

High Throughput Semi Automated Solid Phase Extraction and Concentration of PFAS in Waste-water

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Introduction

- Many of Thousands Samples are now being analyzed and more areas of concern are starting to be analyzed for PFAS
 - Drinking Water
 - Waste-Water
 - Human Serum
 - Biota
 - Soils

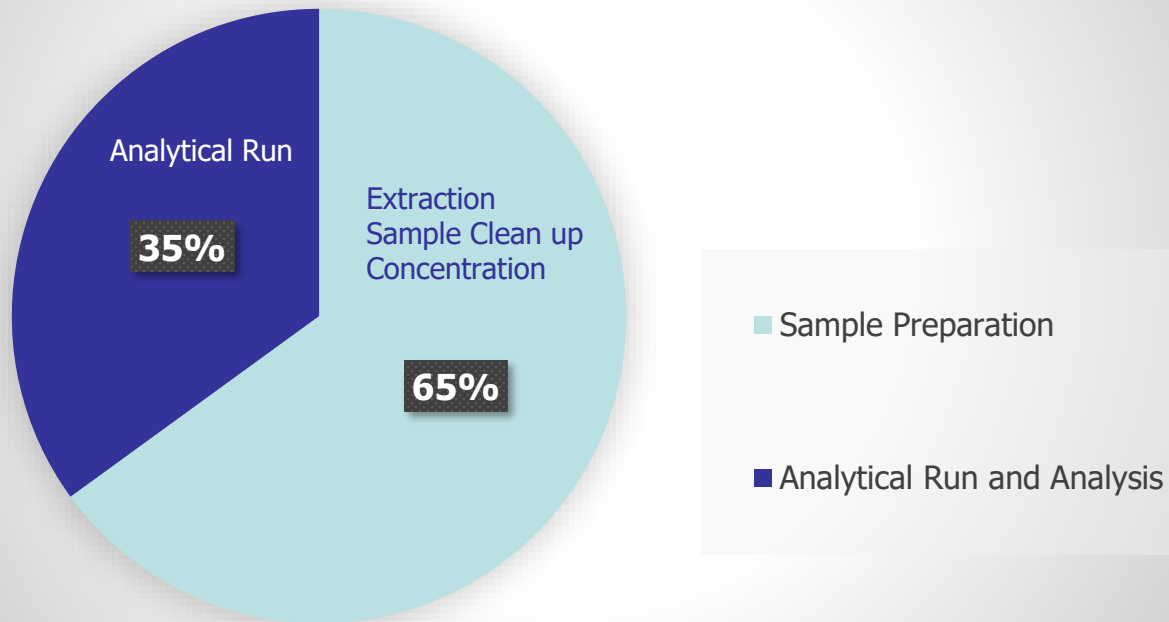


Challenges of Analysis

- The Analytical Systems are expensive
 - UPLC/MS systems
 - Require expertise in a new technology
- Manual Sample Prep processes
 - Inconsistent results
 - Elevated Background issues
 - Labor intensive
 - Extraction can take up to 2 hours
 - Dirty samples
 - Concentration can take up to 2 hours

Laboratory Workflow Breakdown

Sample Prep versus Analytical in Time



Reasons for Semi-Automated SPE

- **Reduced solvent**
- **Reduced Actions**
- **Simplified procedures**
- **Semi-Automated versus Manual protocols = Reproducibility**
- **Increased Sample Throughput**



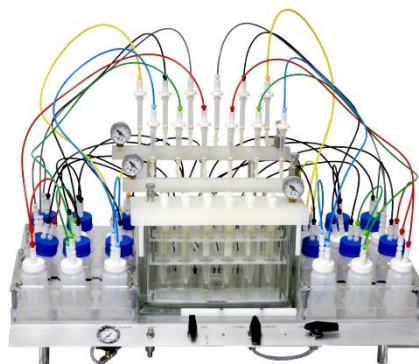
Determining Factors

- **Ability to load samples by vacuum consistently.**
- **Ability to dry cartridges by both vacuum and positive gas pressure (N₂).**
- **Easily handle a wide variety of cartridge designs and sizes without cumbersome modifications.**
- **Simple Sample delivery**
- **Automated Bottle Rinse**

Semi-Automated Solid Phase Extraction front end for GC/MS and LC/MS



EZSPE



EZPFC



Sample Analysis Work Flow

Automated Sample Prep Time

= 80 Minutes



Solid Phase Extraction

35 Minutes



Concentration

45 Minutes

Semi Automated Sample Prep Time

= 80 Minutes

Solid Phase Extraction

35 Minutes



Concentration

45 Minutes

Objective for Semi Automation

- **Use as many features from the FMS 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

- **Self Installable**
 - Unpacking and Installation/training video
- **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 12 samples simultaneously
 - Vacuum for fast loading of large volume samples
- **Closed system**
 - Eliminate potential outside contamination

Goal

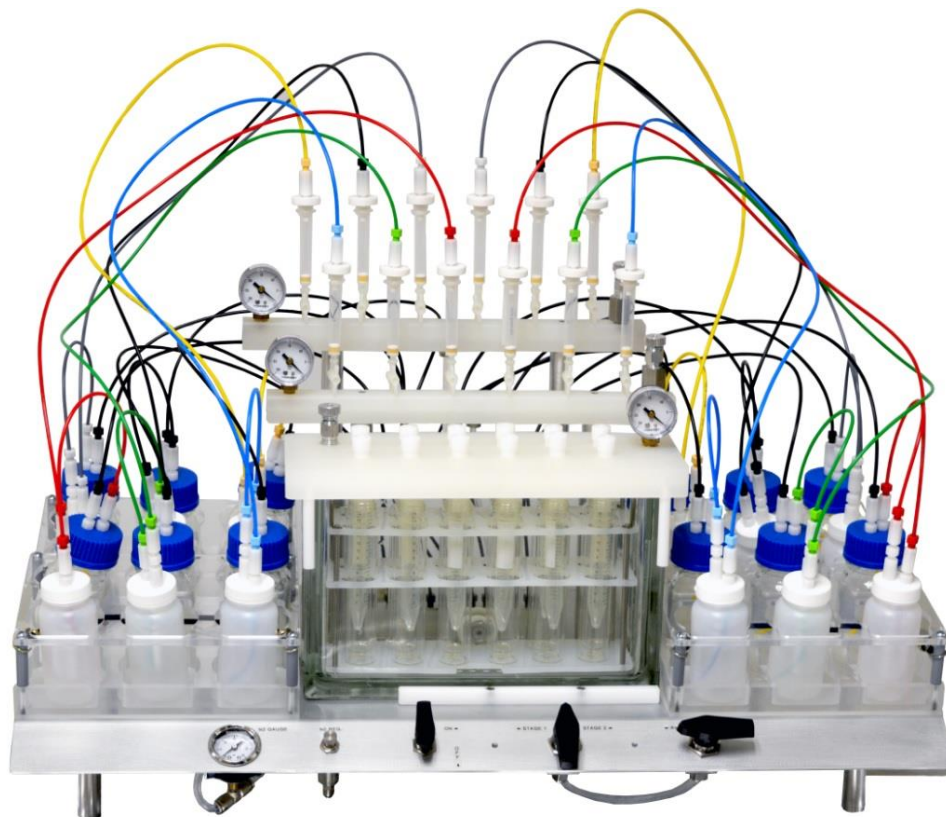
- **Efficient**

- 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

- **Low to No Capital Expense**

- Purchase an FMS Cartridge Contract
- receive an EZSpe at No Charge





EZPFC 12 sample

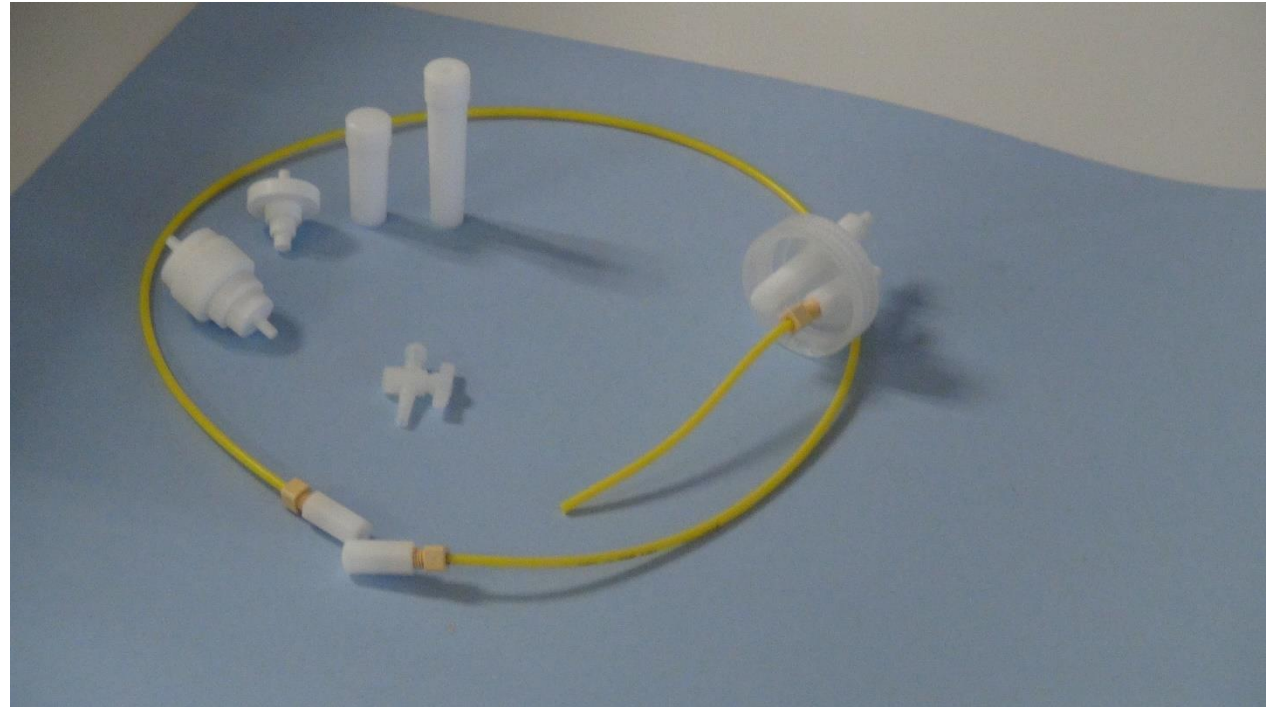
System Components

No Teflon

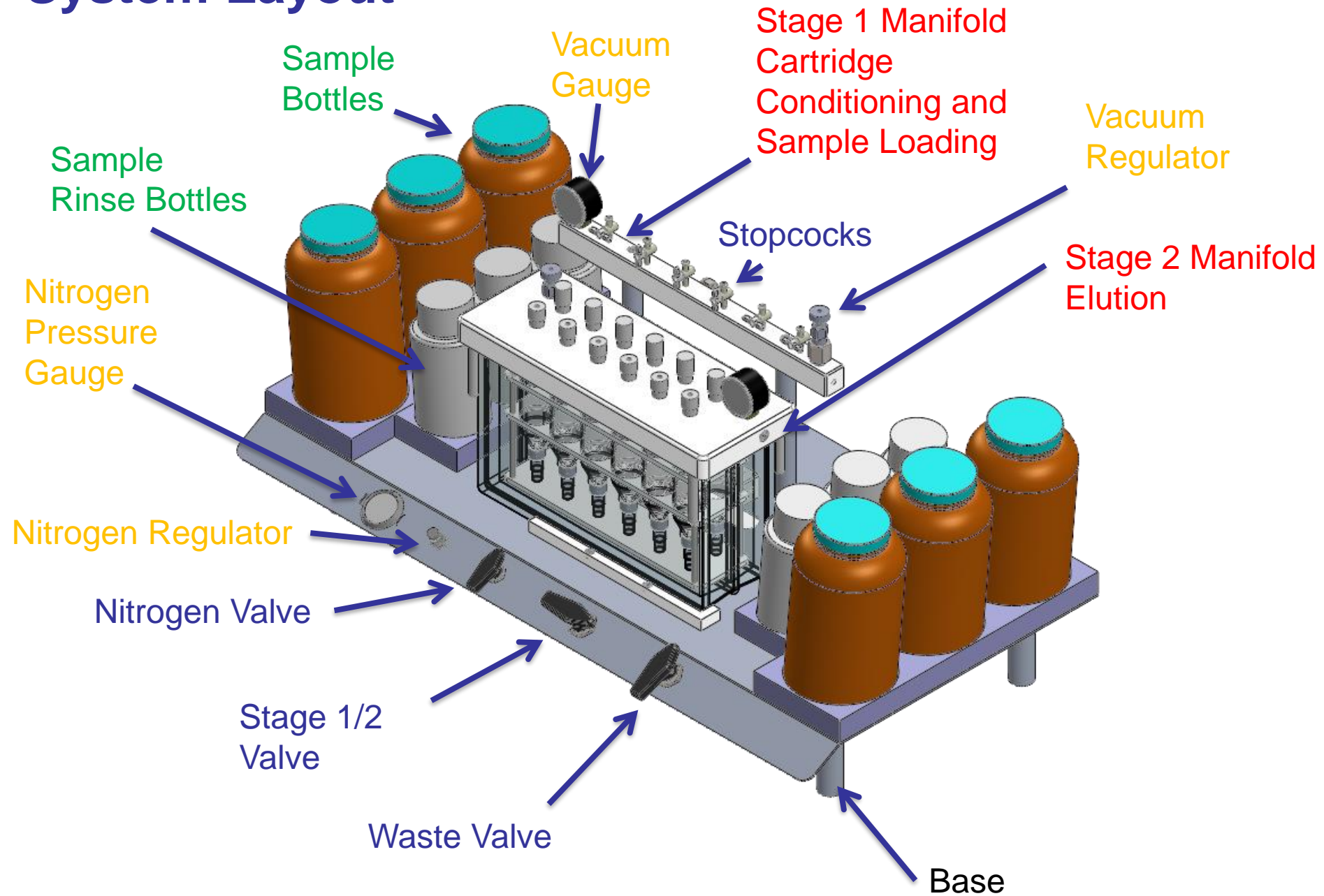
**Tubing - High Density
Polyethylene**

Fittings – Delrin

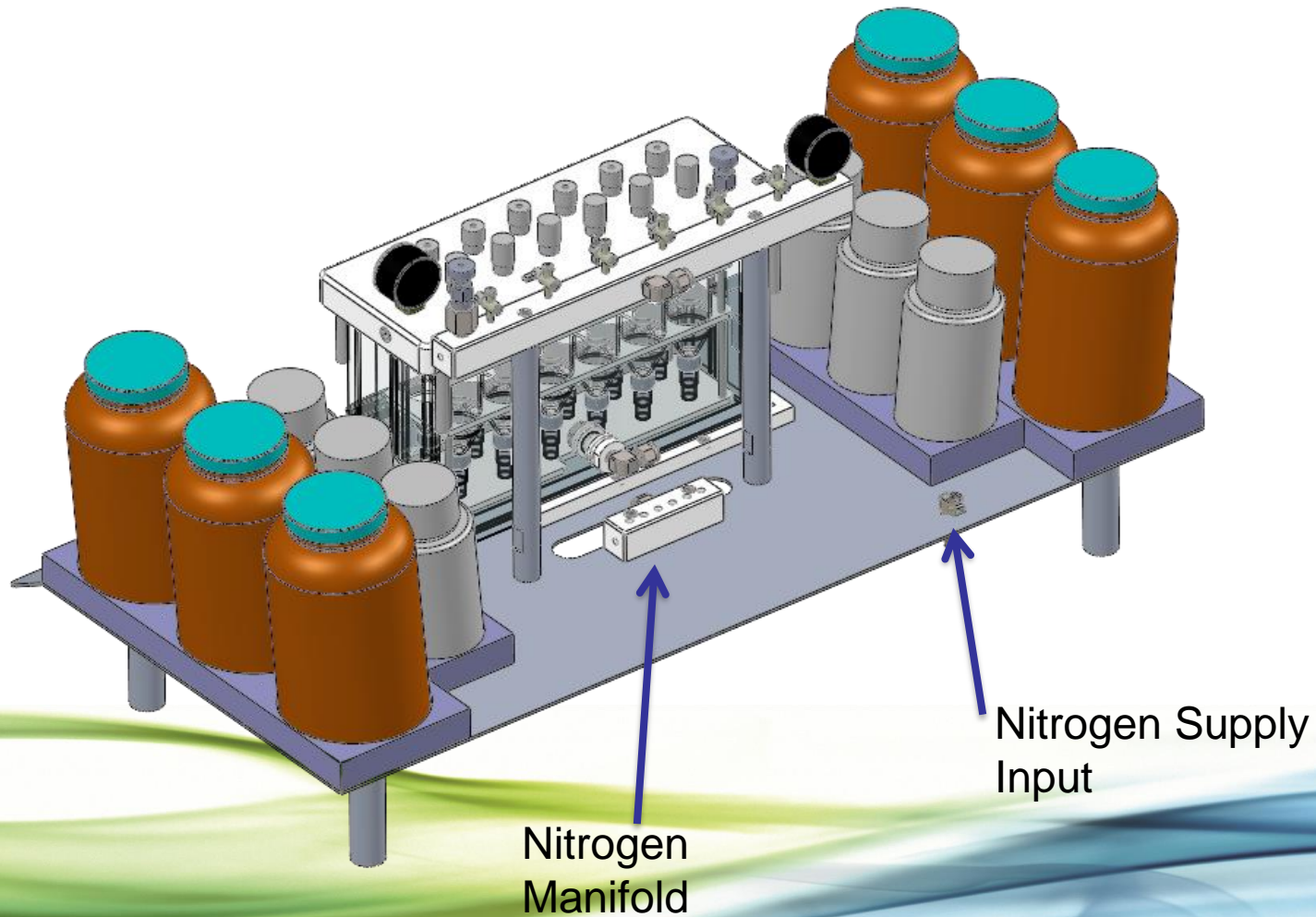
**Cartridge Adapters –
Medical Grade
Polypropylene**



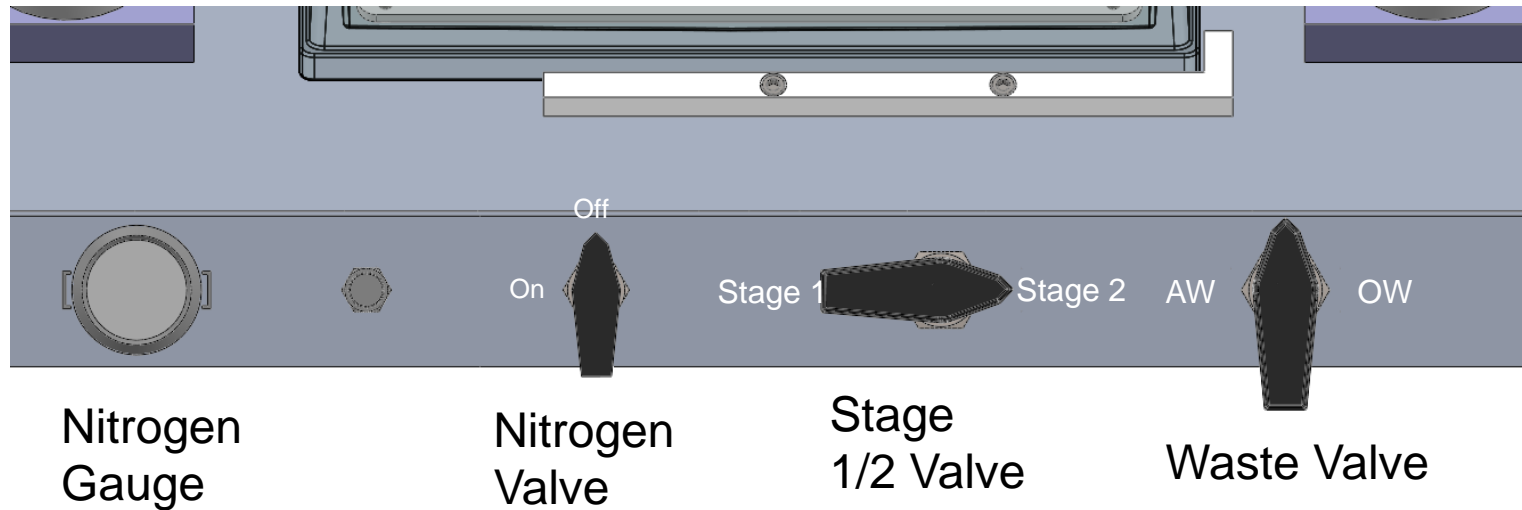
System Layout



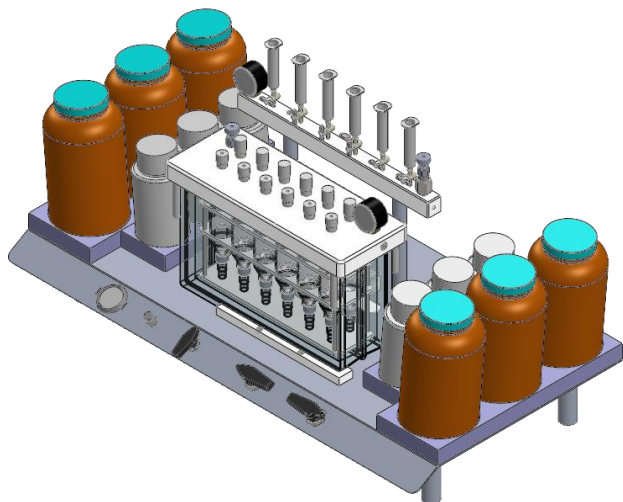
Nitrogen for Bottle Rinse and Cartridge Drying



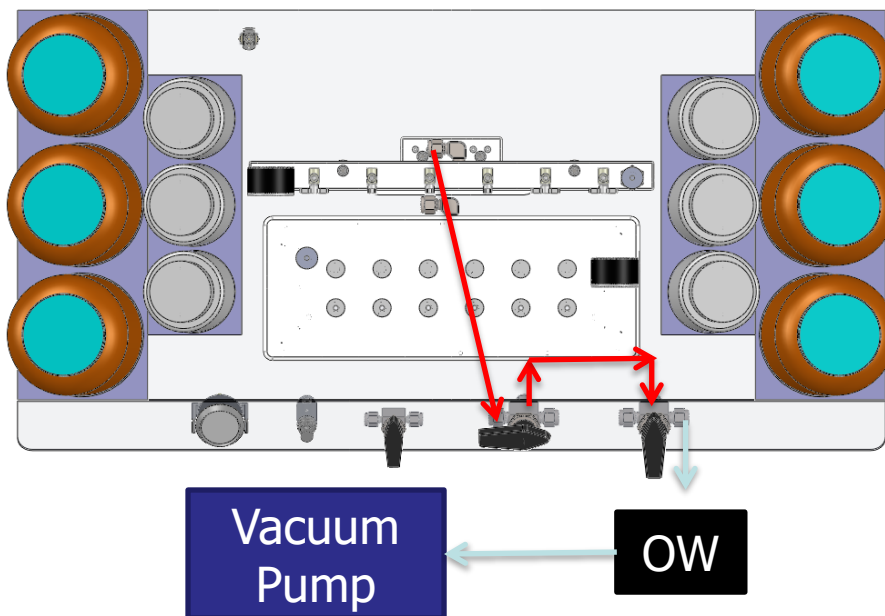
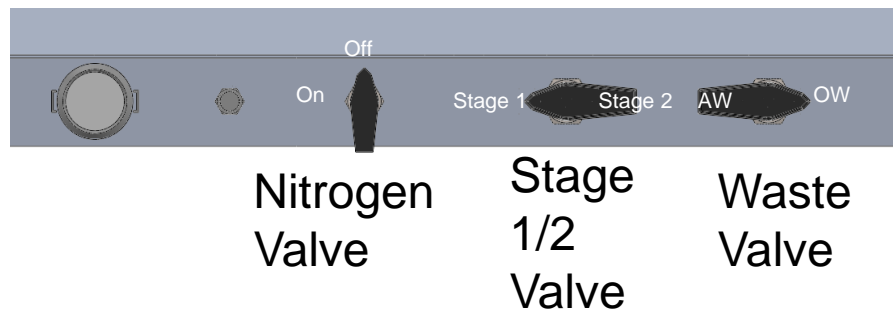
Control Valve Layout



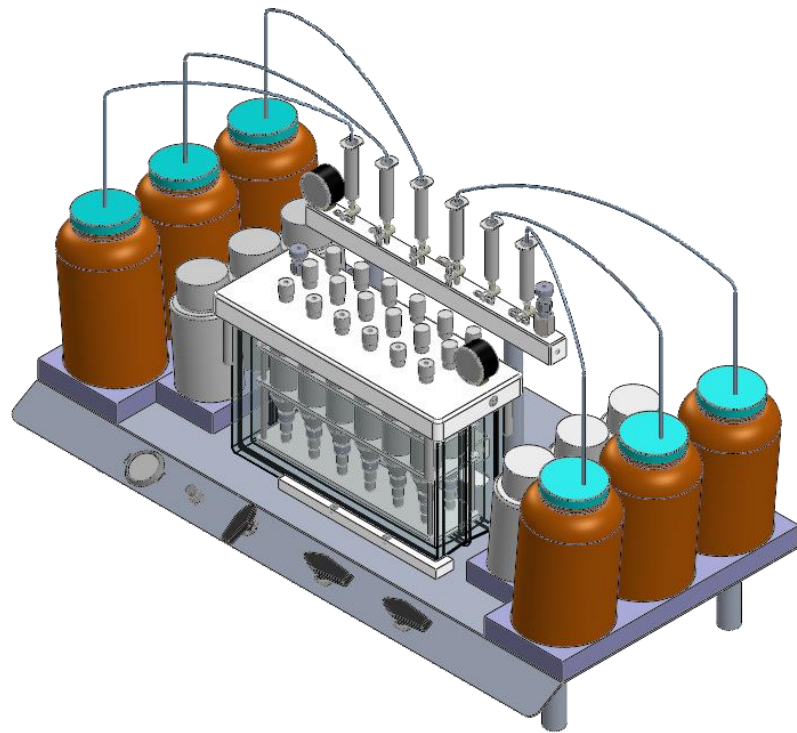
Cartridge Conditioning (Stage 1, Organic Waste)



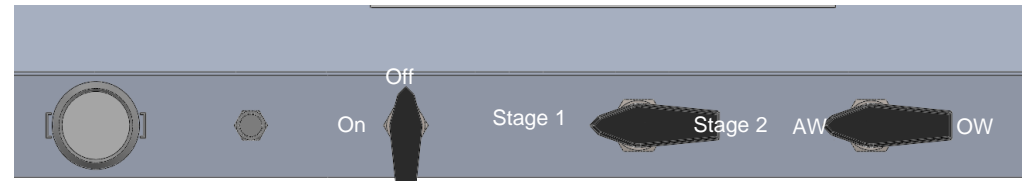
Flow
Path



Sample Loading (Stage 1, Aqueous Waste)



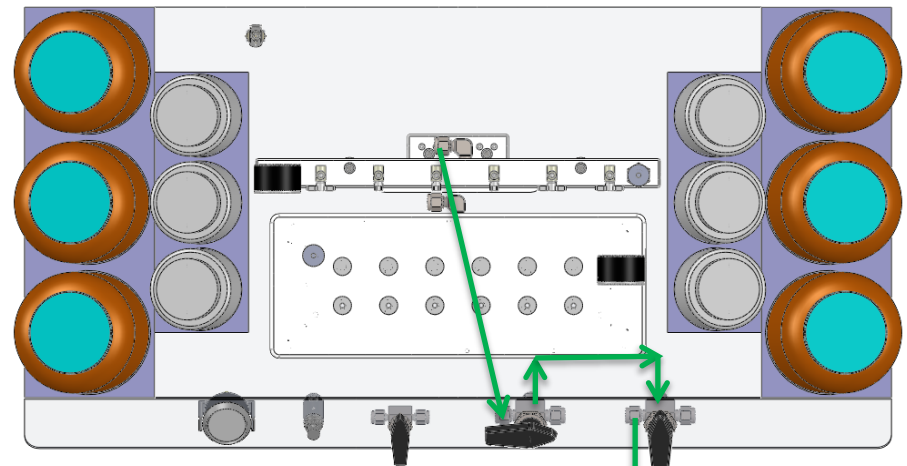
Flow
Path



Nitrogen
Valve

Stage
1/2
Valve

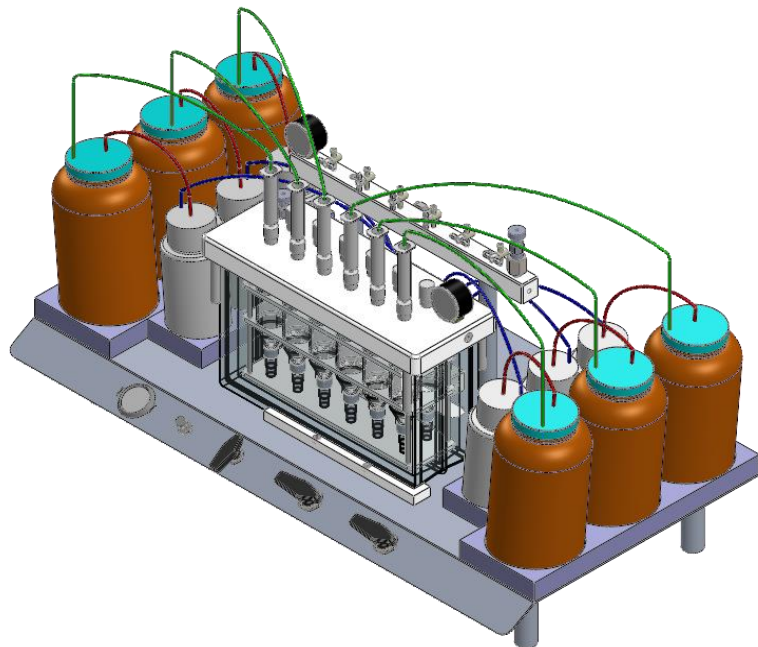
Waste
Valve



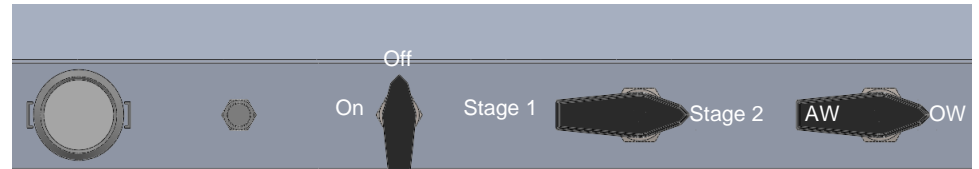
Vacuum
Pump

AW

Sample Bottle Rinse (Stage 1)



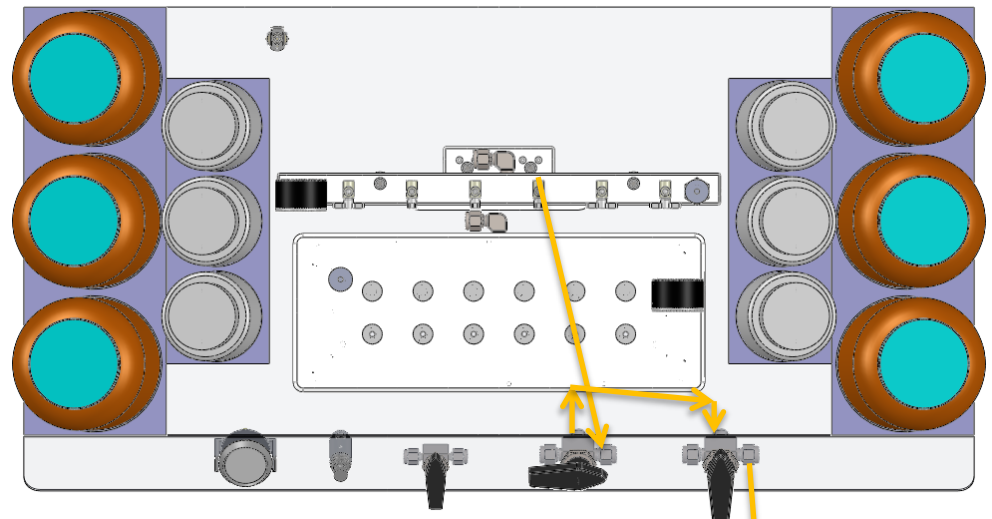
Flow
Path



Nitrogen
Valve

Stage
1/2
Valve

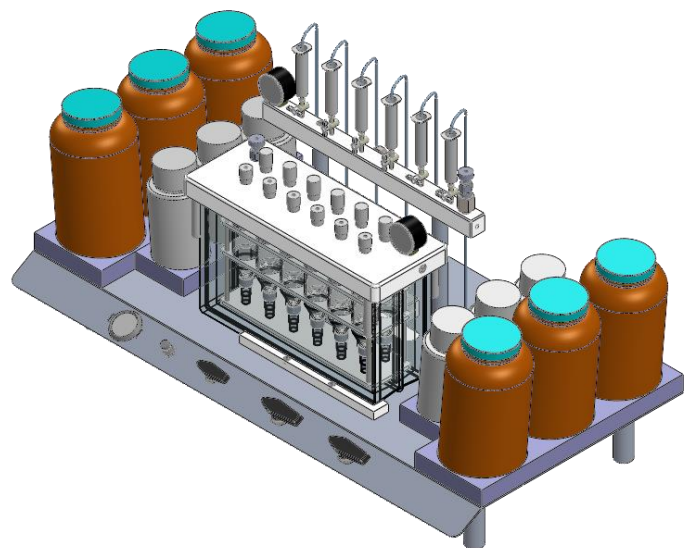
Waste
Valve



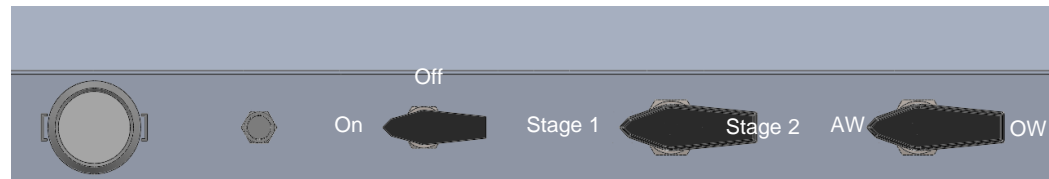
Vacuum
Pump

OW

Cartridge Drying- Nitrogen/Vacuum



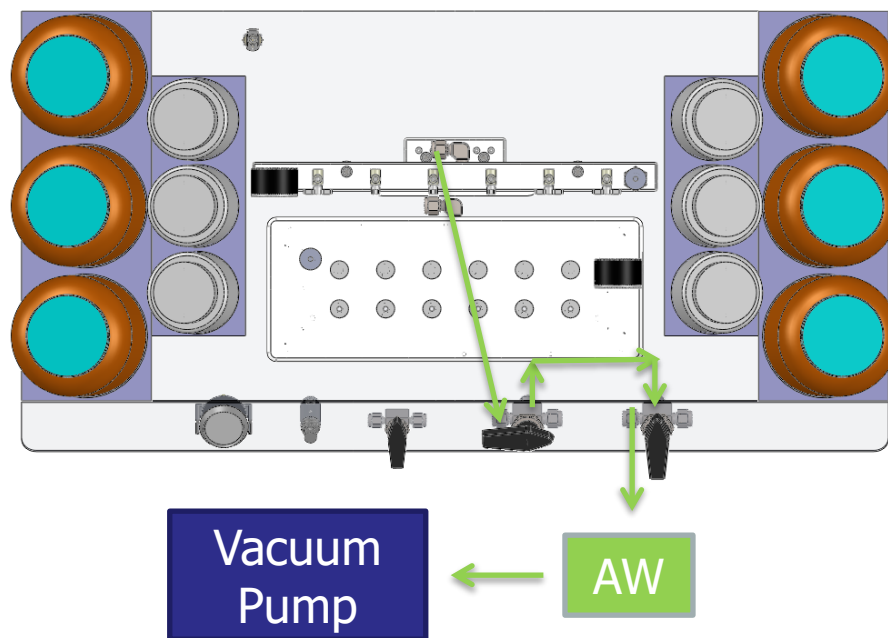
Flow
Path



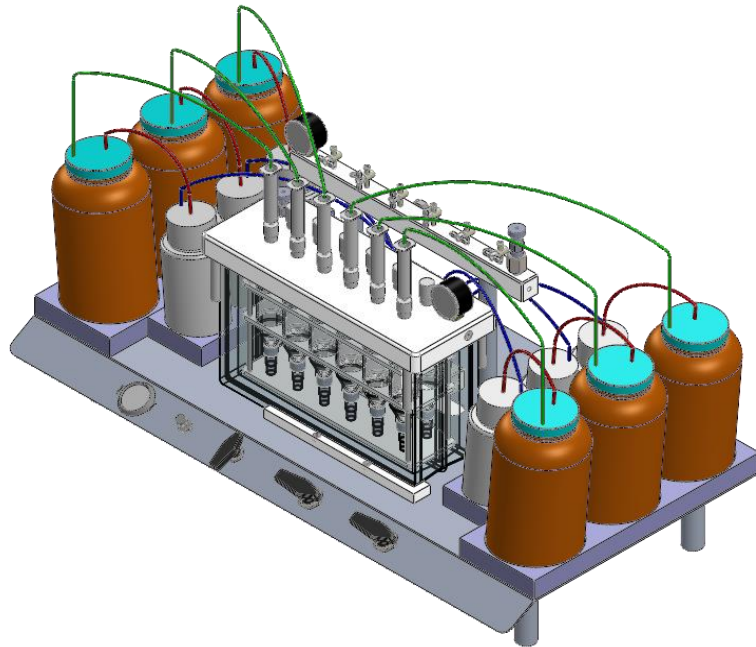
Nitrogen
Valve

Stage
1/2
Valve

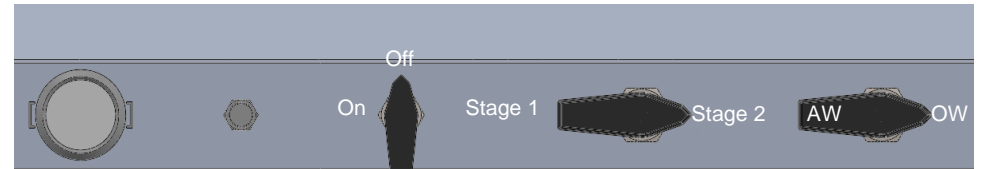
Waste
Valve



Sample Elution (Stage 2)



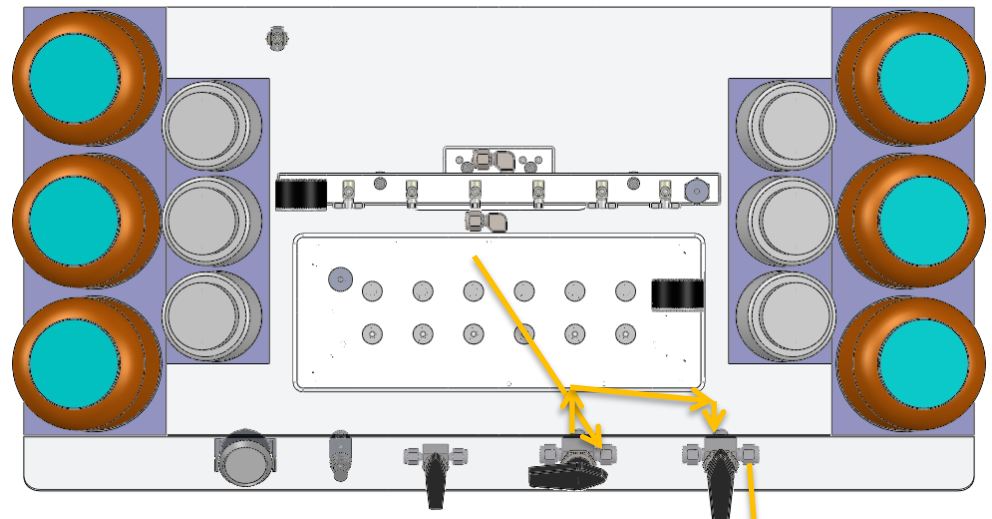
Flow
Path



Nitrogen
Valve

Stage
1/2
Valve

Waste
Valve



Vacuum
Pump

OW

PFAs Methods

- EPA 537.1
- EPA 537 v1.1
- EPA 533
- ISO 25101



Automated Concentration for PFAs

- SuperVap PFC
 - 24 positions
 - 15ml Conical vials
 - Timed Endpoint



SuperVap Features

- **Self Installable**
 - Video unpacking, installation and training video
- **Preprogrammed with most common temperature settings**
- **6 (250mL) and 12 (50mL) position models for extractions, direct-to-vial connections**
- **Dry bath heating element**
- **Time based endpoint**
- **Savable temperature log**



Can this Handle Dirty Samples?

Typical Cartridge can have problems!

- **6ml 500mg DVB**
 - Doesn't do well
 - Frit Surface Area is too small

Yes, A Cartridge will work

- **25ml 500mg DVB cartridge**
 - Does well
 - 3X the Frit Surface Area



Delrin Plastic Wool

- Irregular random stranding
- Slows Particles to the Uniform Frit
- Prevents Clogging



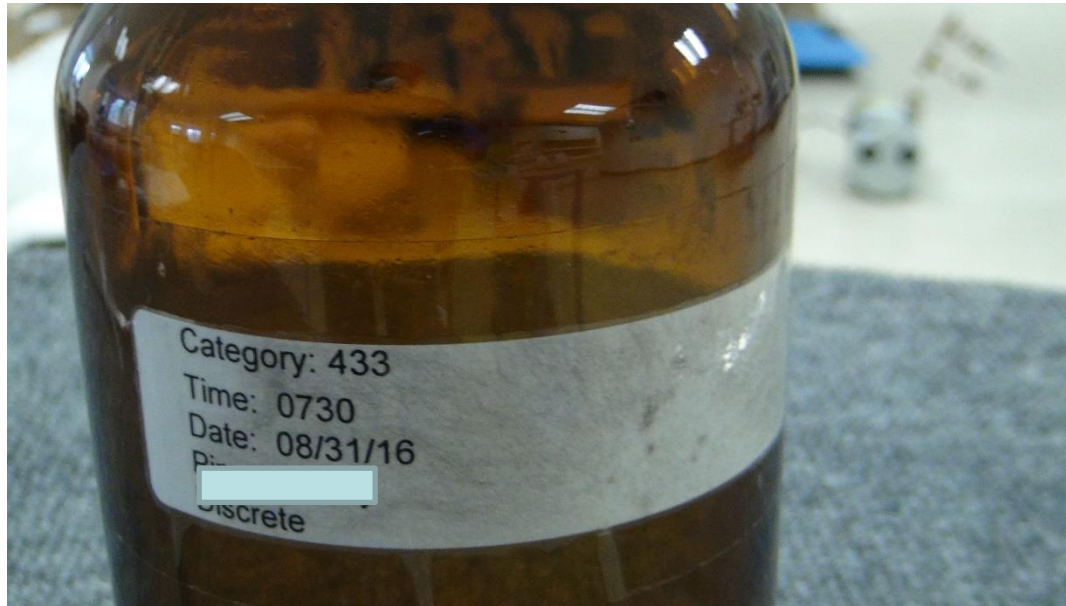
Prepping the 6ml Cartridge with Plastic Filtration Wool

6ml 500mg DVB cartridge with Plastic wool

- Take a little and push it into the barrel of the syringe until it touches the cartridge Frit
- The Sample will not clog, it will take longer to process



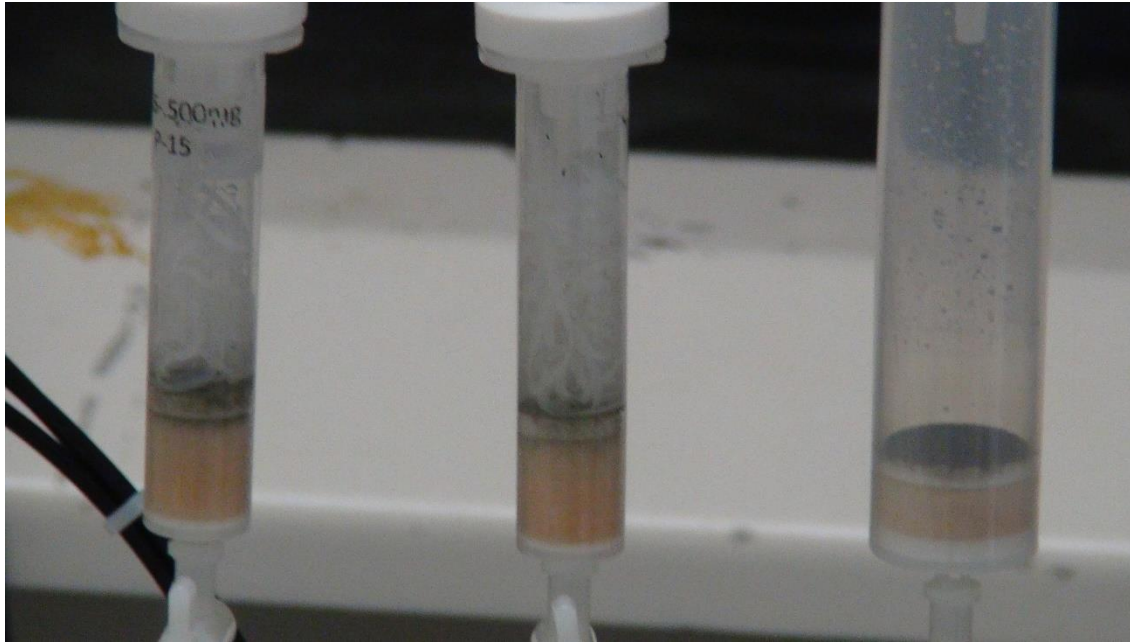
Dirty Sample from a Customer



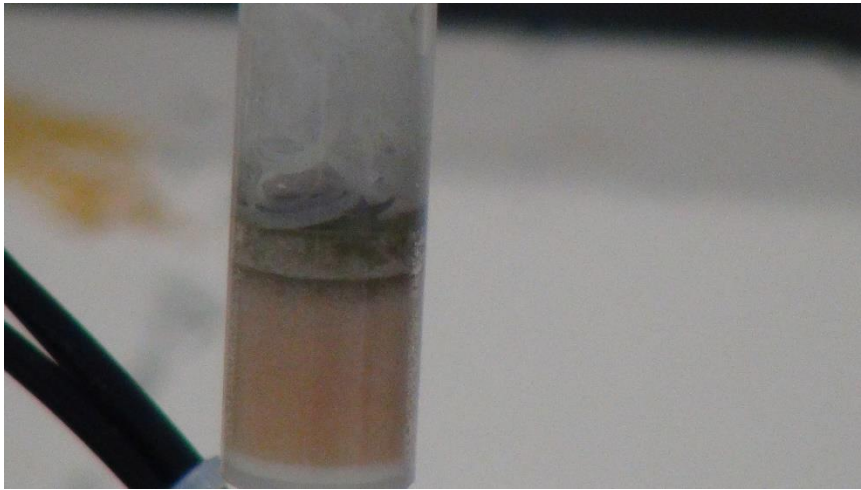
Industrial 433 Matrix 250ml



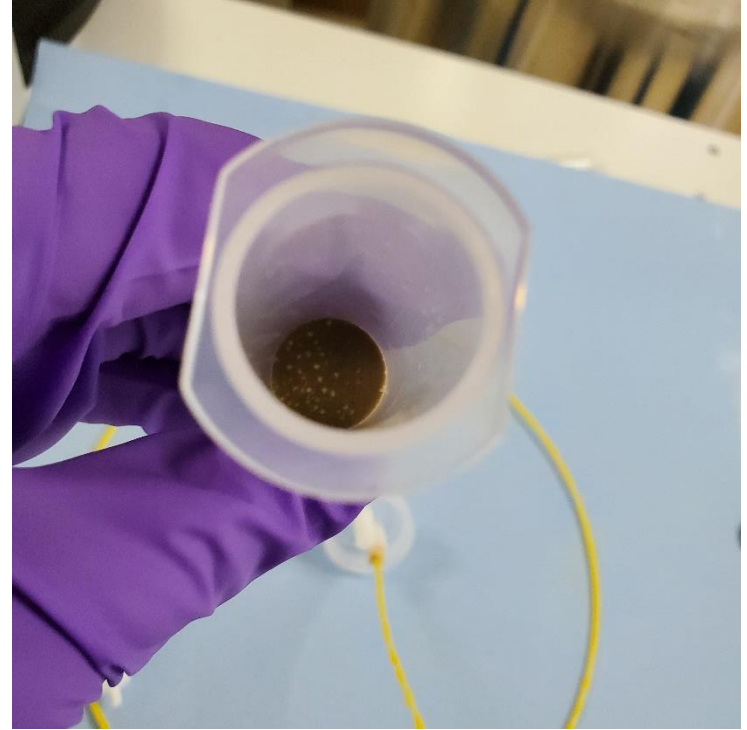
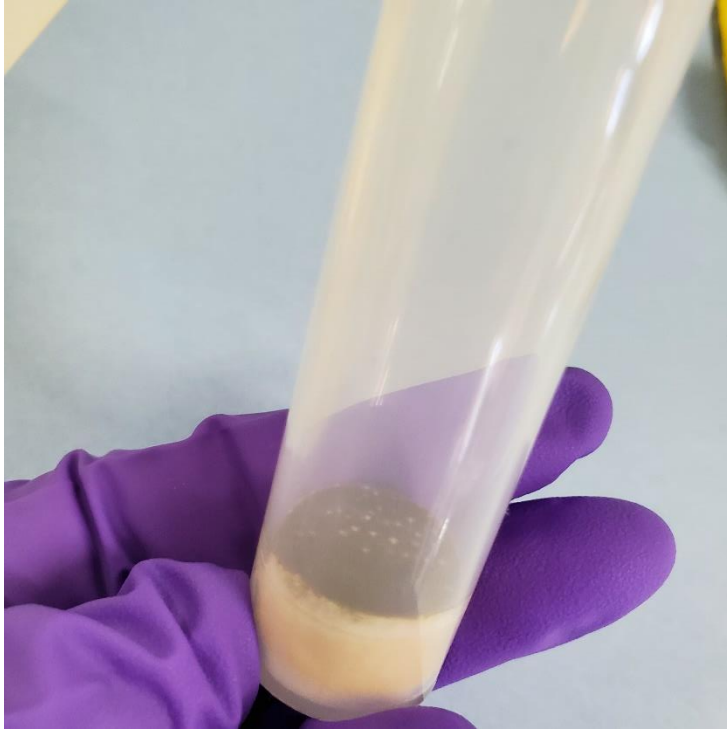
6ml and 25ml Cartridges



250 ml run to completion on 6 ml cartridge with Plastic Wool



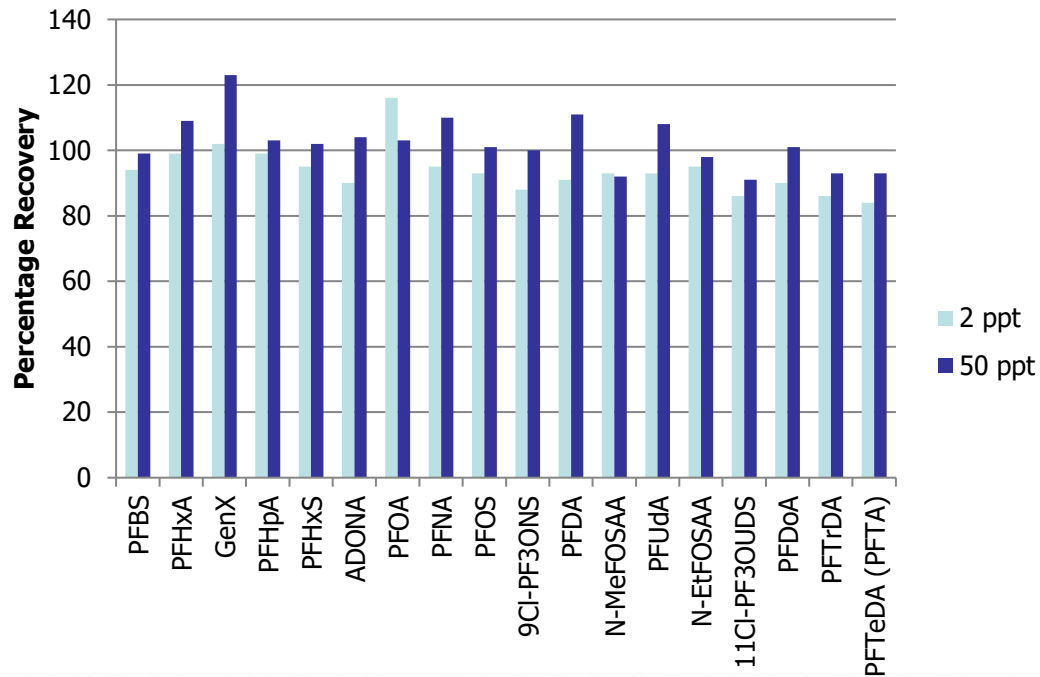
250ml run to completion 25ml cartridge



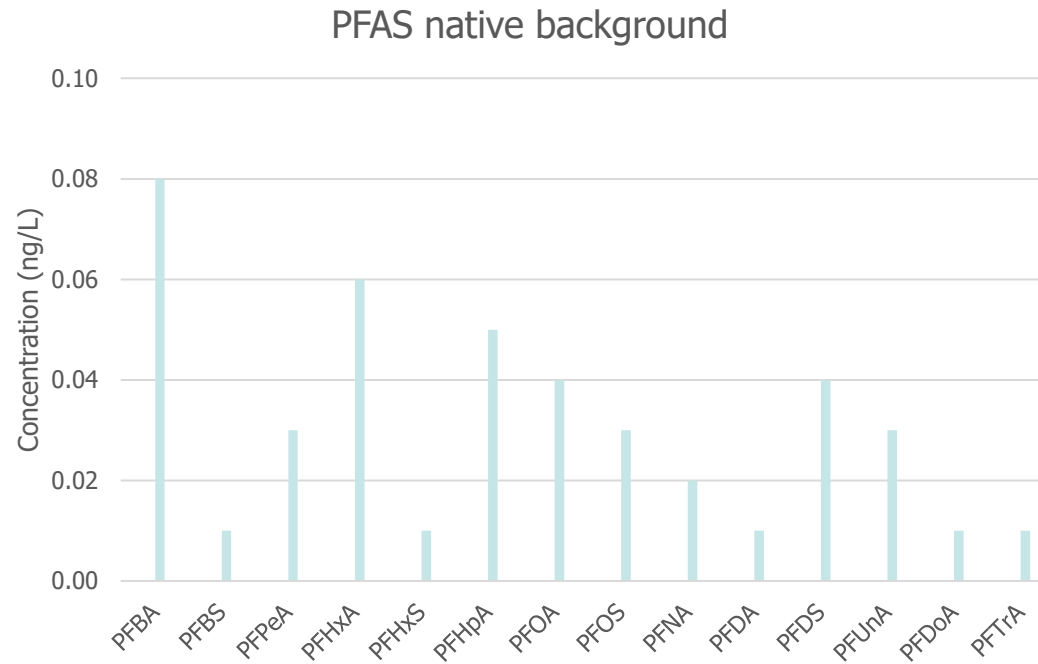
Clean up is easy with no cross contamination

- Back Flush the sample line into the original sample bottle with an IPA non-Teflon squirt bottle.
- Wash the inside of the bottle cap with IPA squirt bottle
- Wash Cartridge Adapters with IPA squirt bottle or sonicate in a beaker
- Ready for the next 12 samples

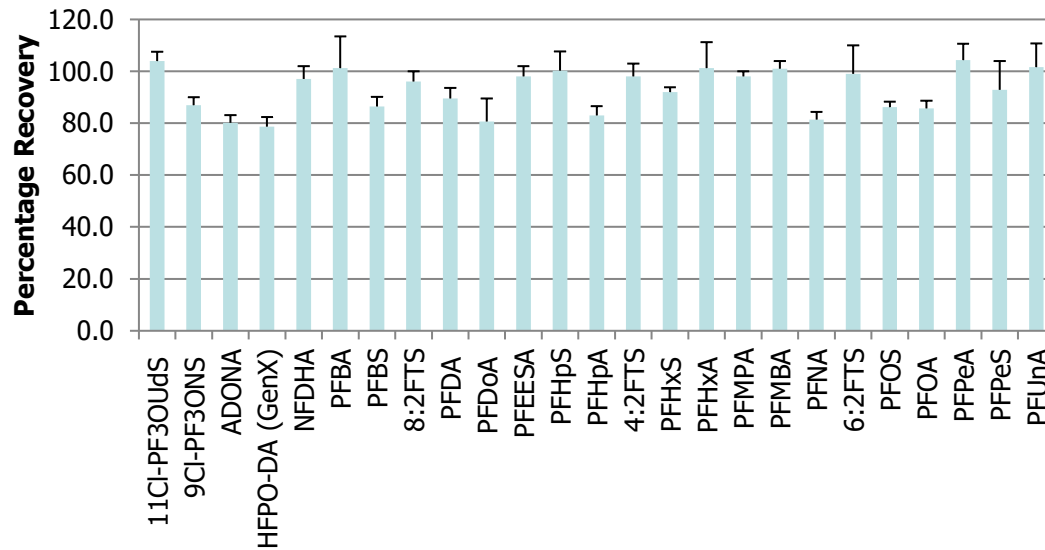
EPA 537.1 Recoveries



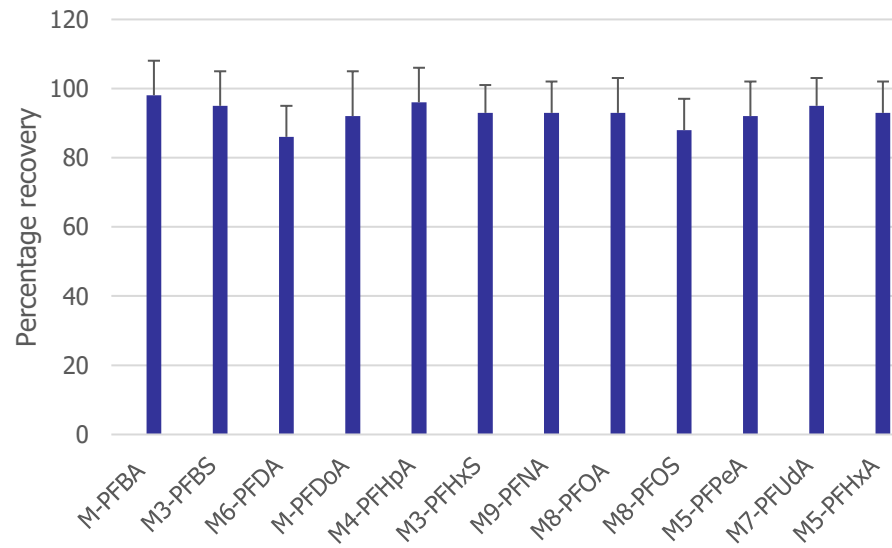
PFAS Background



EPA 533 Recoveries



Labeled PFAS recoveries



- **EZPFC and SuperVap systems are easy to use and install**
 - Complete Water Sample Prep Workflow
- **Low cost, High throughput, Low maintenance solution**
- **EZPFC Extractions and Concentration**
 - Closed System Reduces Contamination
 - Reduces Human error



Summary (2)

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



Summary (3)

- **New Solid Phase Extraction Chemistries and Methods are continuously being developed**
- **EZPFC**
 - Designed for Semi-Automated PFAS Extractions
 - SuperVap PFC Concentrator for 24 samples
- **Capable of performing in line extract drying and/or Cartridge extract clean-ups**



Come see us at booth G-2

Questions?

