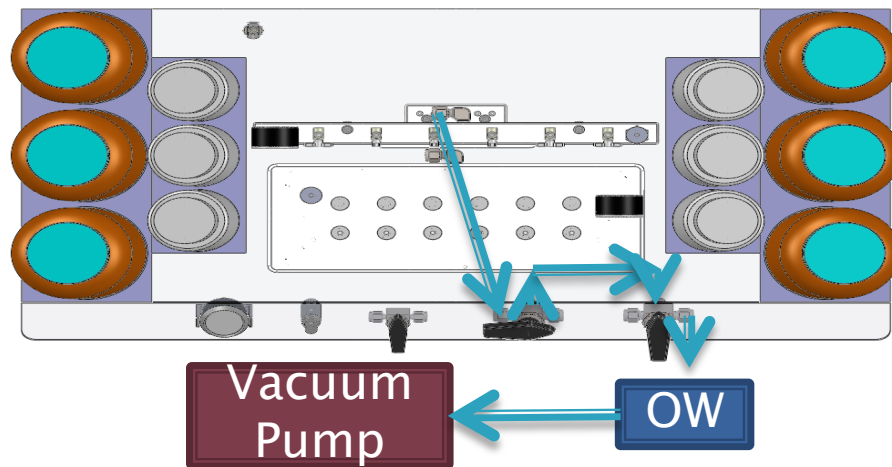
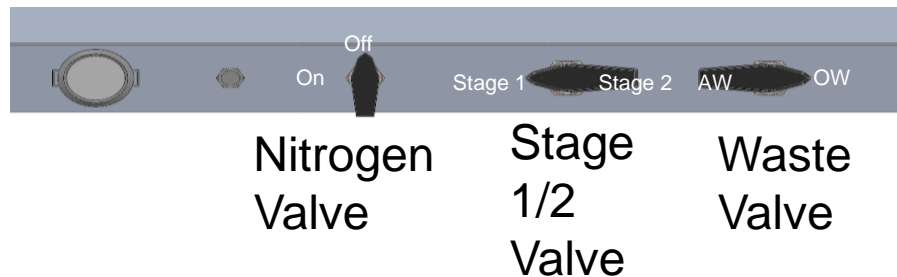
System Layout



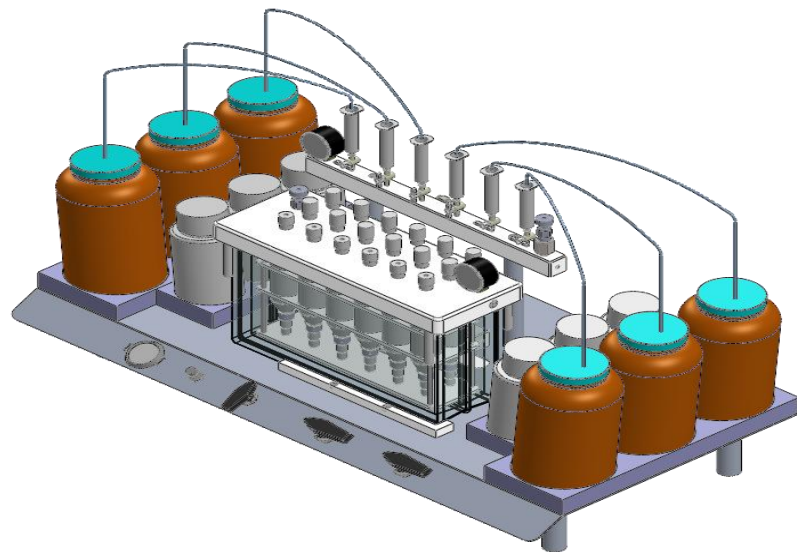
Cartridge Conditioning (Stage 1, Organic Waste)



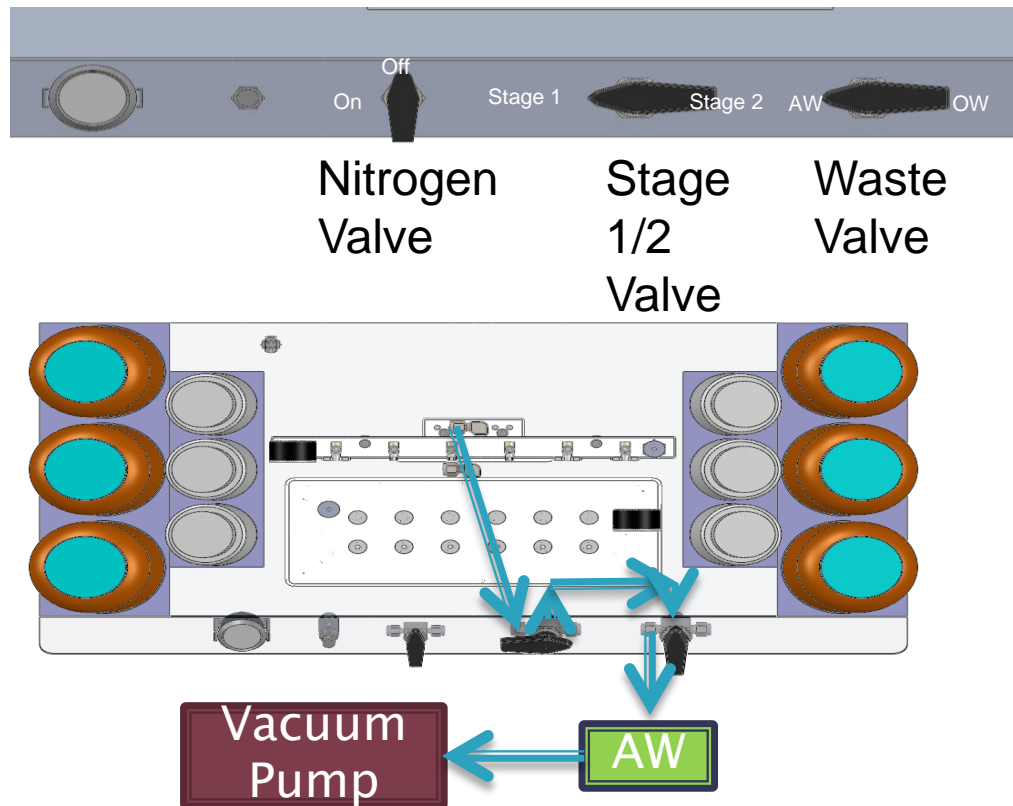
Flow
Path



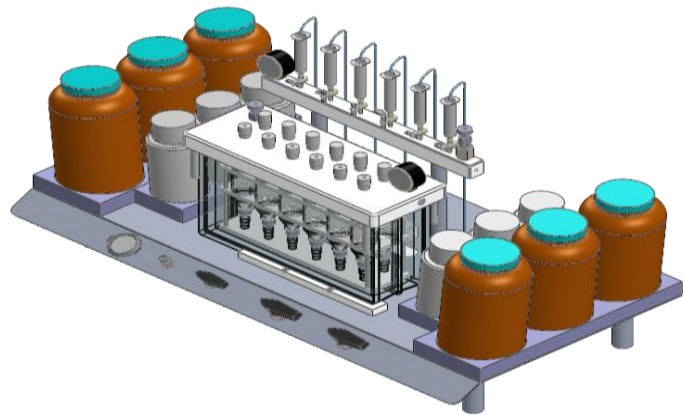
Sample Loading (Stage 1, Aqueous Waste)



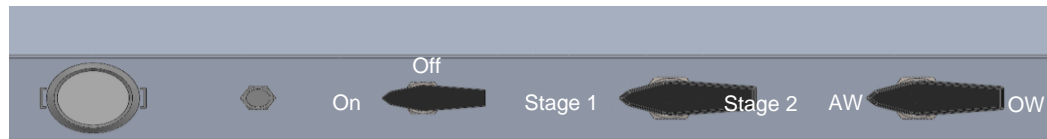
Flow
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Cartridge Drying- Nitrogen/Vacuum



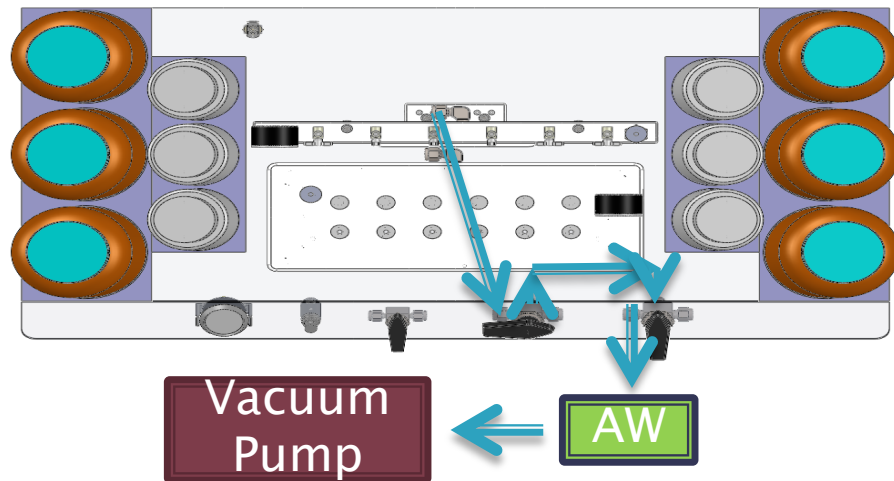
Flow
Path



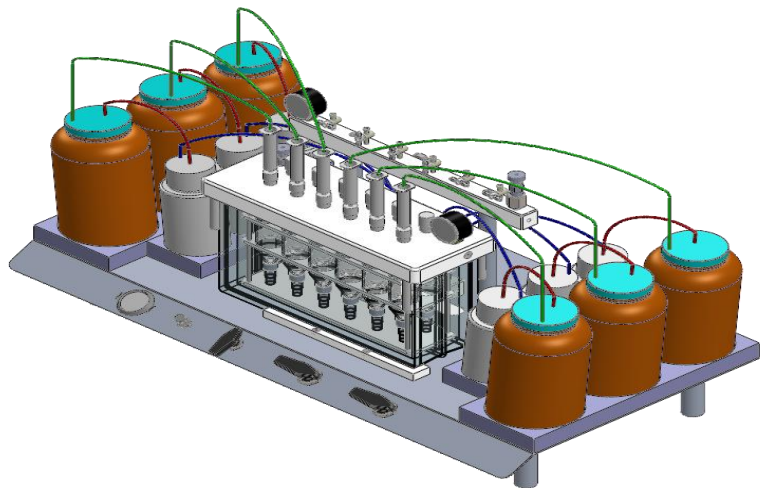
Nitrogen
Valve

Stage
1/2
Valve

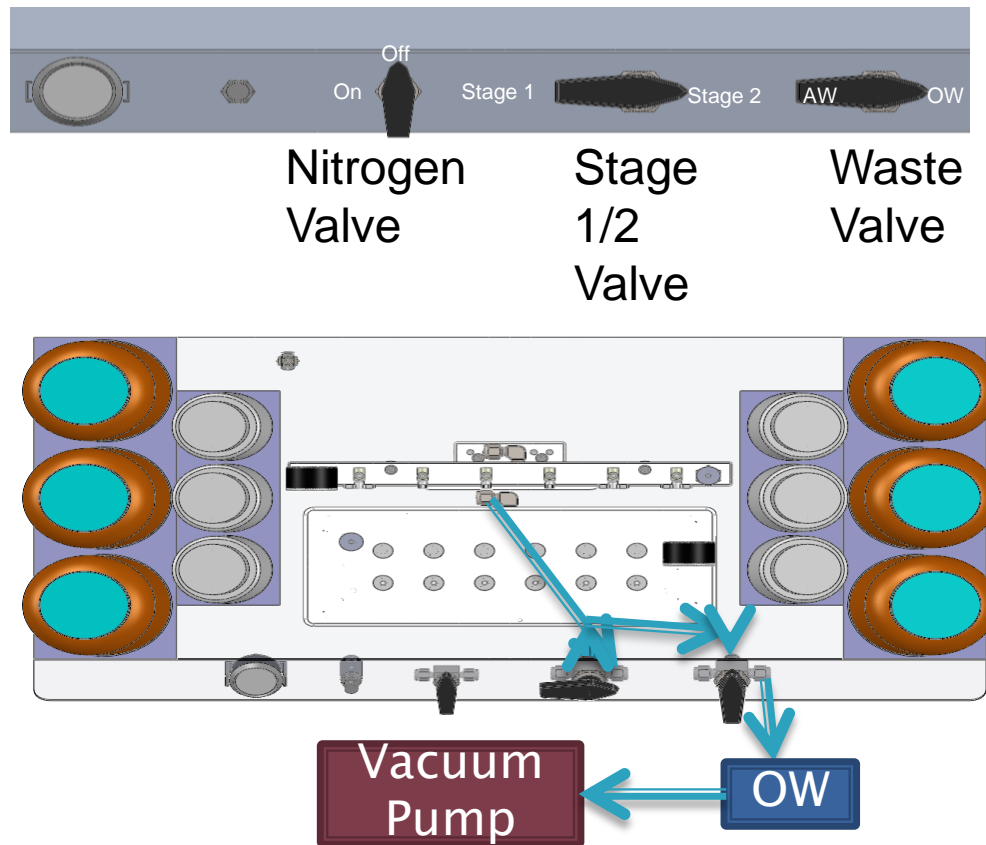
Waste
Valve



Sample Elution (Stage 2)




Flow
Path



Attributes EZSpe (1)

- ▶ Simple to Operate No Computer or Electronics
- ▶ Fast Runs 12 Samples in 20 ~ 50 min (depending on sample size)
- ▶ High Throughput Runs 12 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)

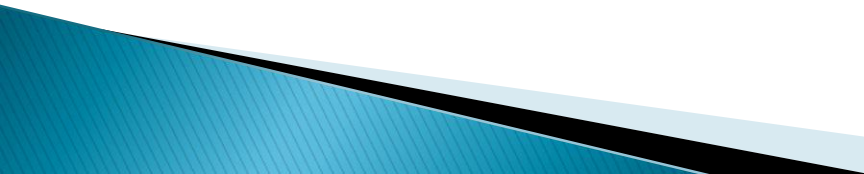
- ▶ Quality Consumables Guaranteed Certified Cartridges
 - ▶ Automated Bottle Rinse
 - ▶ In-line Extract Drying
 - ▶ Reliable No Maintenance Required
 - ▶ Zero Cross-Contamination No Shared Tubing & Fittings
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Procedure (1)

- ▶ Twelve samples (1L water each) are prepared and acidified with 2 mL HCl till pH ~ 2
- ▶ Spike with relevant standards
- ▶ Put sample bottles in place and fill dichloromethane rinse bottles with 35 mL solvent
- ▶ ABN and coconut charcoal cartridges installed in each of the 12 positions.

Procedure (2)

Stage 1:

- ▶ Vacuum is turned on, cartridges are conditioned with dichloromethane, 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 35 mL dichloromethane
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Procedure (3)

Stage 2:

- ▶ Dichloromethane from sample bottles is loaded across the ABN cartridges and collected
- ▶ ABN cartridges eluted with extra 15 mL DCM (Fraction #1)

Procedure (4)

- ▶ **Stage 1**
- ▶ ABN cartridges put back and conditioned with 1% NaOH
- ▶ **Stage 2**
- ▶ Coconut charcoal cartridges eluted with DCM (Fraction # 2)
- ▶ ABN cartridges (now basic) eluted with DCM (Fraction # 2)
- ▶ Dry over sodium sulfate or use in-line cartridges

12 position evaporator 50 mLs



Direct-to-Vial



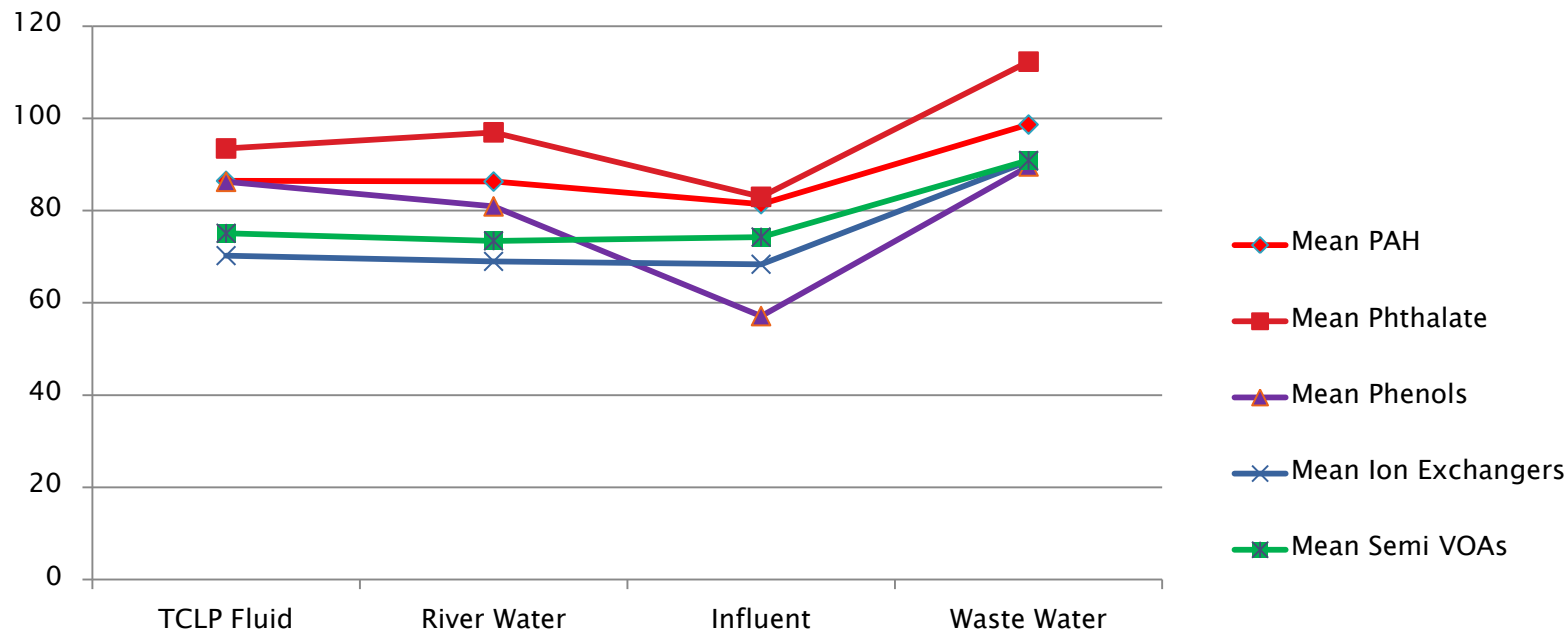
SuperVap Features

- ▶ 6 (250mL) and 12 (50mL) position models for extractions.
- ▶ Typically 40-45 oC, 5-7 psi nitrogen flow
- ▶ Dry bath heating element
- ▶ Independent secondary heater for extract nipple (can be disabled)
- ▶ Sensor controlled
- ▶ Savable temperature log capability

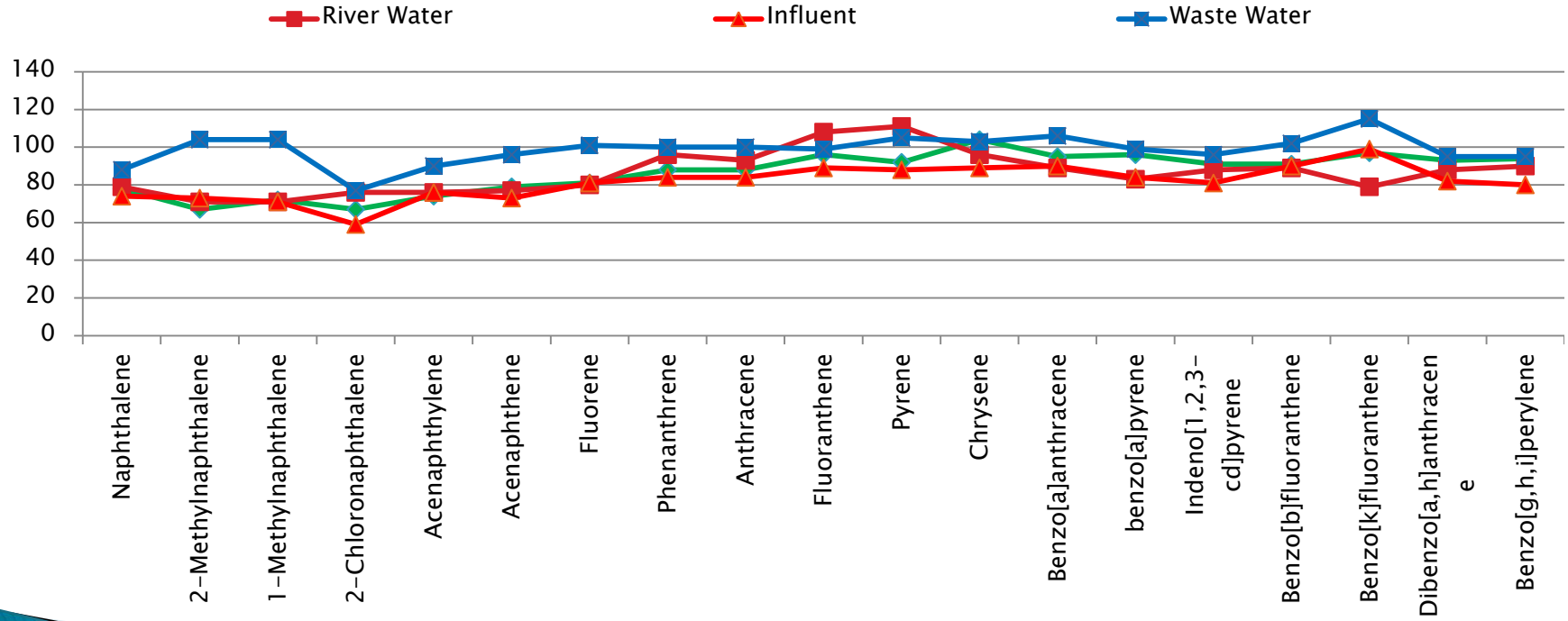
Analysis

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

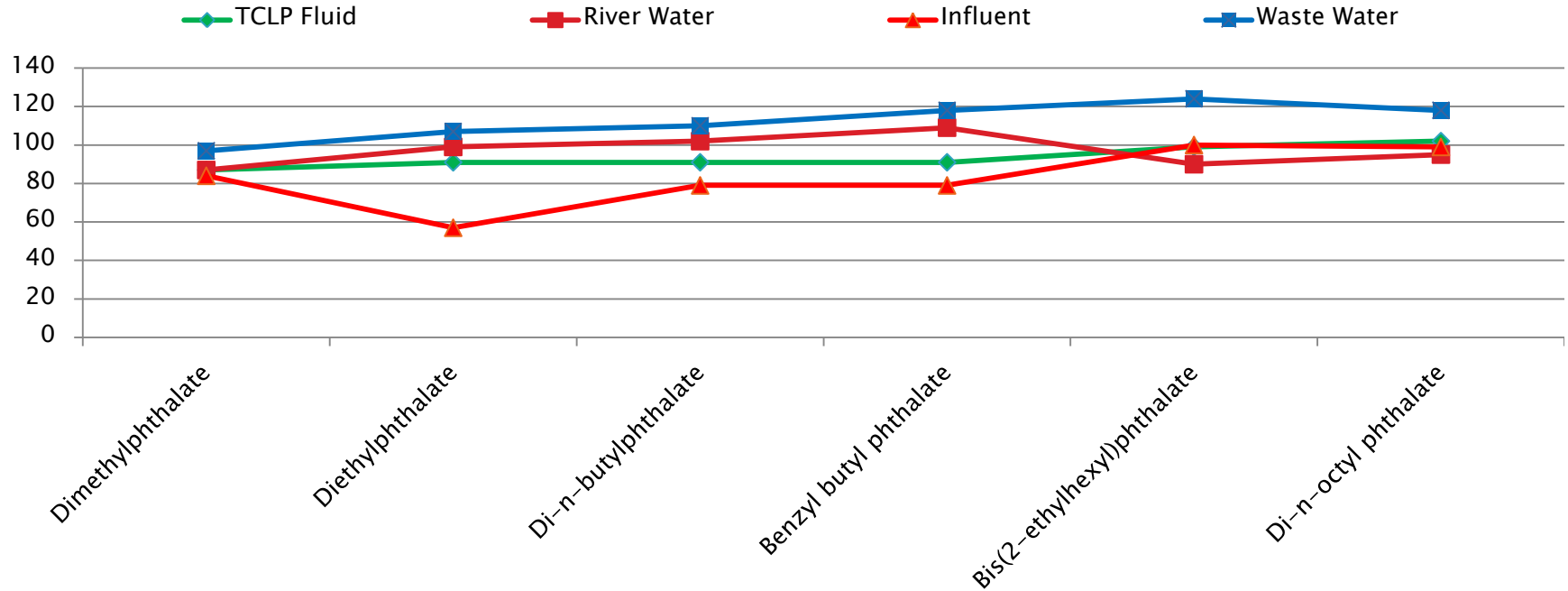
Recoveries by compound class



PAHs

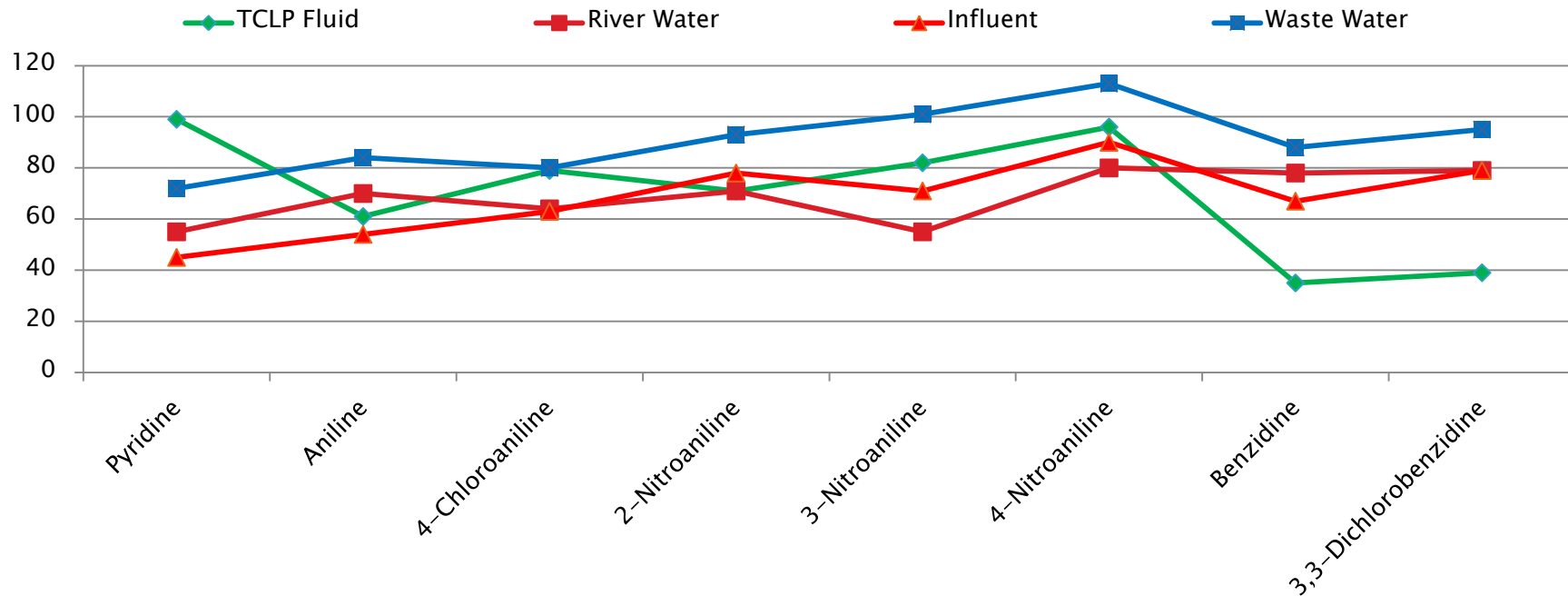


Phthalates





Ion exchangers





Conclusions

- ▶ EZSpe delivers excellent recoveries for 625/8270D Semi-Volatiles
- ▶ Runs 12 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