

# **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**

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. Semi-Automated SPE Methods (2)

## **LLE/CLE**

No Separation of waste

More volume to evaporate

Massive solvent emission

CLE uses a lot of power

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



# 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

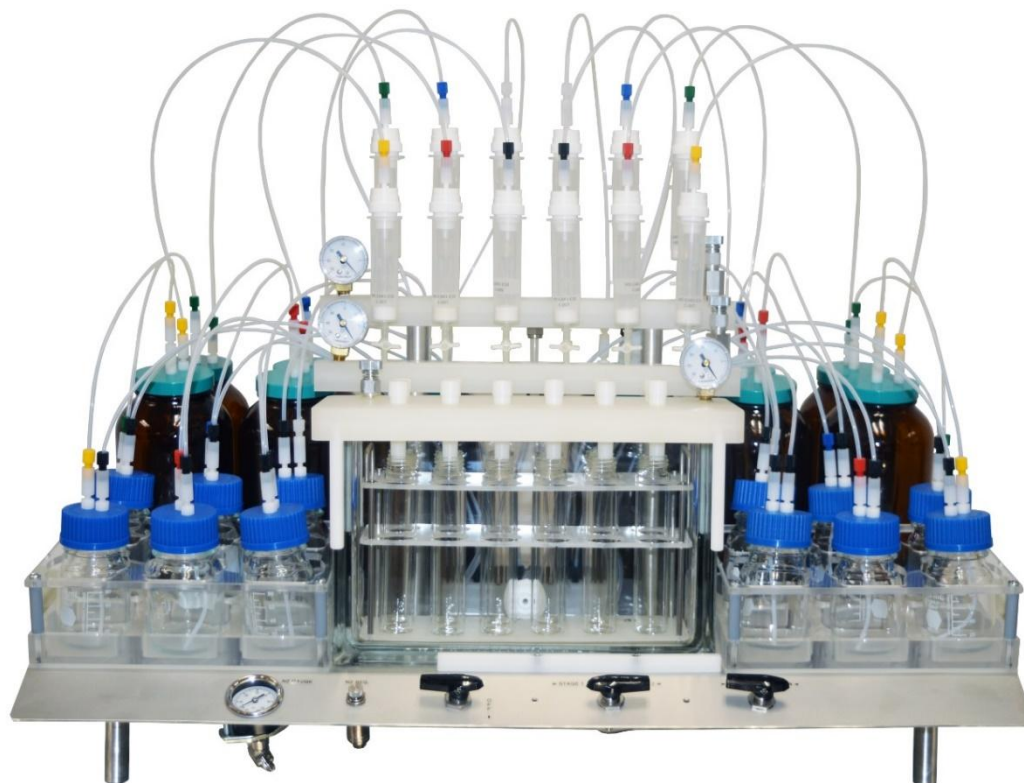


# Goal (2)

- **Efficient**

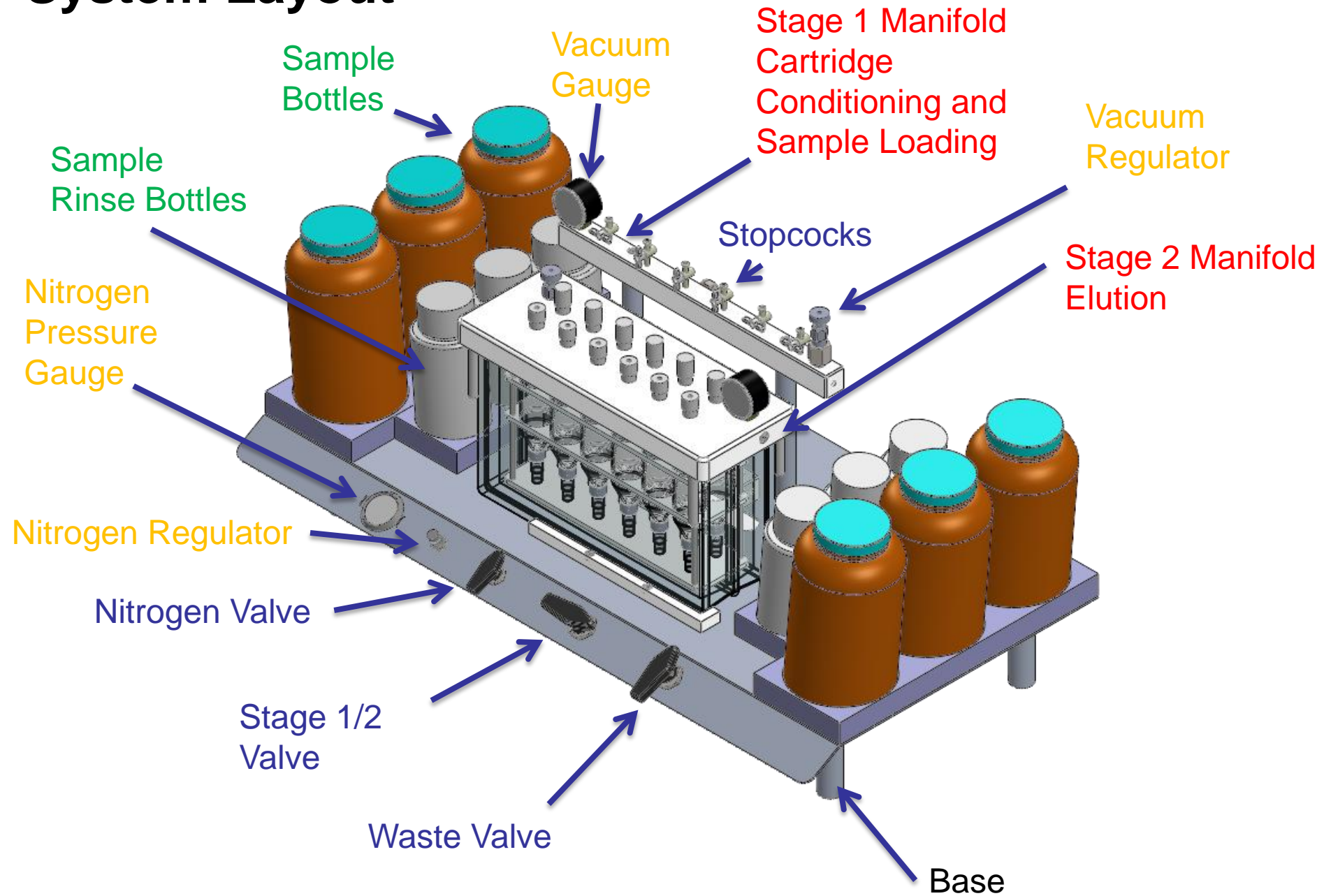
- Uses all SPE cartridge sizes
- Dedicated manifold for cartridge conditioning and sample loading
- Dedicated manifold for extraction and extracts
- Separates Organic from Aqueous waste
- Vacuum cartridge drying, Nitrogen cartridge drying or combined
- Automated Bottle Rinse and Elution
- Inline Extract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

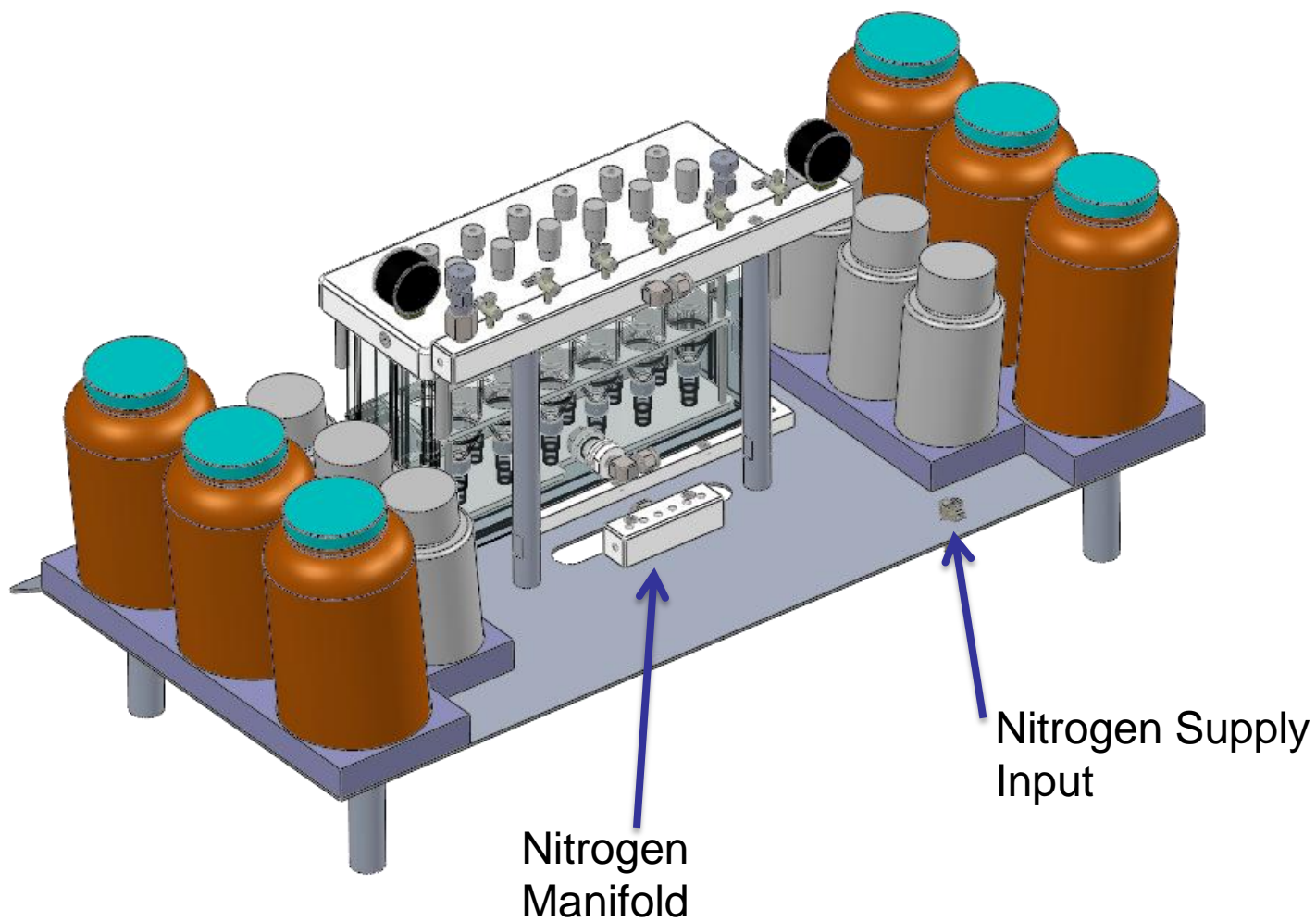




EZSpe 12 samples

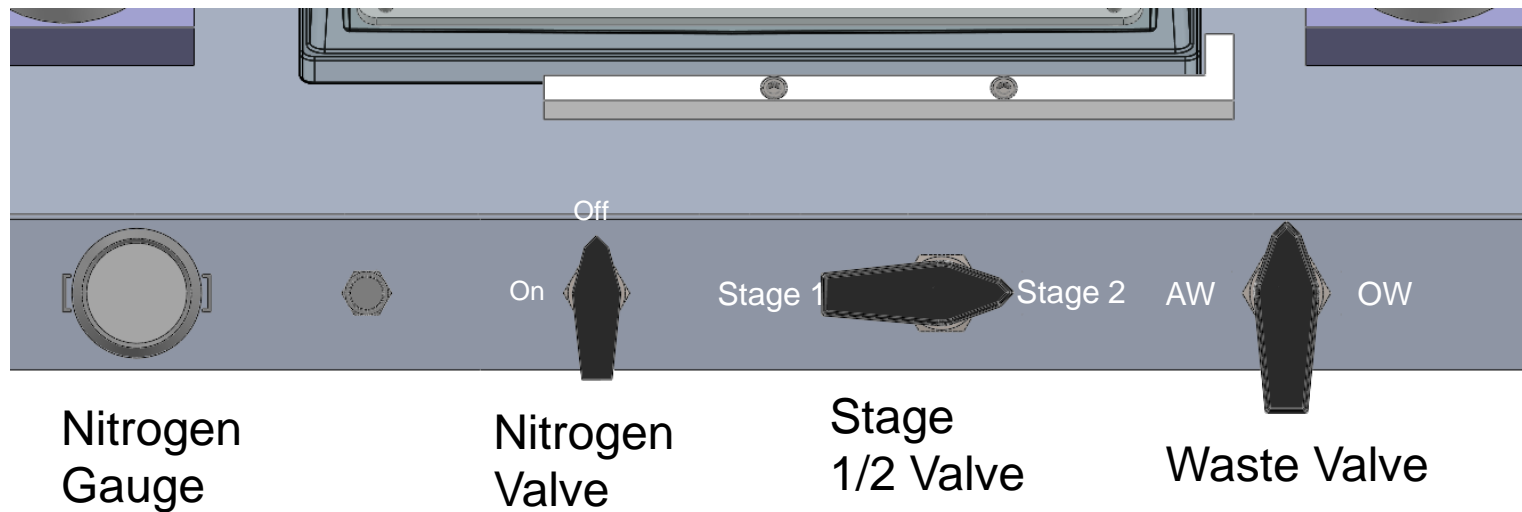
# System Layout





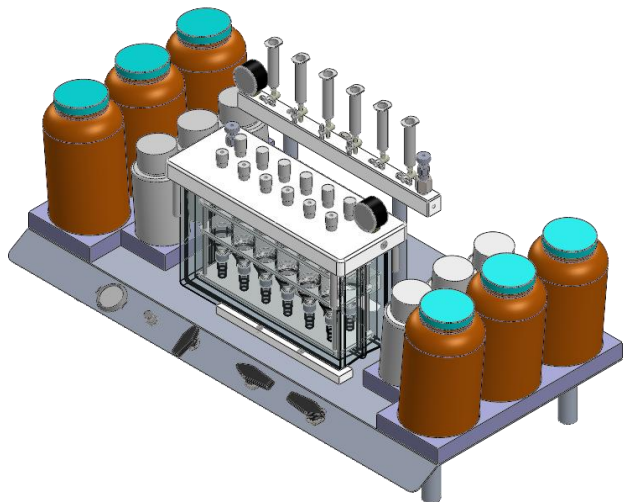


# Control Valve Layout



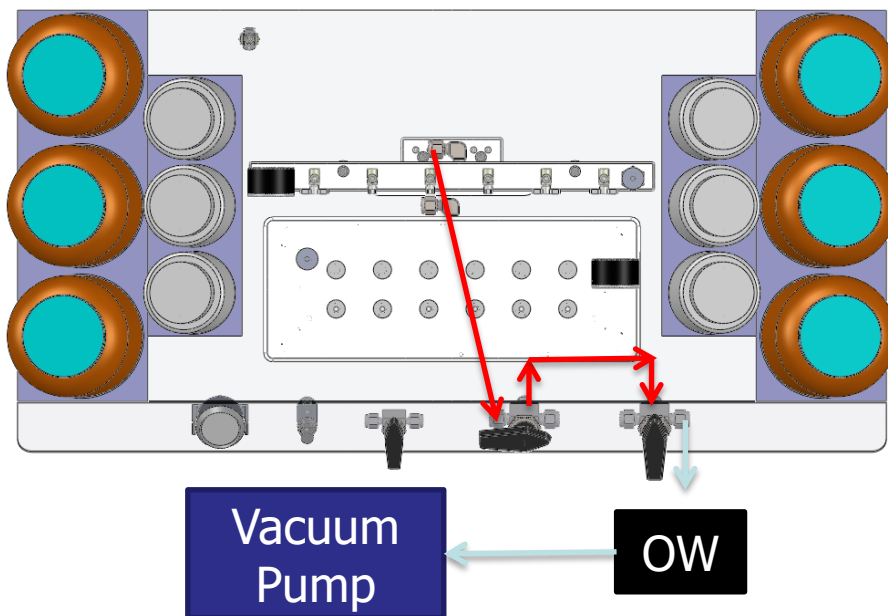
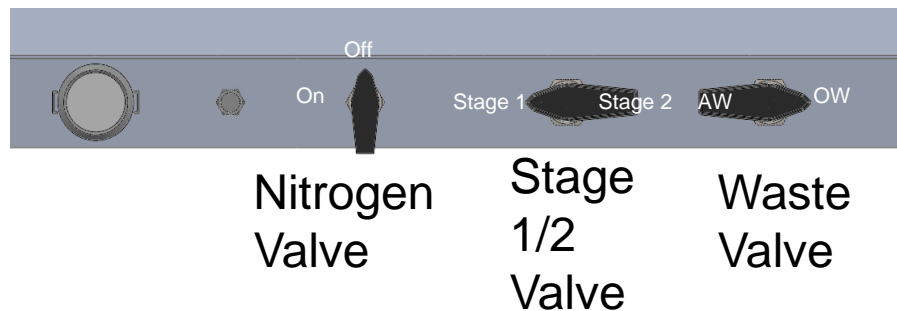


# Cartridge Conditioning (Stage 1, Organic Waste)

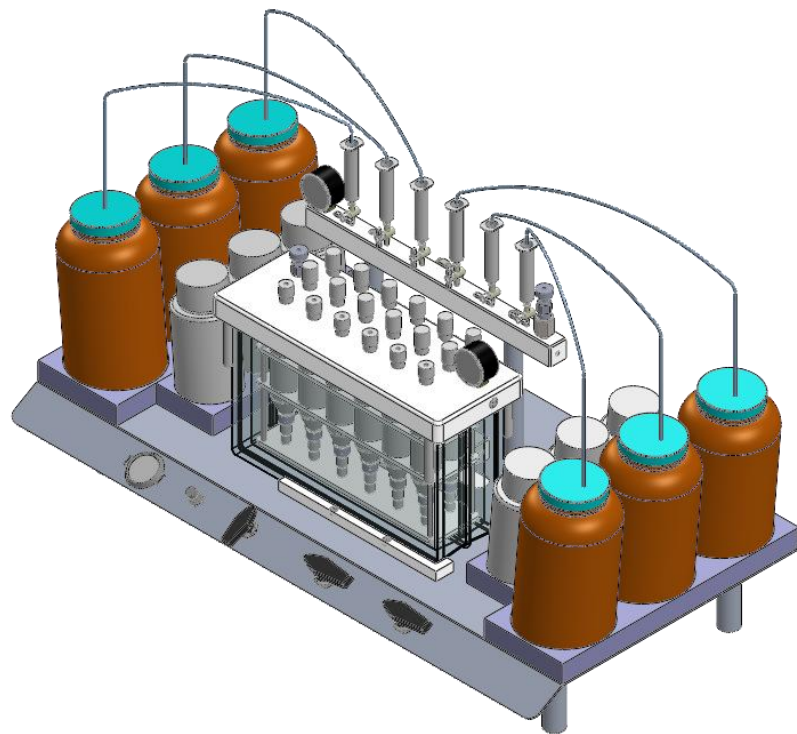


Flow  
Path

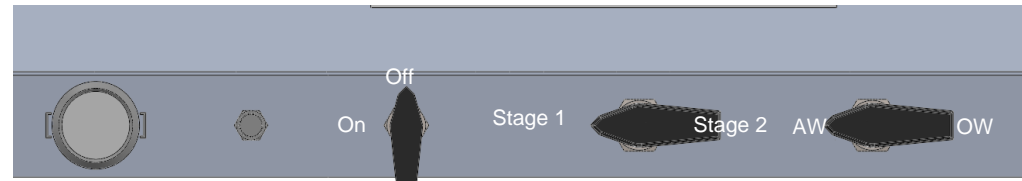
---



# Sample Loading (Stage 1, Aqueous Waste)



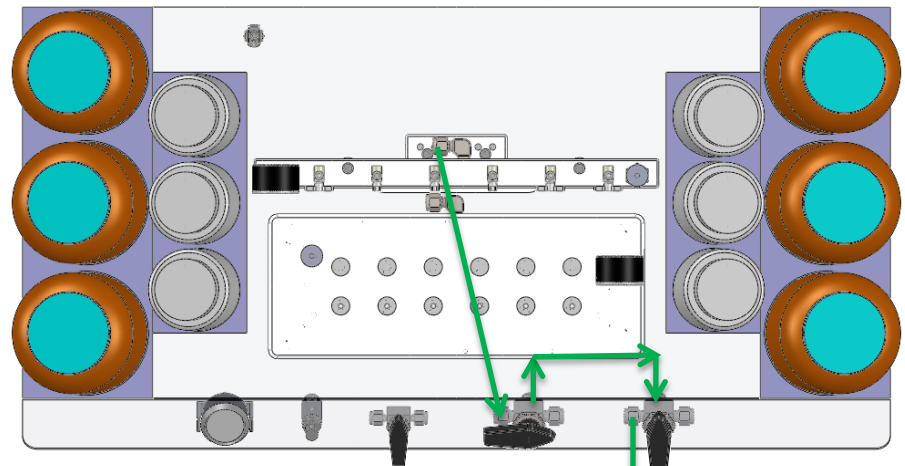
Flow  
Path



Nitrogen  
Valve

Stage  
1/2  
Valve

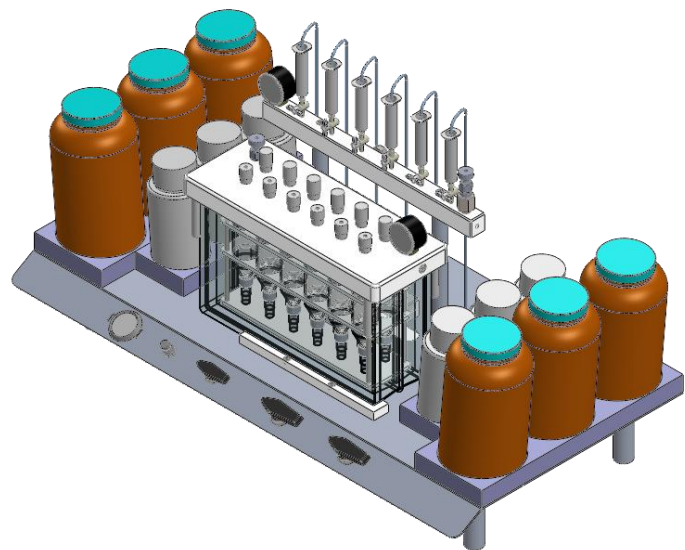
Waste  
Valve



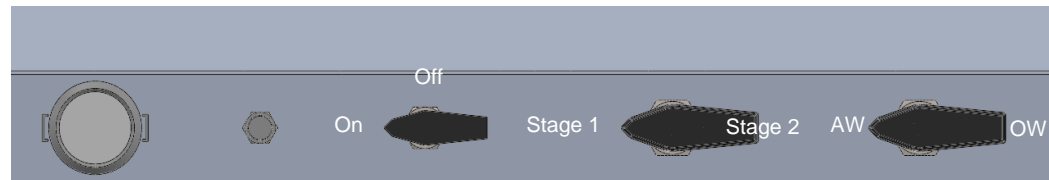
Vacuum  
Pump

AW

# Cartridge Drying- Nitrogen/Vacuum



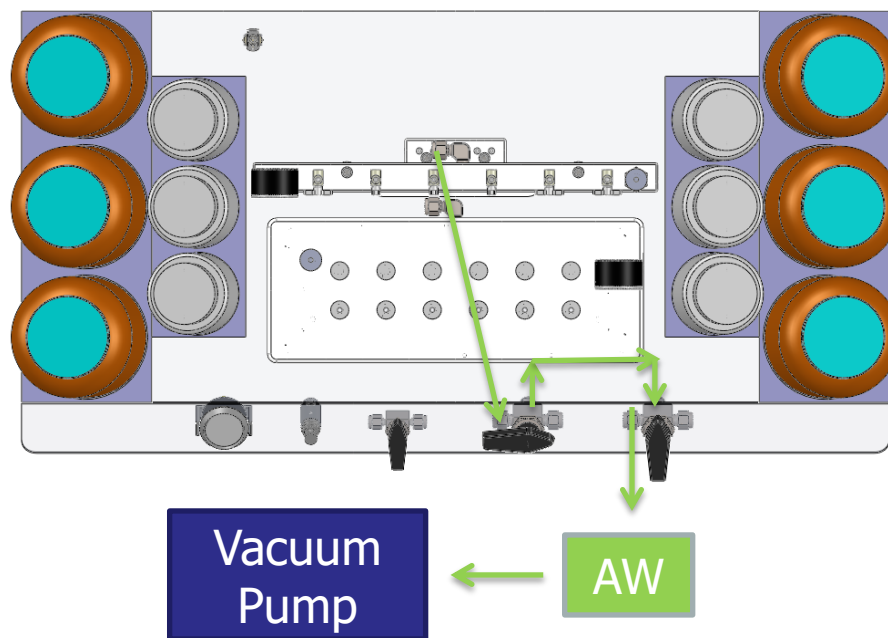
Flow  
Path



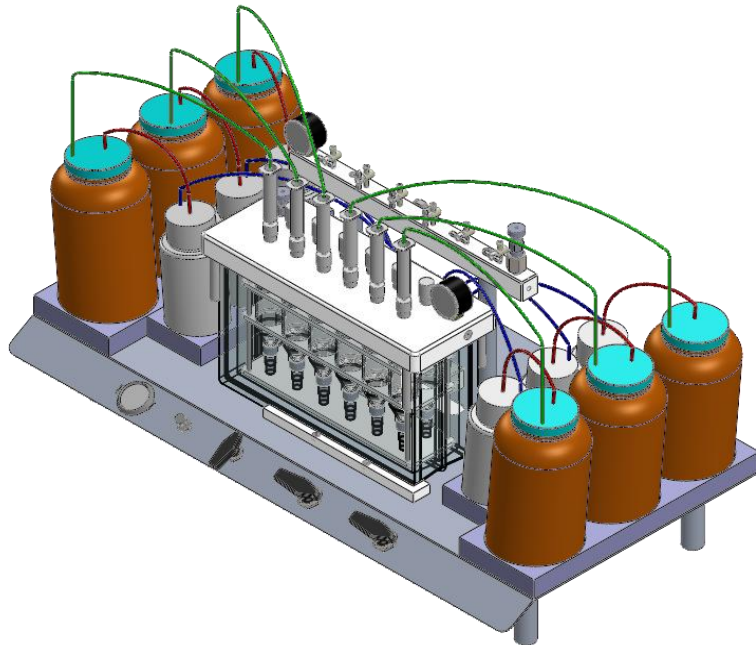
Nitrogen  
Valve

Stage  
1/2  
Valve

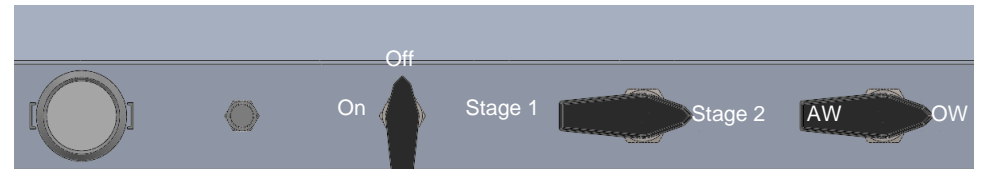
Waste  
Valve



# Sample Elution (Stage 2 )



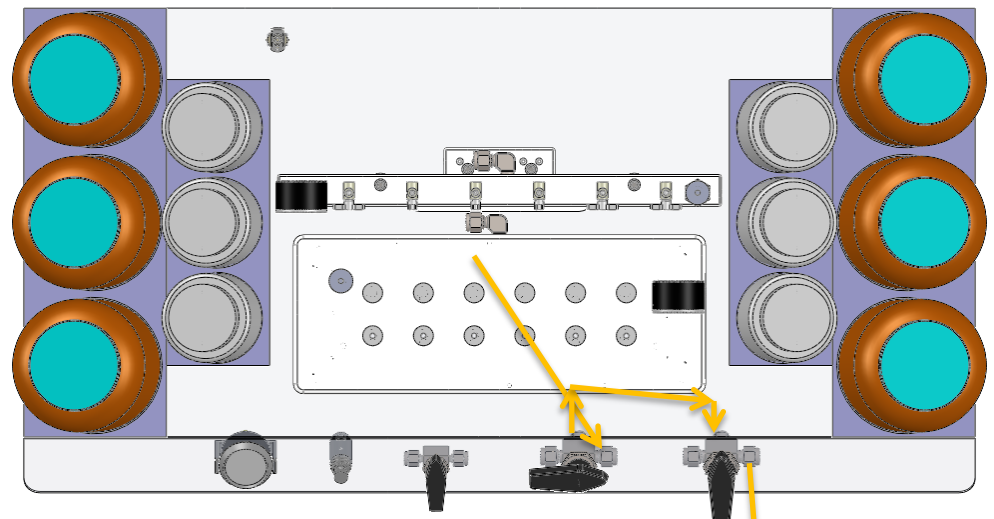
Flow  
Path



Nitrogen  
Valve

Stage  
1/2  
Valve

Waste  
Valve



Vacuum  
Pump

OW

# Procedure (1)

- 12 samples (1L water each) are prepared and acidified with 6 N HCl till  $\text{pH} < 2$
- 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)



# Direct to Vial Concentration



# SuperVap Concentrator

- 12 position
- 50mL vessel



# 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.

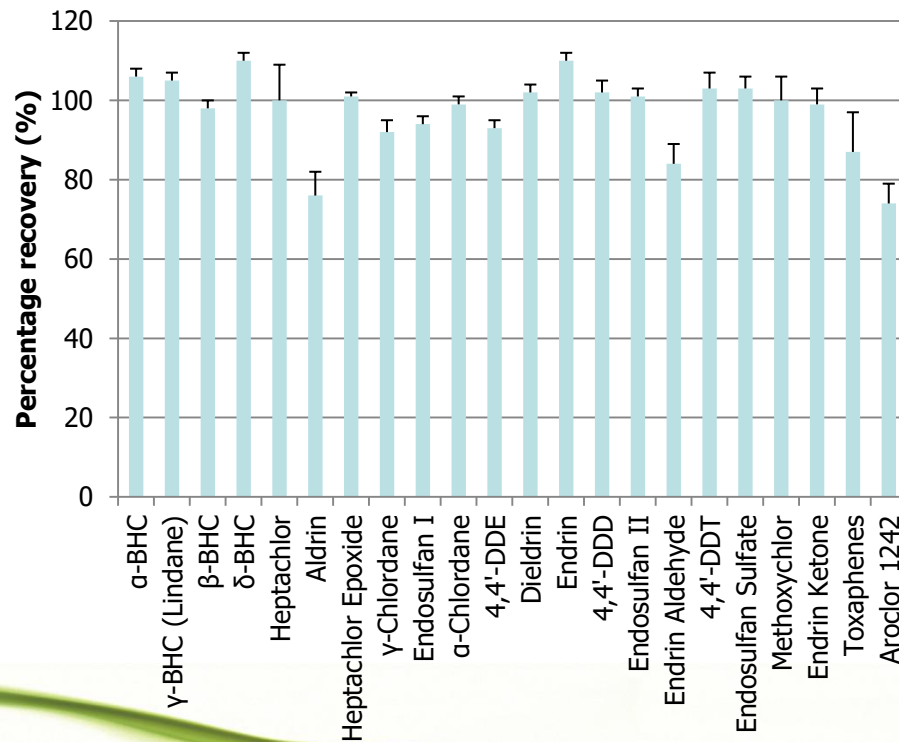


# Evaporation

- Pre-heat temp: 55 °C
- Pre-heat time: 15 min
- Heat in Sensor mode at 55 °C 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](mailto:raddink@fms-inc.com) (Ruud Addink)  
[thall@fms-inc.com](mailto:thall@fms-inc.com) (Tom Hall)

