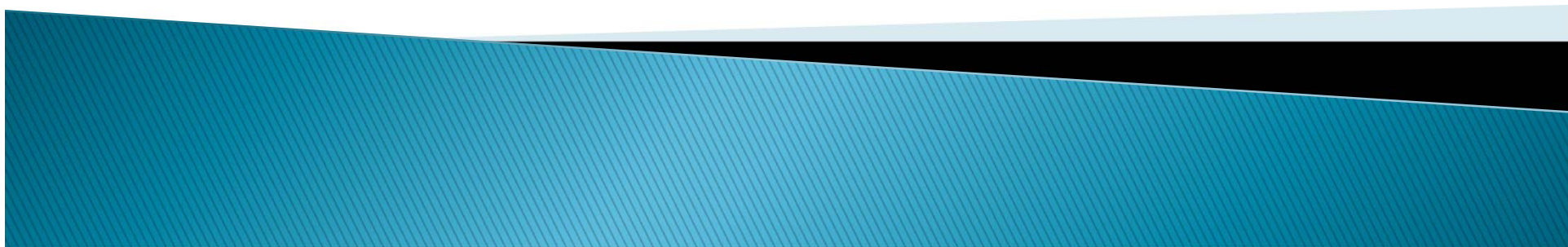


Analysis of Polychlorinated Dibenzop-dioxins, Furans, Biphenyls and Organochlorine Pesticides in Drinking Water with Semi-Automated Solid Phase Extraction

Ruud Addink
Toxic Report, LLC
Watertown MA



Introduction (1)

- ▶ POPs (PCDD/Fs, PCBs) continue to attract interest around the world due to strict regulations in force in many countries (Stockholm Convention).
- ▶ Rapid sample clean up and analysis needed for many laboratories processing samples.
- ▶ Processing times and solvent use are important considerations.
- ▶ In US EPA methods 1613 and 1668



Introduction (2)

- 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



Analysis for PCDD/F/PCBs/OCPs

- 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

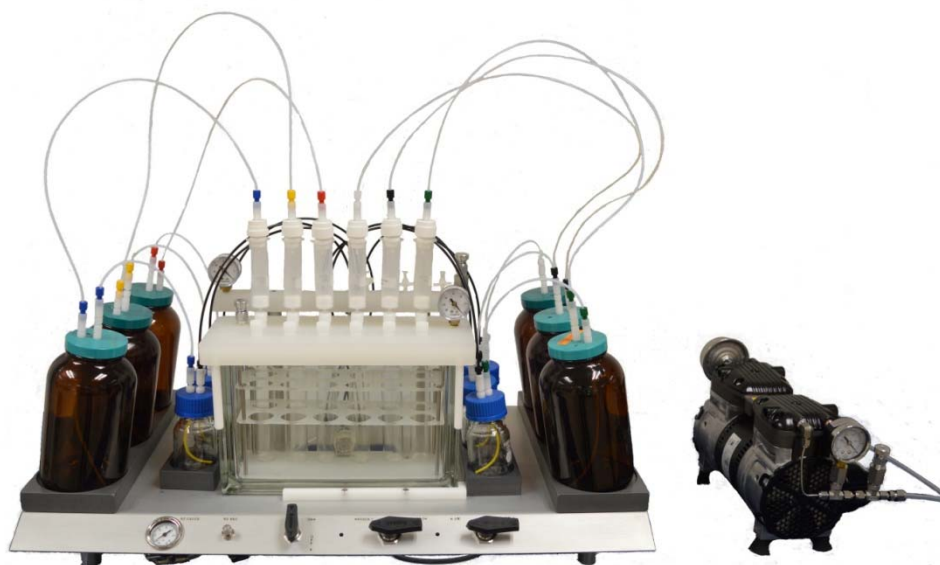


Semi-Automated SPE

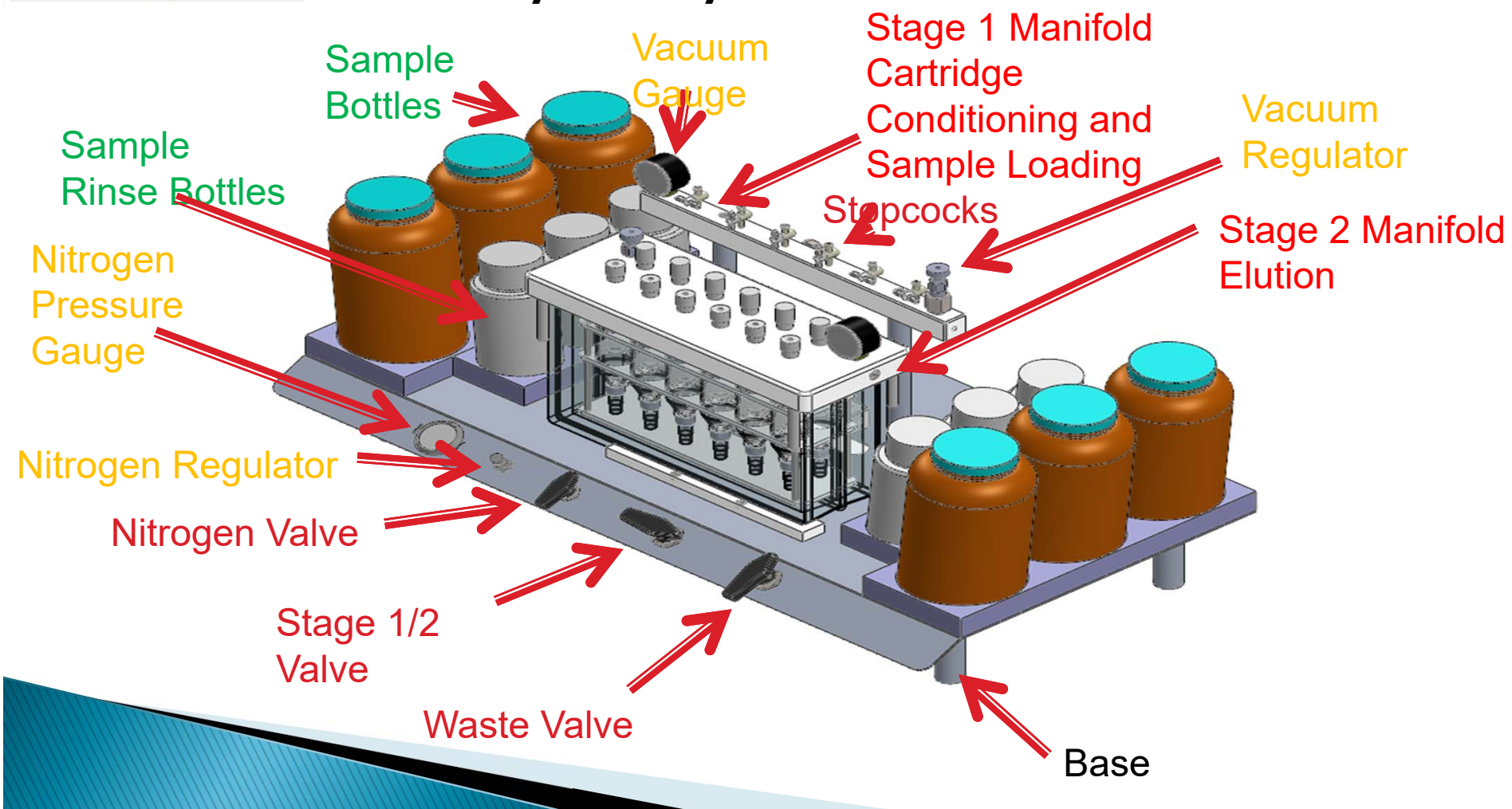
- ▶ Semi-automated SPE done by many labs around the world
- ▶ Cheaper than fully automated systems
- ▶ Important that system is reliable and fast
- ▶ Should be able to use variety of cartridges



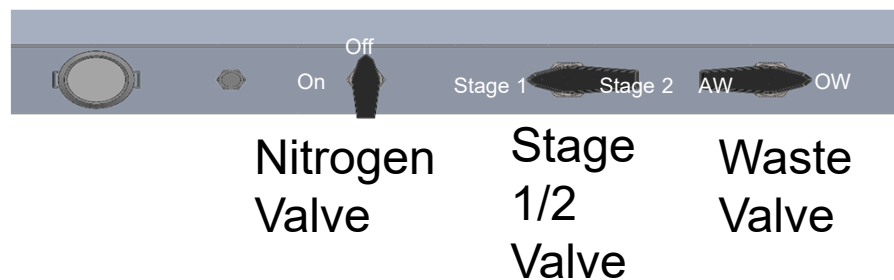
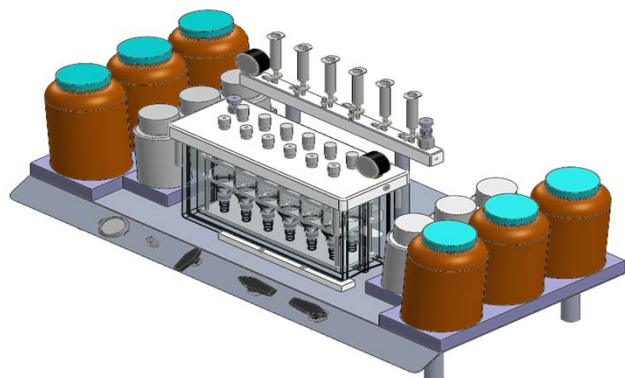
Semi-Automated FMS System (EZSpe®)



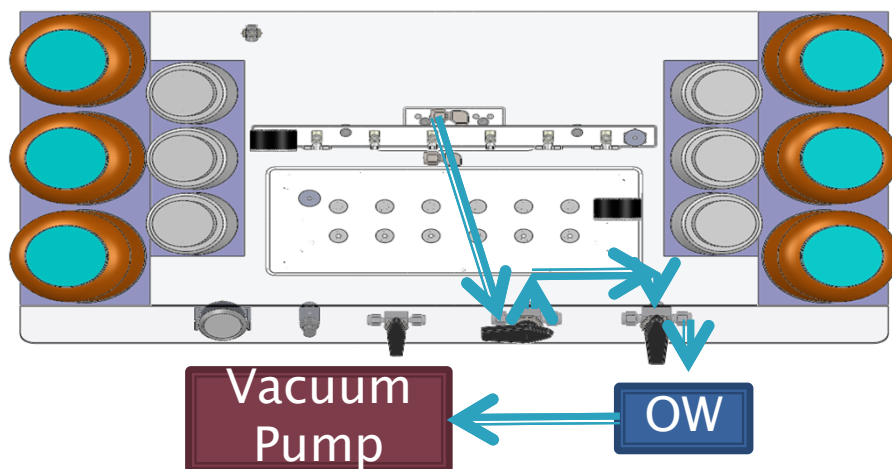
System Layout



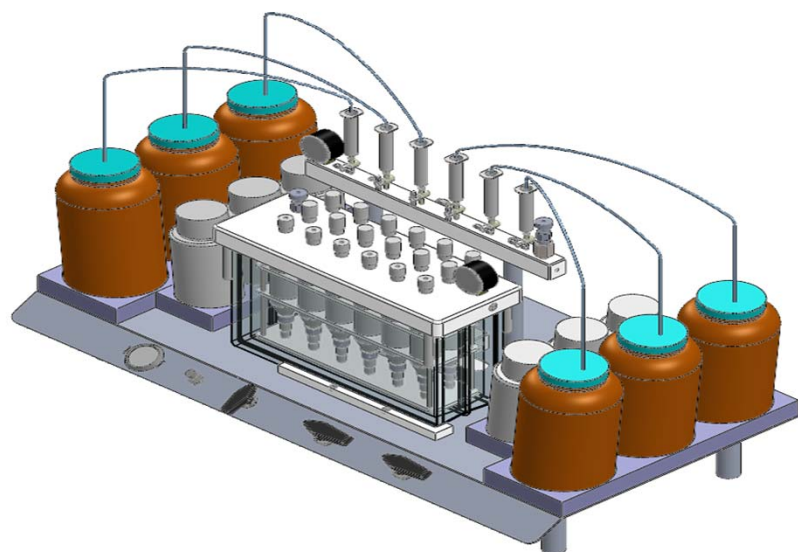
Cartridge Conditioning (Stage 1, Organic Waste)



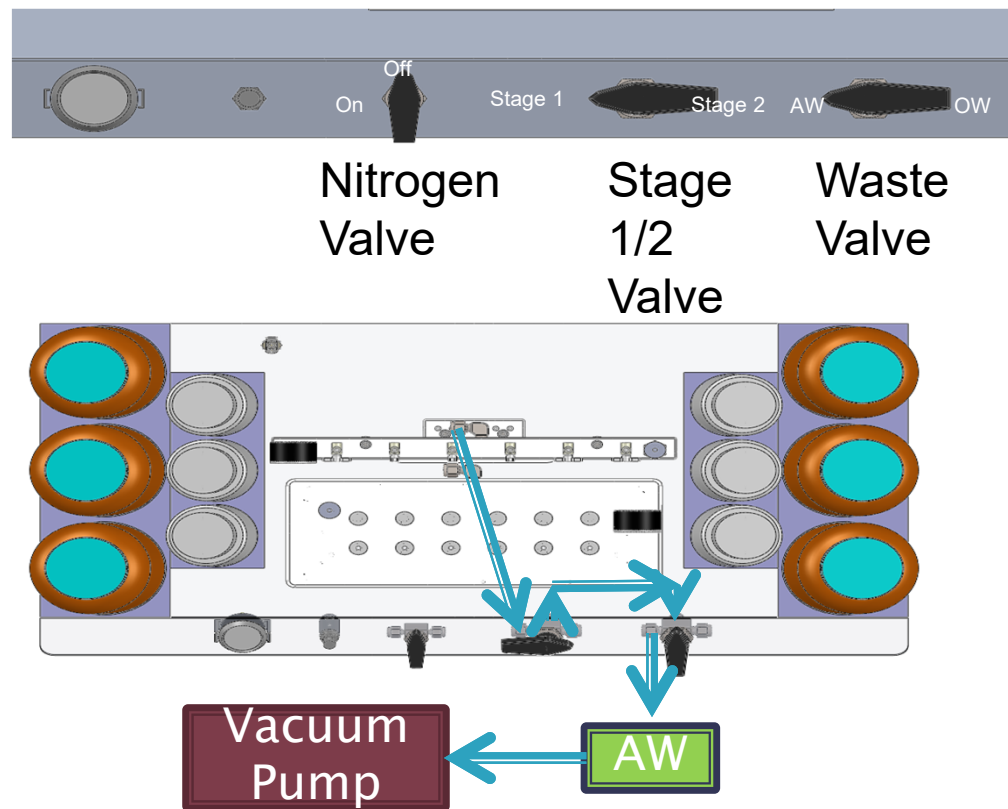
Flow
Path



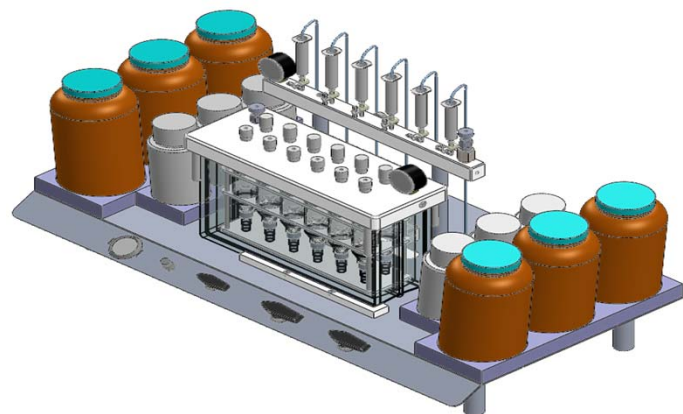
Sample Loading (Stage 1, Aqueous Waste)



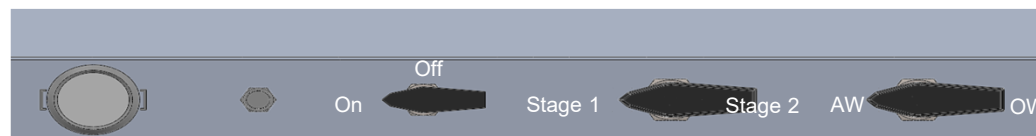
Flow
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Cartridge Drying- Nitrogen/Vacuum



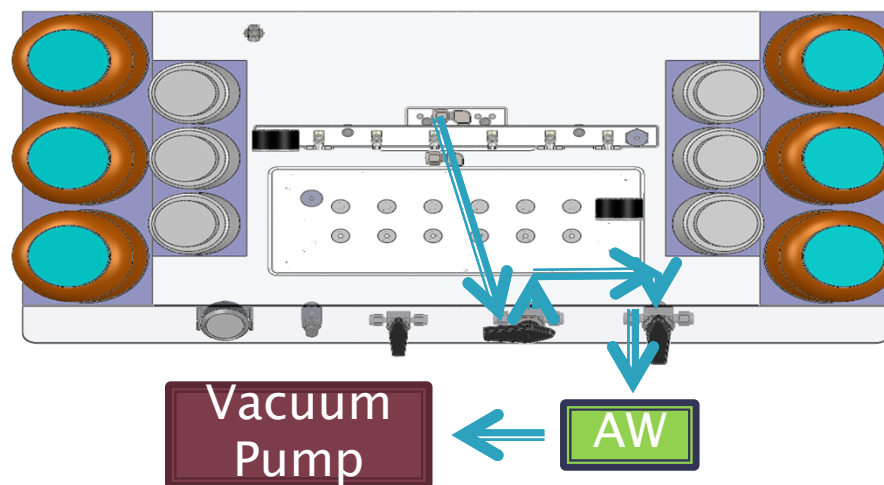
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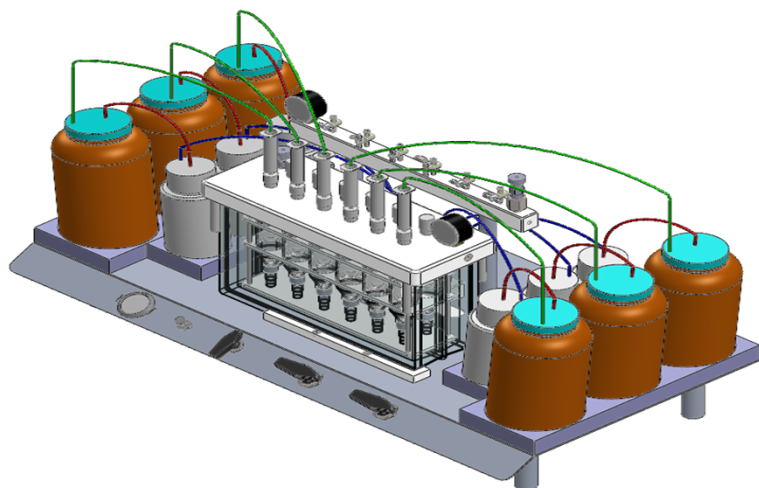
Nitrogen
Valve

Stage
1/2
Valve

Waste
Valve



Sample Elution (Stage 2)



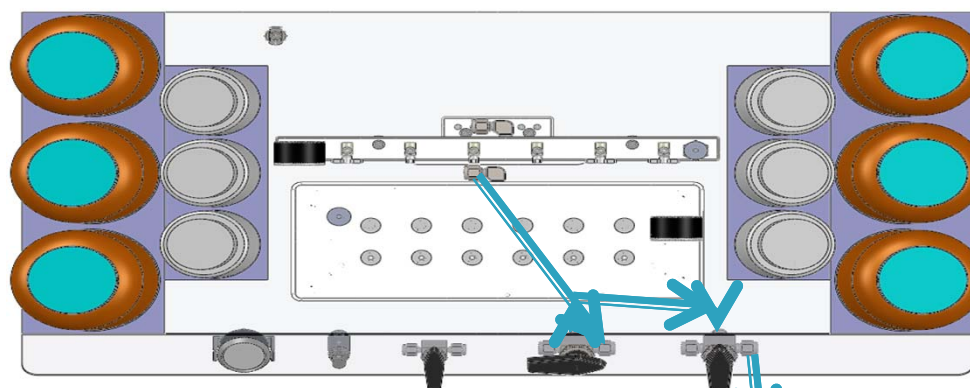
Flow
Path



Nitrogen
Valve

Stage
1/2
Valve

Waste
Valve



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 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 \sim 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 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 25 mL dichloromethane



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



12 position evaporator 50 mLs



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.

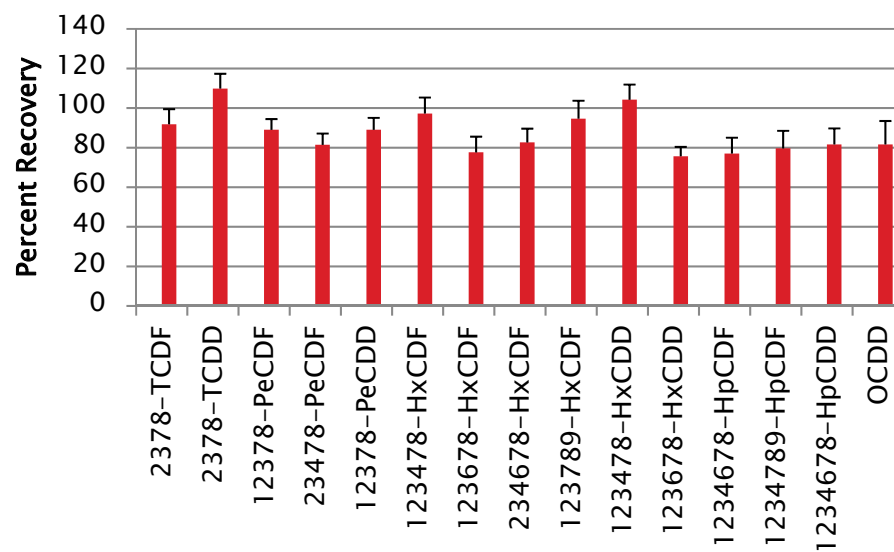


Analysis

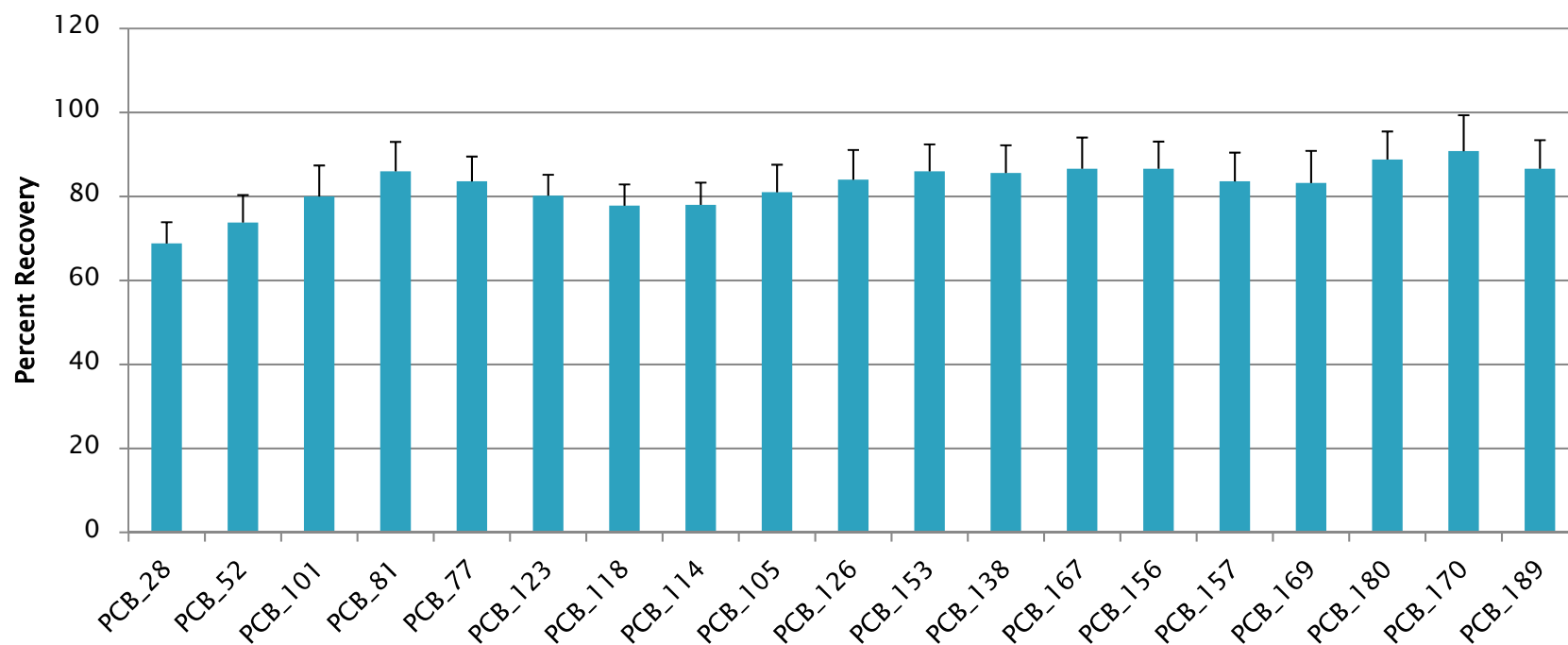
- ▶ Samples reduced to 1 mL under nitrogen flow
- ▶ OCPs analyzed in 1 mL DCM with GC/ECD
- ▶ PCDD/Fs and PCBs further reduced to 10 uL and analyzed with high resolution GC/MS (DFS)



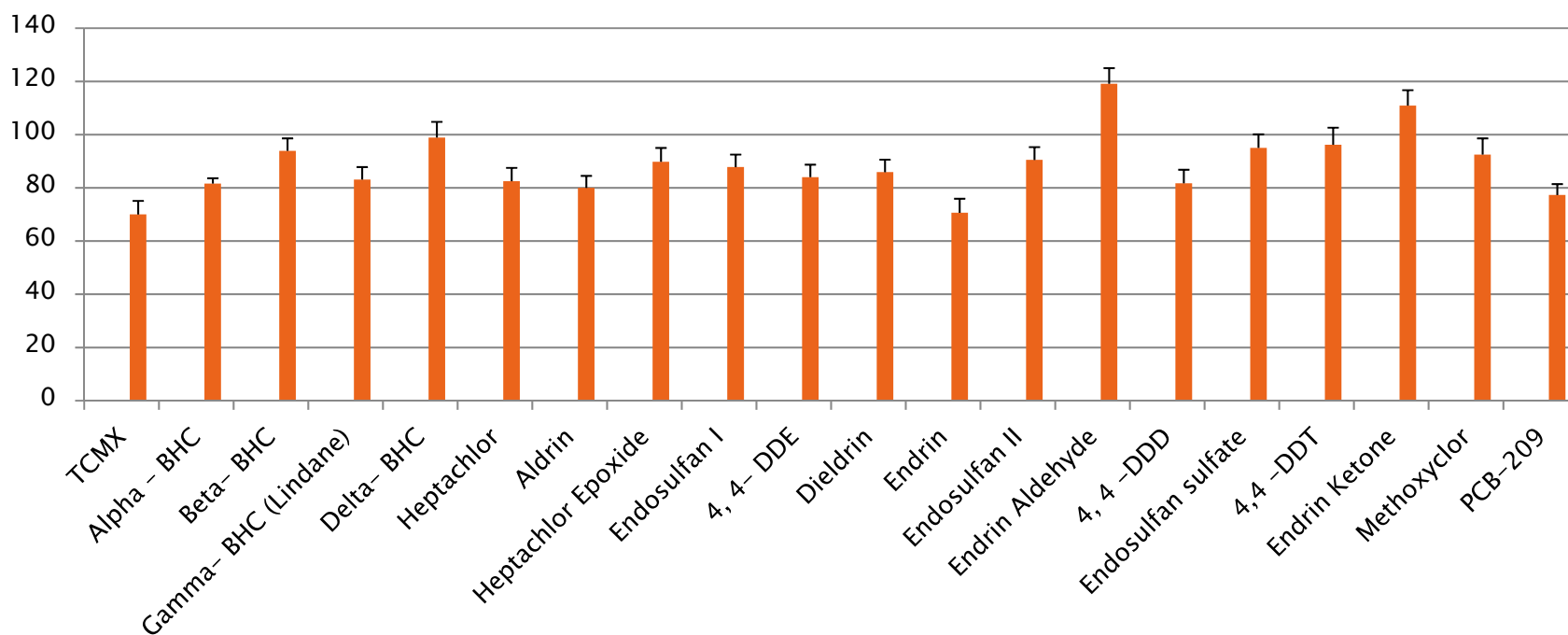
EZSpe data for PCDD/Fs (Drinking Water)



EZSpe data for PCBs (Drinking Water)



EZSpe data for OCPs (Drinking Water)



Conclusions

- ▶ EZSpe delivers excellent recoveries for PCDD/Fs, PCBs and OCPs
- ▶ Runs 6 samples in parallel
- ▶ Gets data in under 4h
- ▶ No maintenance required
- ▶ No separate water removal step needed (in-line drying)
- ▶ Other applications are beverages, milk and serum



Questions

Contact us at:

raddink@fms-inc.com (Ruud Addink)

thall@fms-inc.com (Tom Hall)

nkaurin@fms-inc.com (Nem Kaurin)

