High Throughput Solid Phase Extraction, Cleanup and Concentration of Organochlorine Pesticides in Waste Water Using EPA Method 608.3

Ruud Addink and Tom Hall
Fluid Management Systems
Billerica, MA

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 have been added to Stockholm Convention (2009-2015)
- EPA method 608.3 - Waste Water

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

Analysis of 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. Semi-Automated 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. Semi-Automated 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 power	Easily Capture Solvent
Requires lots of solvent for cleaning	Lower solvent costs
	Lower Disposal Costs

Reduced Solvent Usage

Objective for Semi Automation

- Use as many features from Automated systems and implement them into a Semi automated platform
- Develop as many SPE procedures for the testing lab using a single extraction platform.
- Minimize manual steps to lessen error and maximize limited man hours

Goal (1)

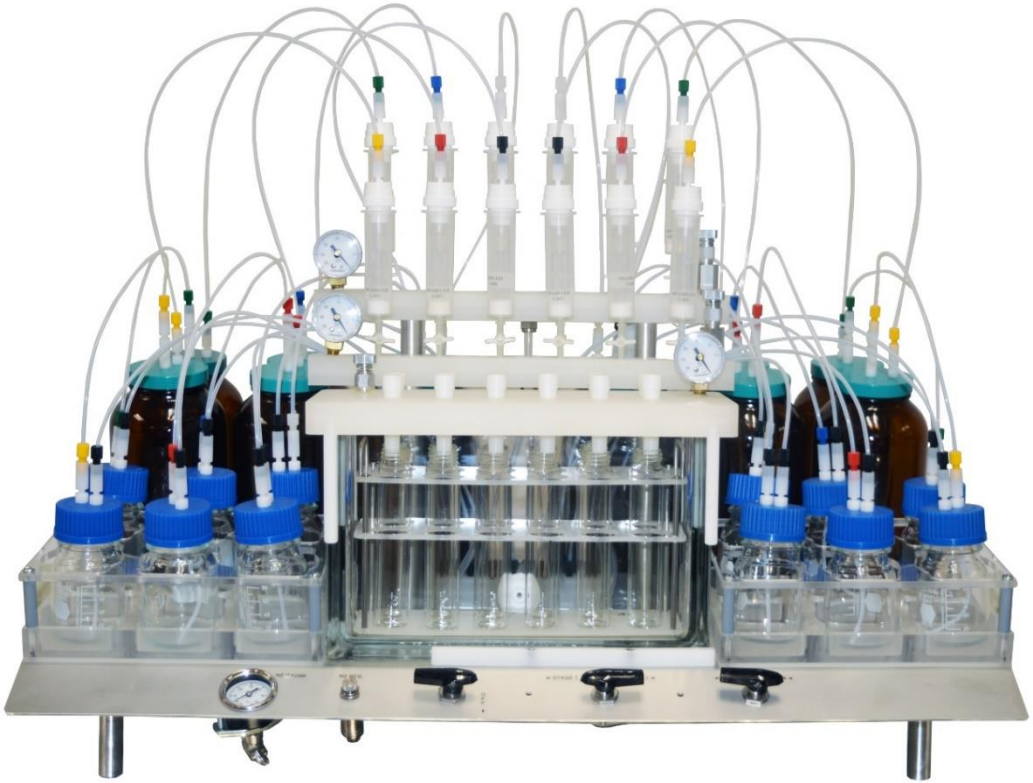
- **Easy to Operate**
  - No Computers or Electronics to fail or maintain
- **Semi - Automated**
  - Hyphenates the entire Solid Phase Extraction Process – Extraction, Bottle Rinse, Inline Drying and Optional Direct to GC Vial Concentration
- **Fast**
  - The fastest sample processing available for SPE
  - Run up to twelve samples simultaneously
  - Vacuum for fast loading of large volume samples
  - Unattended Sample loading walkaway time
- **Closed system**
  - Eliminate potential outside contamination

Goal (2)

- **Efficient**
  - Uses all SPE cartridge sizes
  - Dedicated manifold for cartridge conditioning and sample loading
  - Dedicated manifold for extraction and extracts
  - Separates Organic from Aqueous waste
  - Vacuum cartridge drying, Nitrogen cartridge drying or combined
  - Automated Bottle Rinse and Elution
  - Inline Extract Drying
  - Small number of components to clean

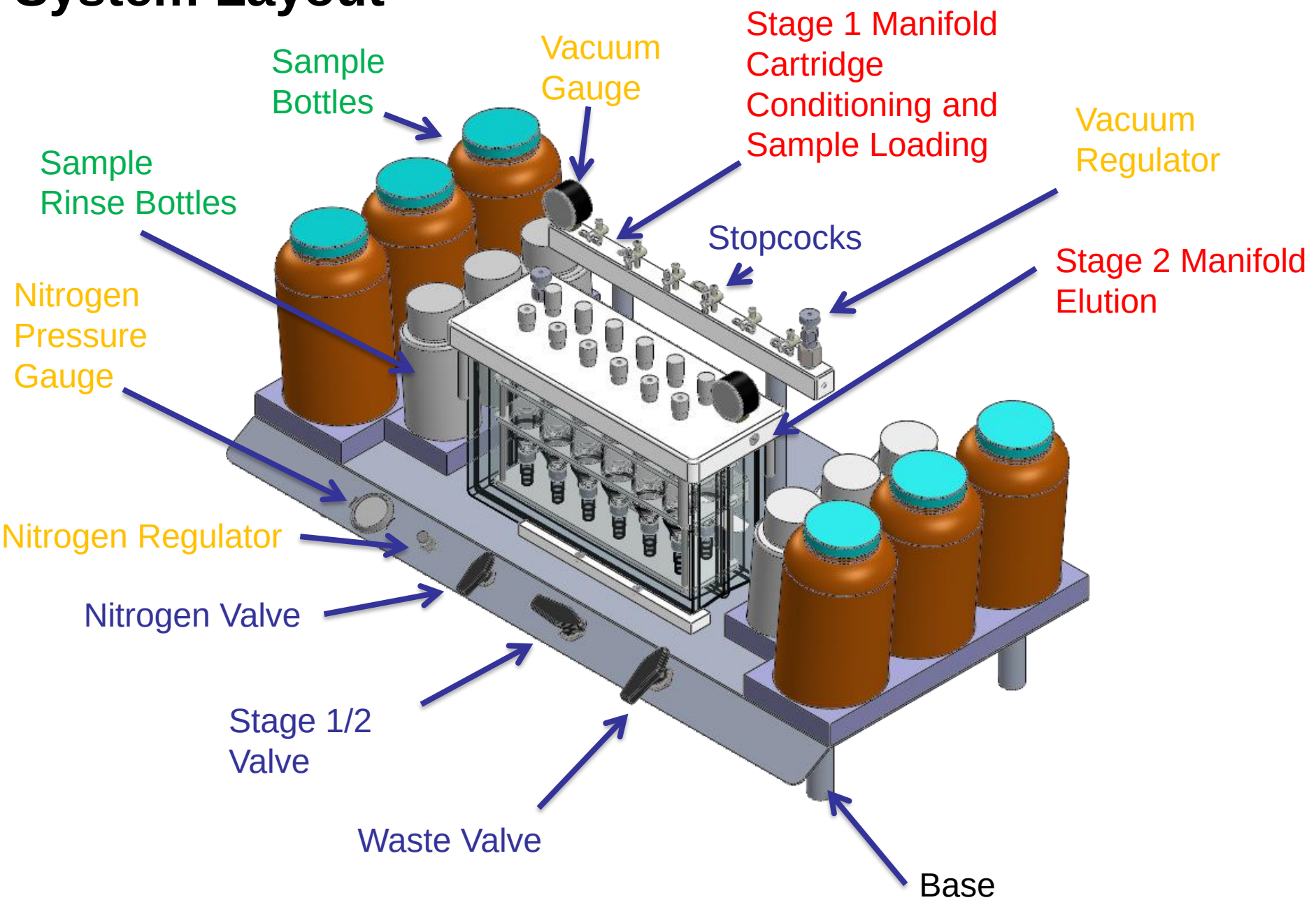
FMS
Fluid Management Systems

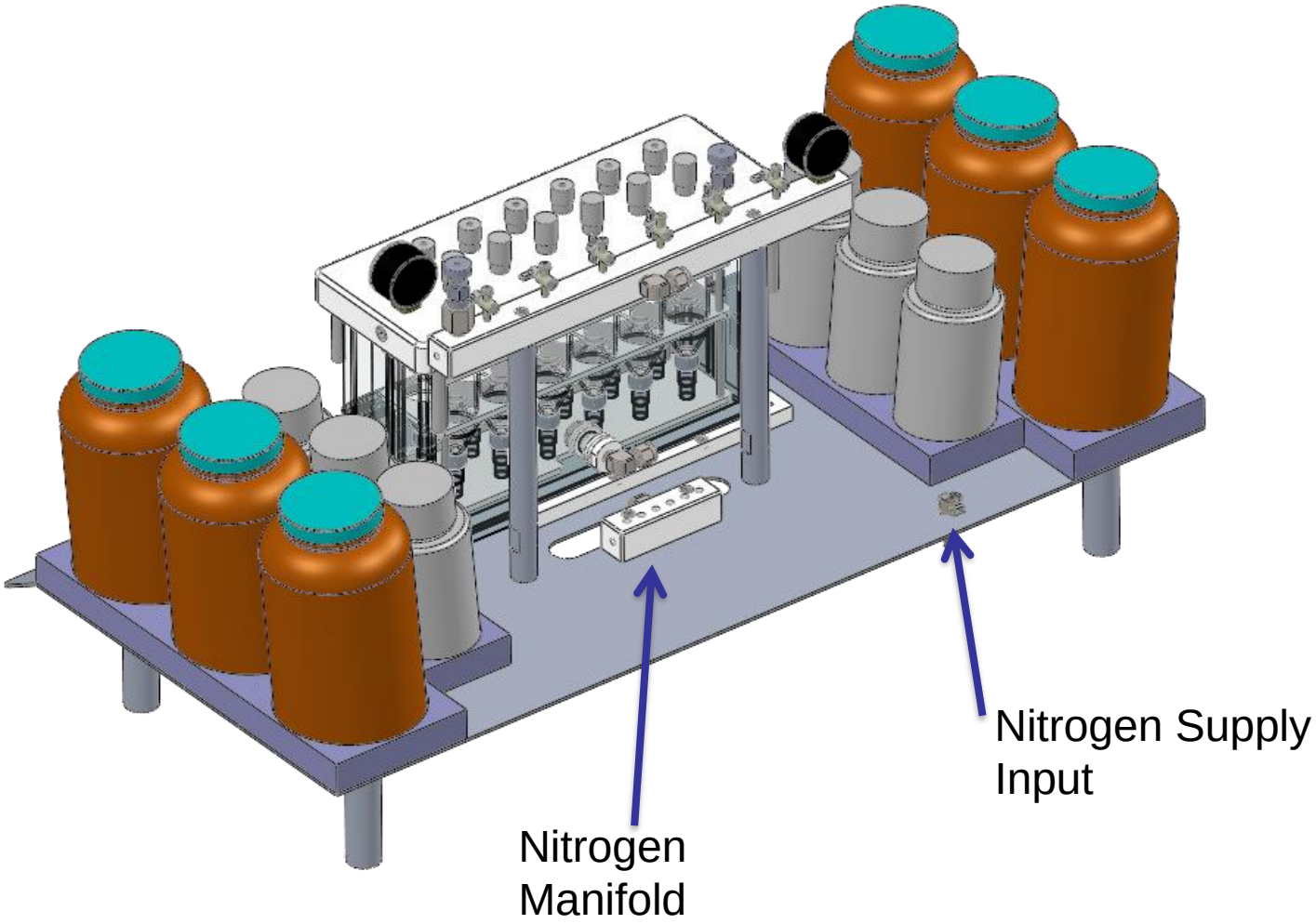
EZSpe®



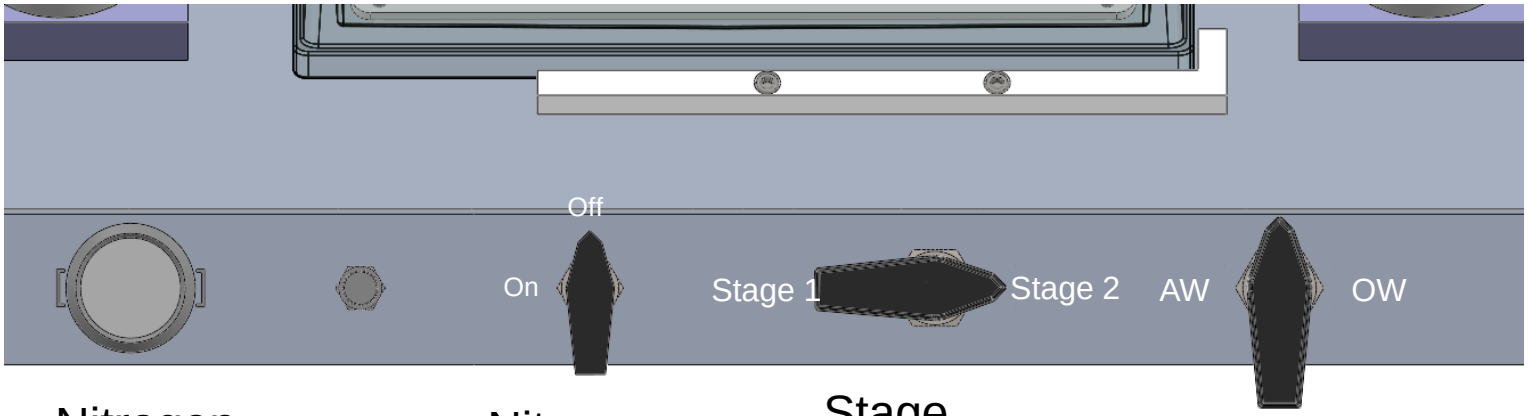
EZSpe 12 samples

System Layout




Nitrogen Supply Input
Nitrogen Manifold

Control Valve Layout



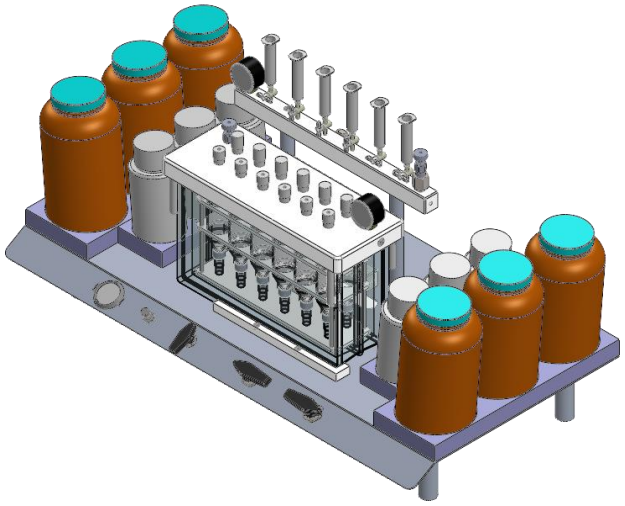
Nitrogen Gauge

Nitrogen Valve

Stage 1/2 Valve

Waste Valve

Cartridge Conditioning (Stage 1, Organic Waste)

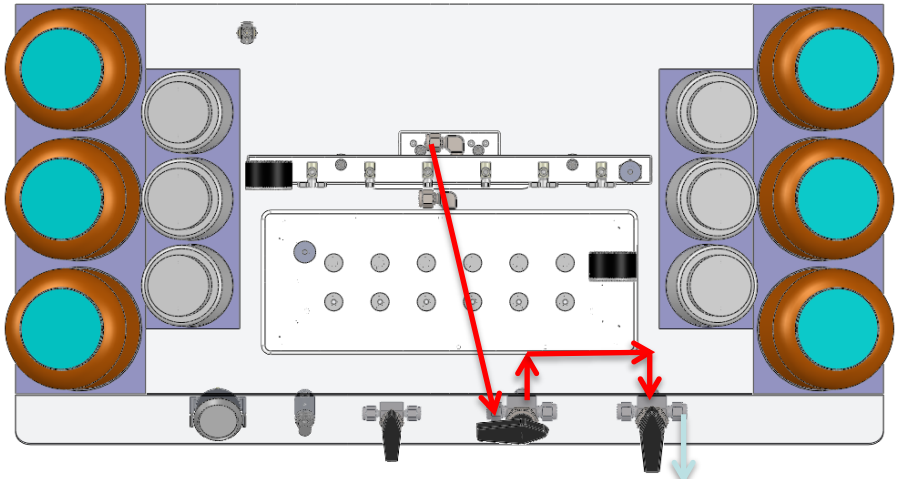


Nitrogen Valve

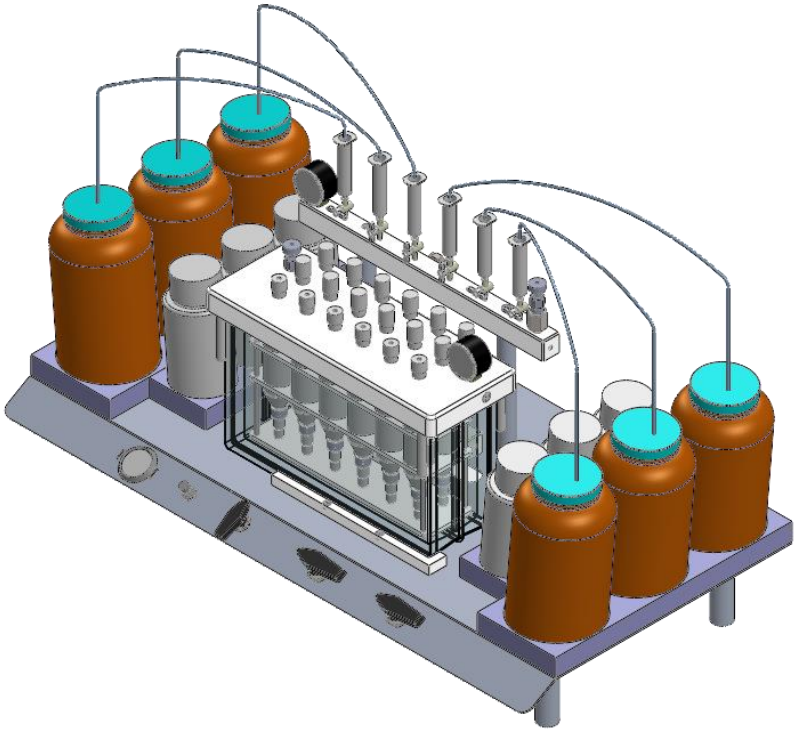
Stage 1/2 Valve

Waste Valve

Flow Path



Sample Loading (Stage 1, Aqueous Waste)

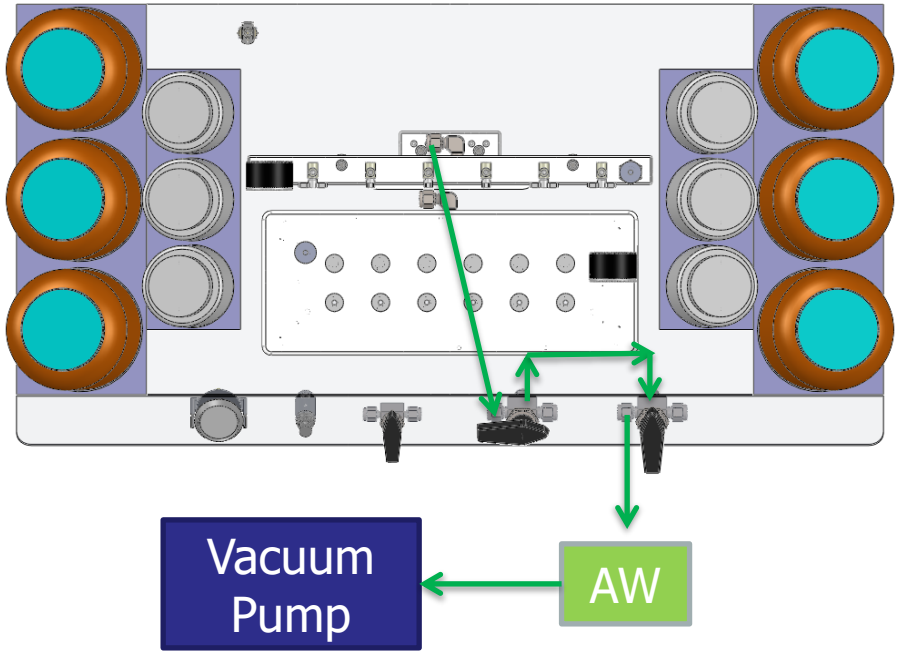


Flow Path

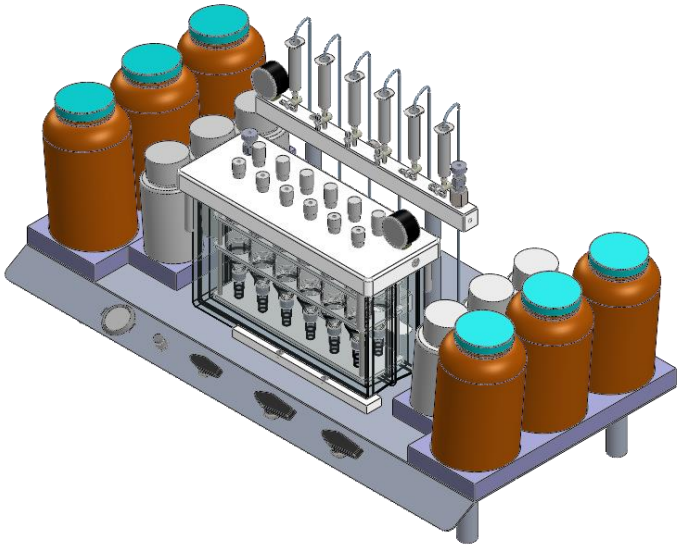
Nitrogen Valve

Stage 1/2 Valve

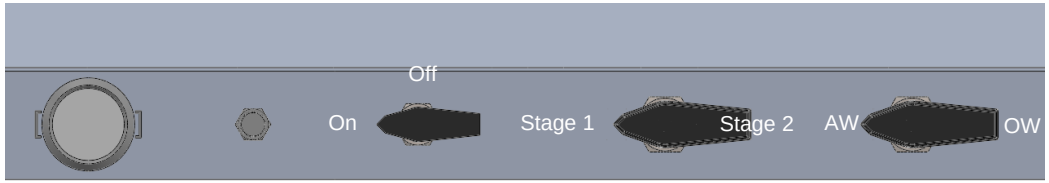
Waste Valve



Cartridge Drying- Nitrogen/Vacuum



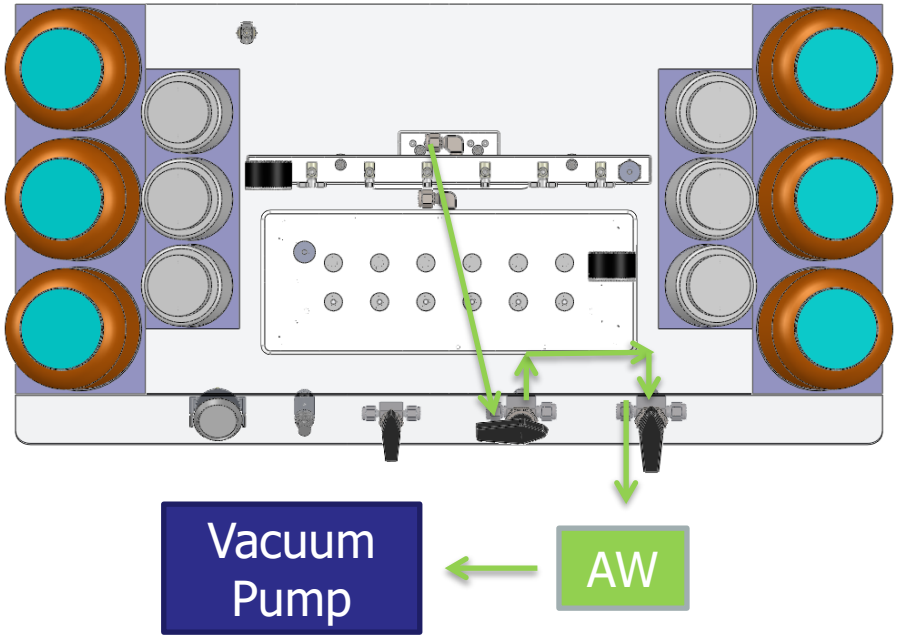
Flow Path



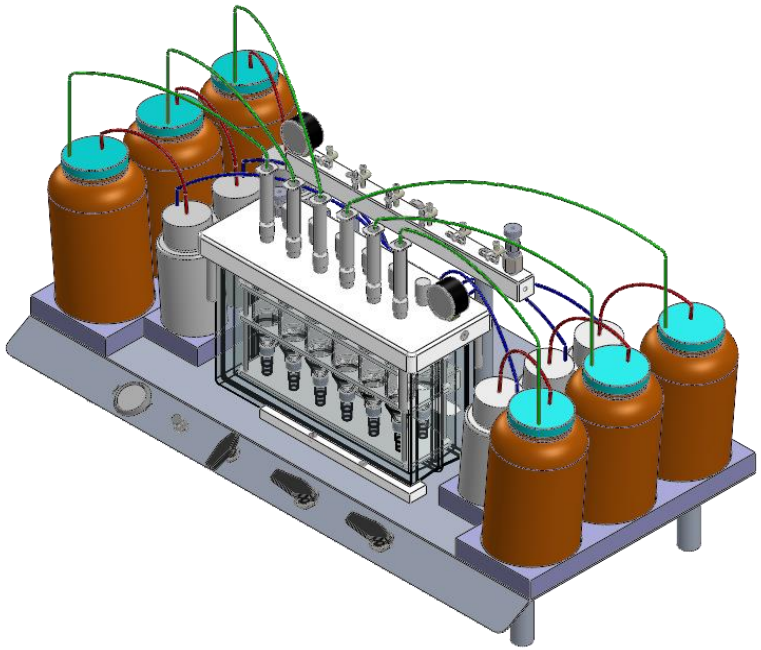
Nitrogen Valve

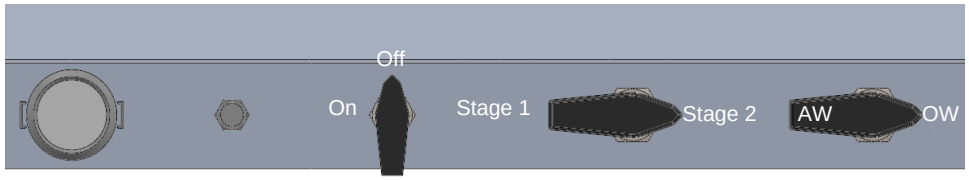
Stage 1/2 Valve

Waste Valve



Sample Elution (Stage 2)

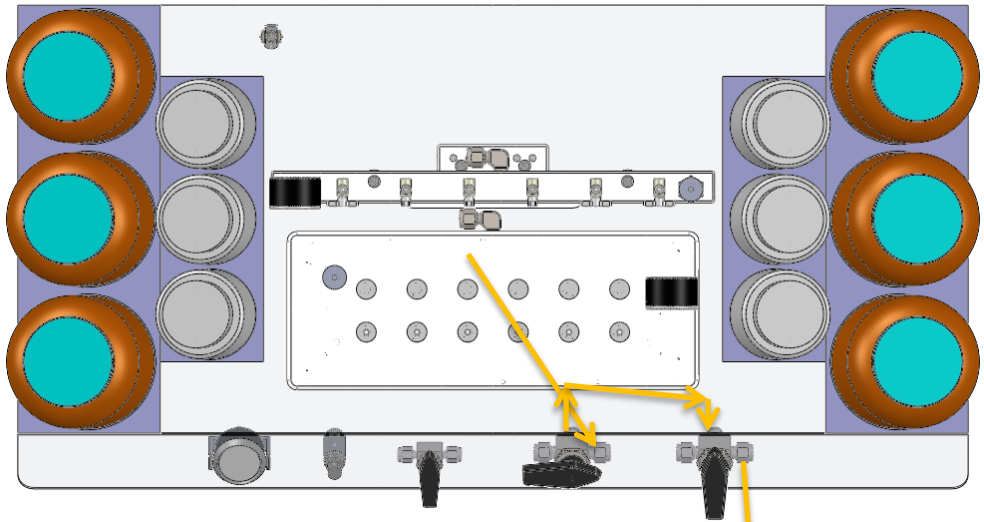




Nitrogen Valve

Stage 1/2 Valve

Waste Valve



Flow Path

Vacuum Pump

OW

Procedure (1)

- 12 samples (1L water each) are prepared and acidified with 6 N HCl till pH < 2
- Spike with relevant standards
- Put sample bottles in place and fill automated rinse bottles with 40 mL 10% acetone/hexane
- C-18 cartridges are installed in each of the 12 positions.

Procedure (2)

Stage 1:

- Vacuum is turned on
- C-18 cartridges conditioned with 10 mL acetone (2 min soak), and 20 mL water
- Samples are loaded across cartridges under vacuum (~ 12 inch Hg)
- C-18 cartridges are dried under vacuum for 10 min and then removed
- Hydromatrix cartridges on top of Florisil cartridges conditioned with 20 mL 10% acetone/hexane

Procedure (3)

Stage 2:

- C-18 cartridges on top of Hydromatrix/Florisil cartridges assembly on top of Stage 2 manifold
- Sample bottles are automatically rinsed from the rinse bottles with 40 mL 10% acetone/hexane
- Sample bottles rinses loaded across all three cartridges and collected for analysis (Direct-to-Vial)

FMS
Fluid Management Systems

Direct to Vial Concentration

SuperVap Concentrator

- 12 position
- 50mL vessel

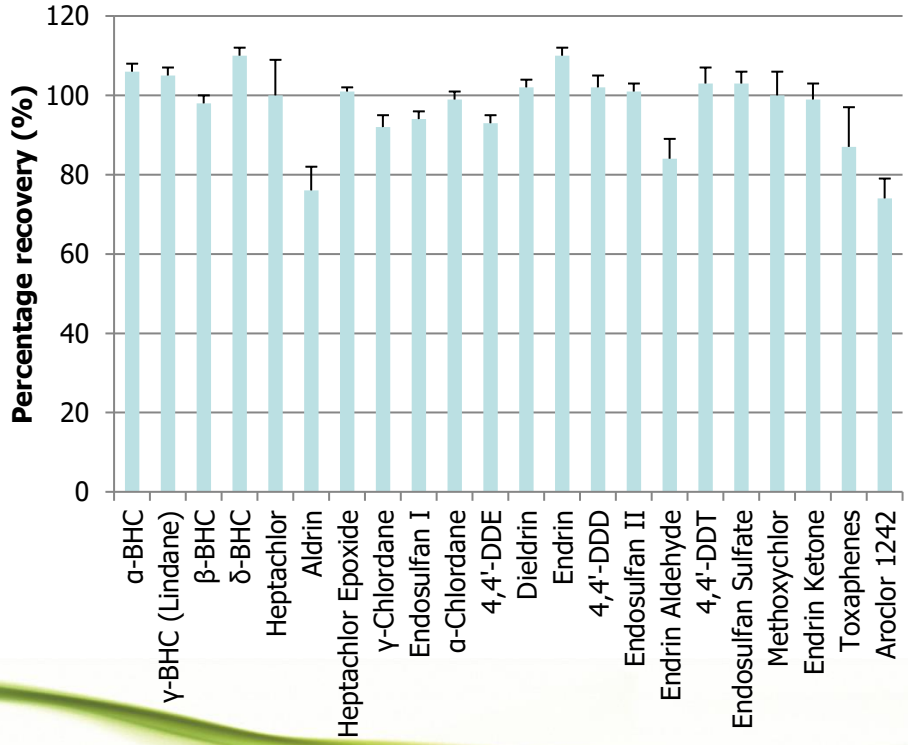
Concentration Features

- 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.

Evaporation

- Pre-heat temp: 55 ºC
- Pre-heat time: 15 min
- Heat in Sensor mode at 55 ºC under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis (ECD)

608.3 Results



Semi-Automated SPE in Summary

- EZSpe and SuperVap systems are easy to use and install
  - Complete Water Sample Prep Workflow
- Low cost, High throughput, Low maintenance solution
- EZSpe Extractions and Concentration is a very green technique
  - Reduces Solvent Use
  - Reduces Solvent Disposal Costs
  - Reduces Solvent emissions

Semi-Automated Summary

- Semi-automated SPE and SuperVap systems deliver consistent, reproducible results
- Handles a wide range of Sample sizes and matrix types
- Uses all SPE Cartridge and Column sizes
- Comply with existing methods that require vacuum, positive pressure and precise delivery of sample and solvents

Questions

Contact us at:

raddink@fms-inc.com
(Ruud Addink)

thall@fms-inc.com
(Tom Hall)