Toxic Reports™

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

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# 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

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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 |

# Comparison of LLE/CLE vs SPE Methods (2)

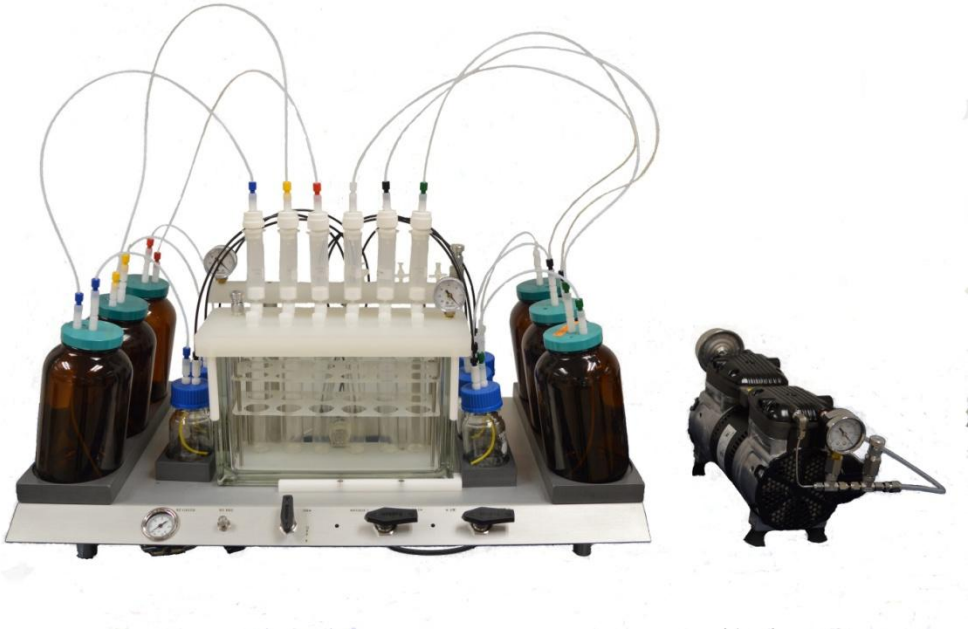
| LLE/CLE | Semi-Automated SPE |
| --- | --- |
| No Separation of waste | Separates Aqueous and Organic Waste |
| More volume to evaporate | <60mls solvent to evaporate |
| Massive solvent emission | 6 times less solvent emission |
| CLE uses a lot of solvent | Easily Capture Solvent |
| Requires lots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

# Reduced Solvent Usage

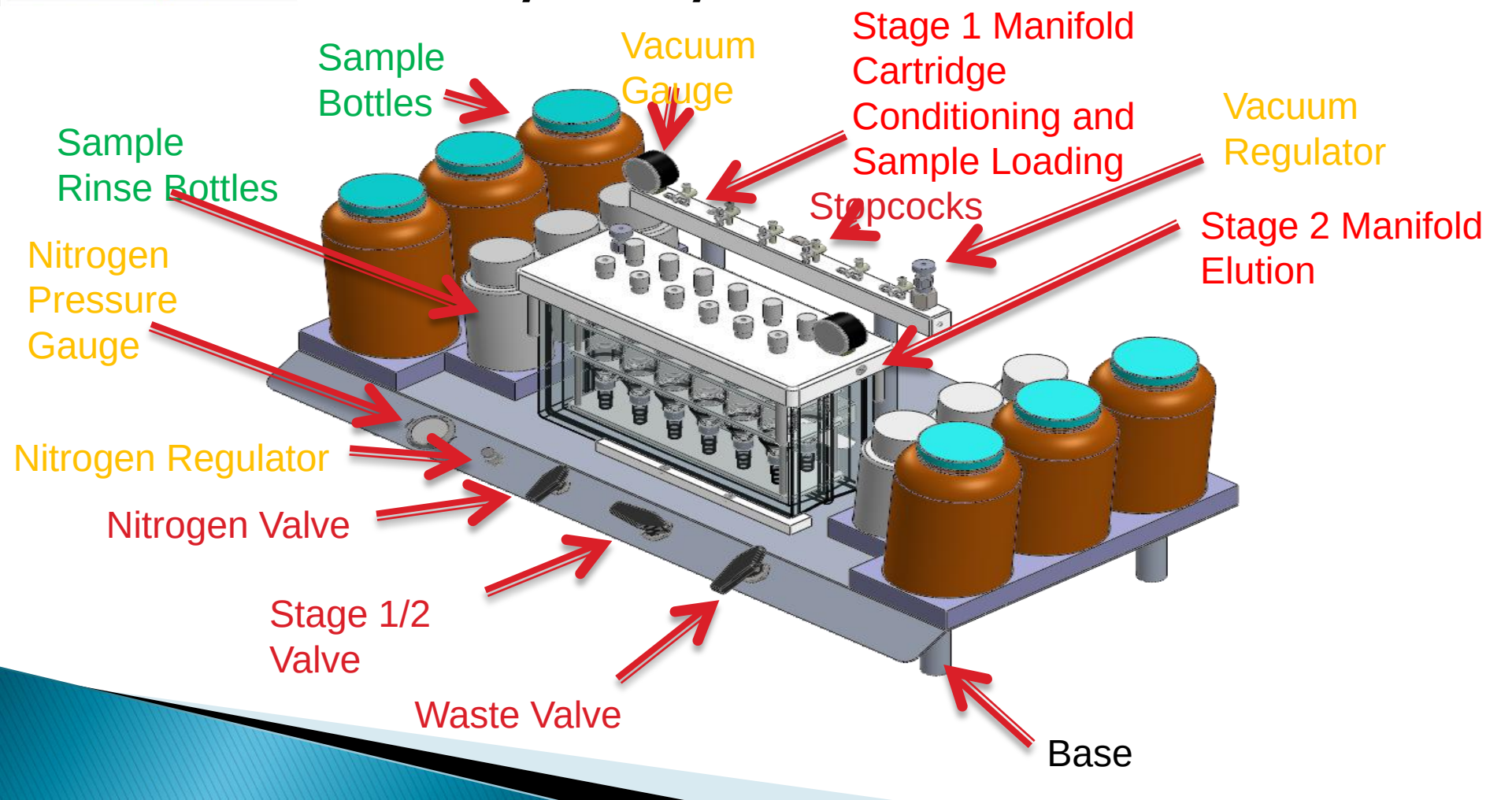

# Semi-Automated SPE

- 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

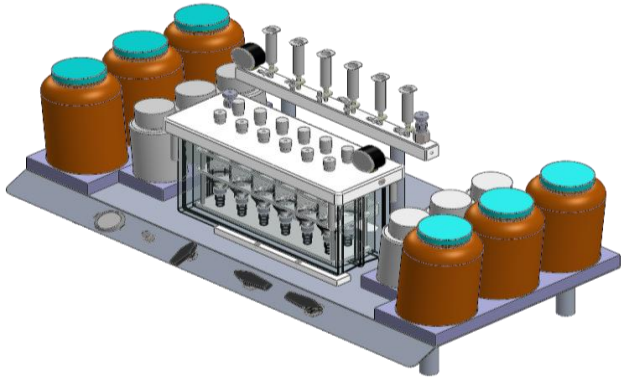
# Semi-Automated FMS System (EZSpe®)



# System Layout



# Cartridge Conditioning (Stage 1, Organic Waste)

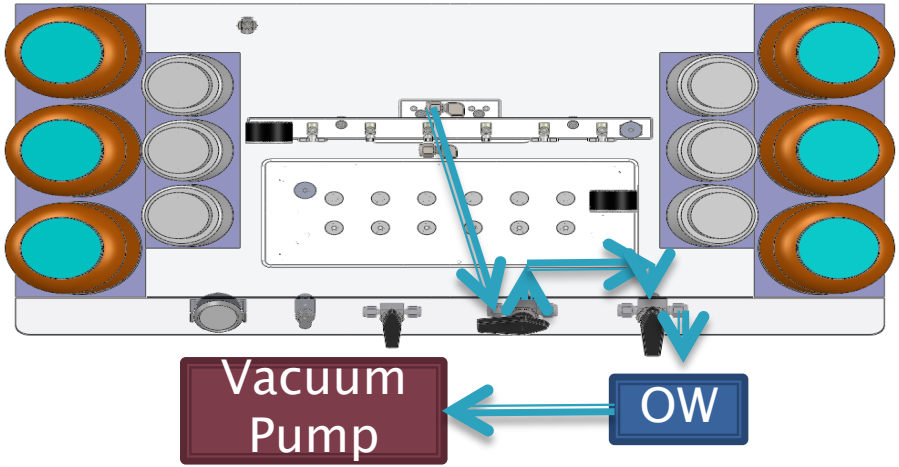



Nitrogen Valve

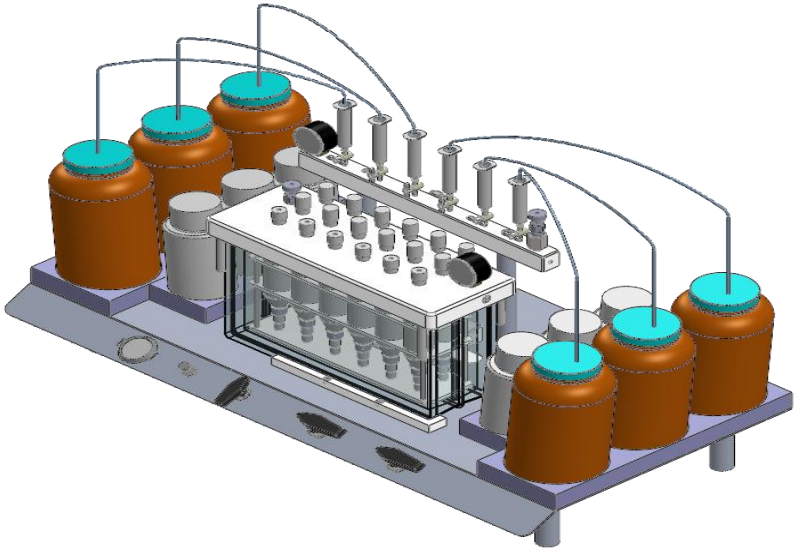
Stage 1/2 Valve

Waste Valve

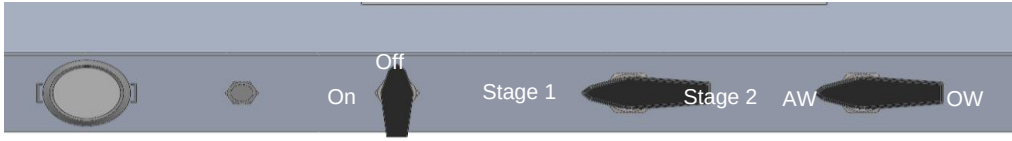
Flow Path



# Sample Loading (Stage 1, Aqueous Waste)



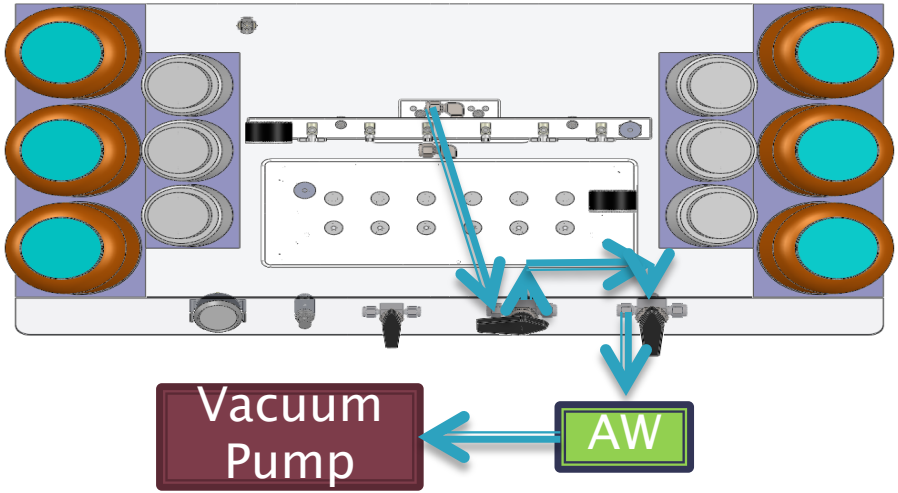
Flow Path



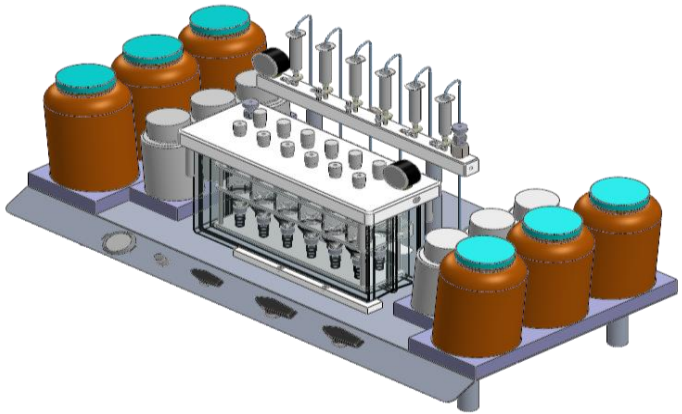

Nitrogen Valve

Stage 1/2 Valve

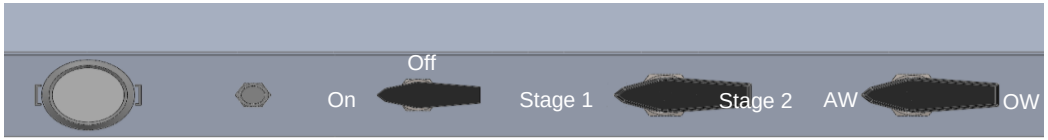
Waste Valve



# Cartridge Drying- Nitrogen/Vacuum



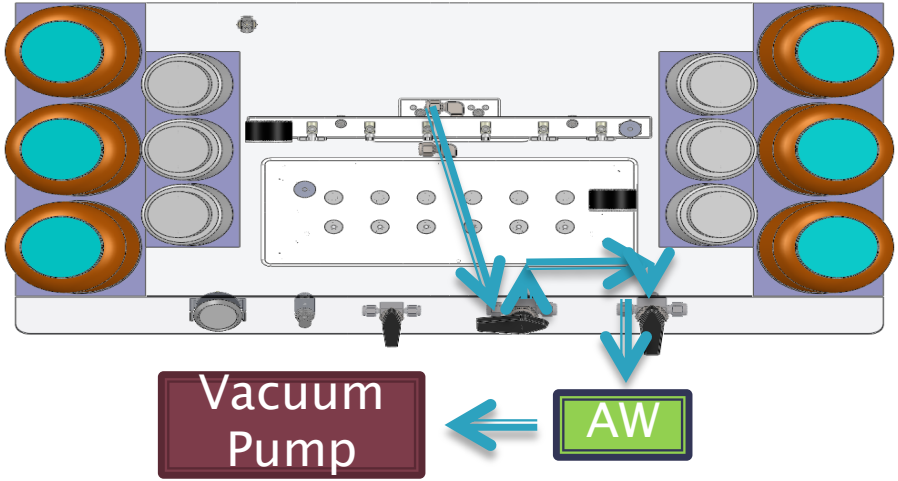
Flow Path



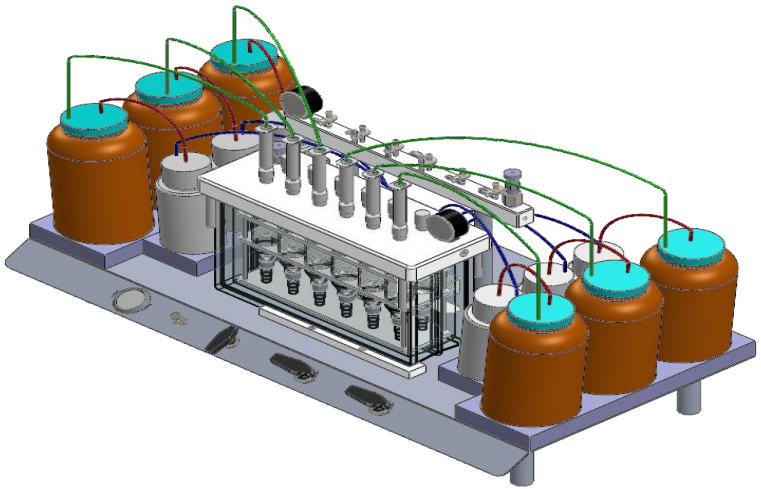

Nitrogen Valve

Stage 1/2 Valve

Waste Valve



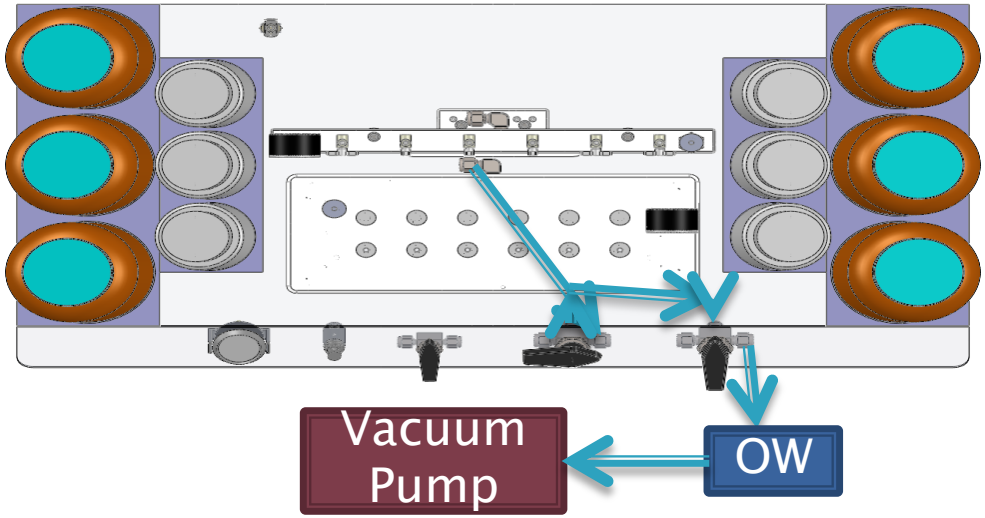
# Sample Elution (Stage 2 )




Nitrogen Valve

Stage 1/2 Valve

Waste Valve




Flow Path

# 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 ~ 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

# 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

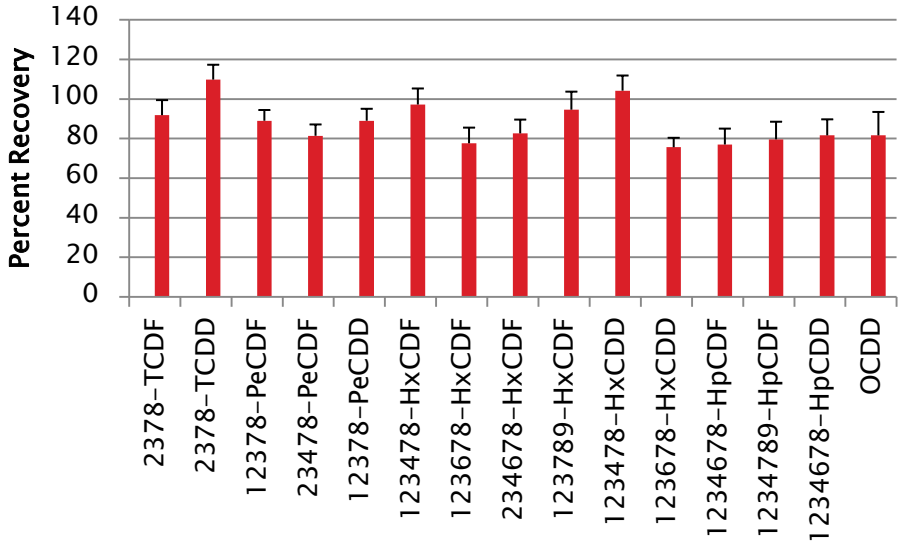
# SuperVap Features

- 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.

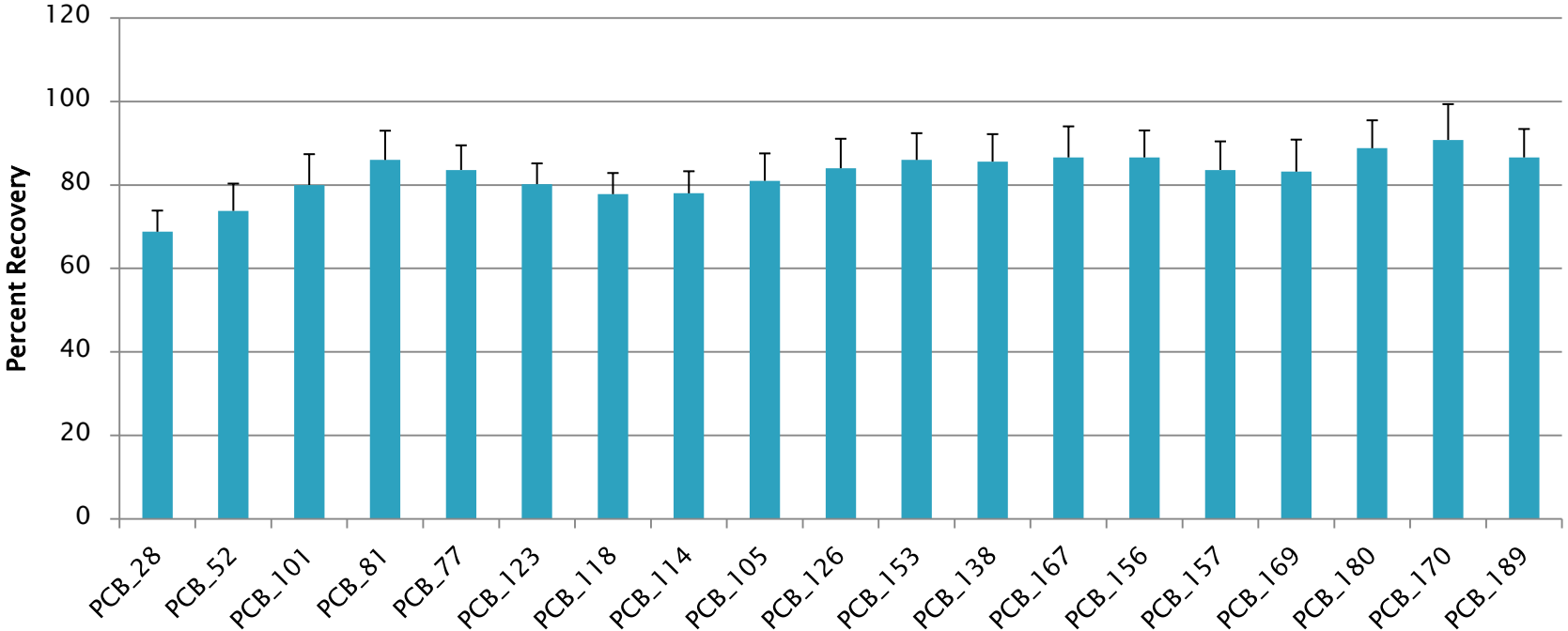
# Analysis

- 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)

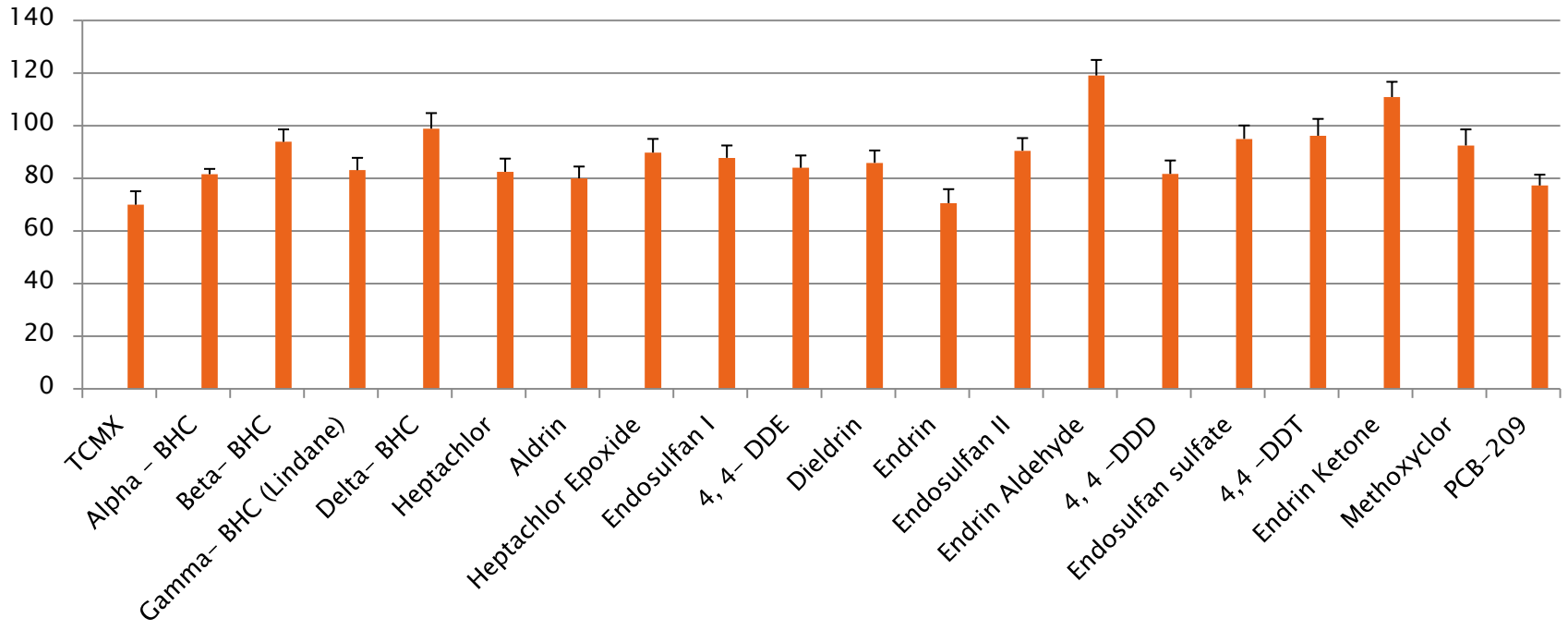
# EZSpe data for PCDD/Fs (Drinking Water)



# 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