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Semi-Automated Solid Phase Extraction and Analysis of Wastewater with EPA Method 625

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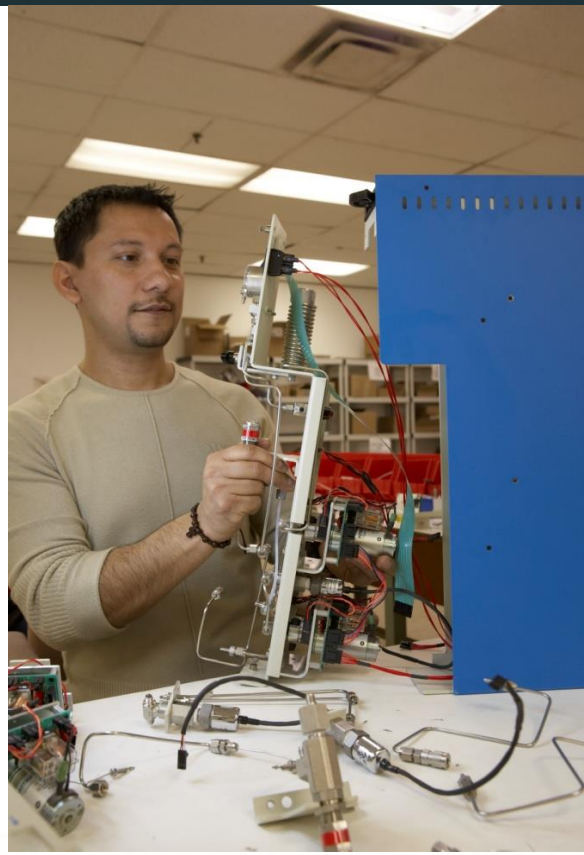
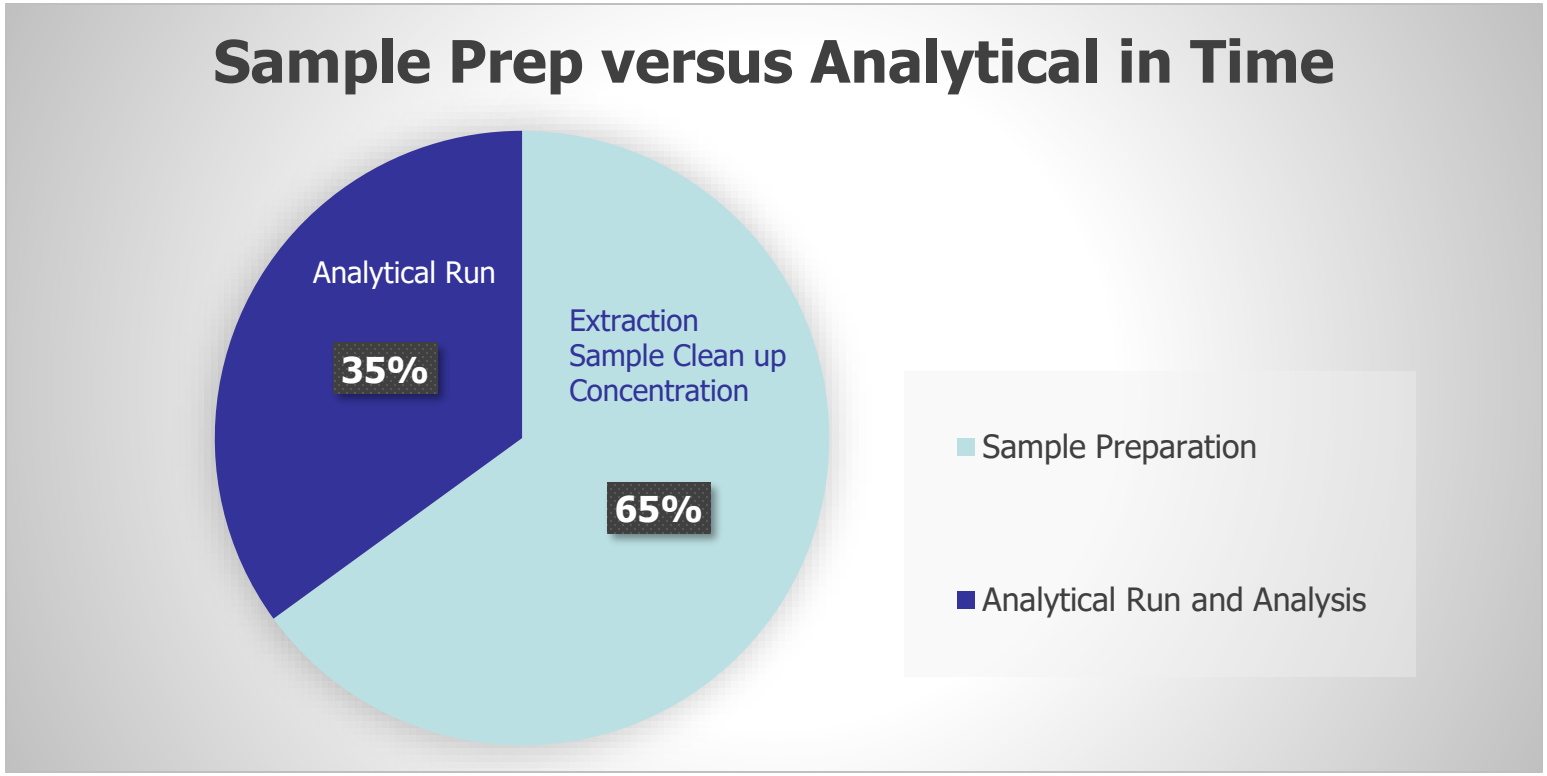
Made in the USA



Laboratory Workflow Breakdown



Solid Phase Extraction

Automated

EconoTrace®
Drinking Water

TurboTrace®
Drinking &
Waste Water

TurboTrace® ABN
EPA Methods 625
& 8270

TurboTrace® PFC
Drinking & Waste
Water

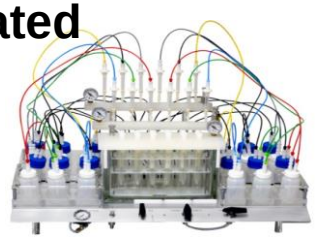
Concentration

SuperVap®
Concentration and
Evaporation

Direct to Vial

Semi-Automated

12 Position EZSPE®
Drinking Water &
Waste Water
Analysis



12 Position EZPFC
Drinking Water & Waste
Water
PFAS/PFOS/PFOA
Analysis

Overview

- **Development of a fully automated extraction system for EPA 8270/625**
  - **Full Validation package available reviewed by the US EPA**
- **Establishment of an extraction procedure capable of implementation of various aqueous matrices**
  - **Implementation of platform across other EPA methodologies**
- **Participation in ILI SPE Demonstration project for method suitability**
- **Performed Independent Validation Study of Many Waste Water Matrices**
- **Developed a Semi-Automated extraction system for EPA 8270/625 (Semi Volatiles in Water)**

Matrices

- **DI and Tap**
- **ASTM 5909 synthetic Wastewater**
- **TCLP Fluid**
- **Pond, River and Reservoir surface water**
- **Real world industrial effluents and influents**
- **Groundwater**

Comparison of LLE/CLE vs. Automated SPE Methods

LLE/CLE	Automated/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
	Fast and Unattended
	Low Solvent Usage

Fast Flow Vacuum based Sample Processing

Sample Sizes Unlimited

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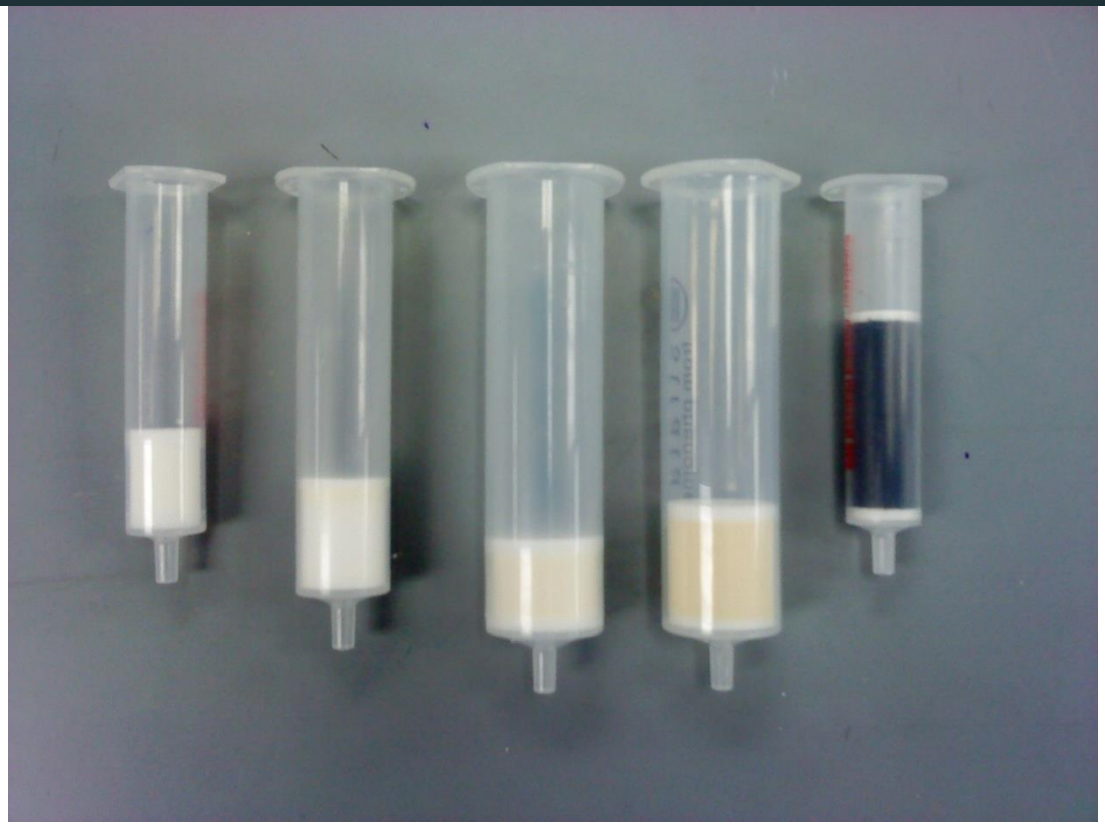
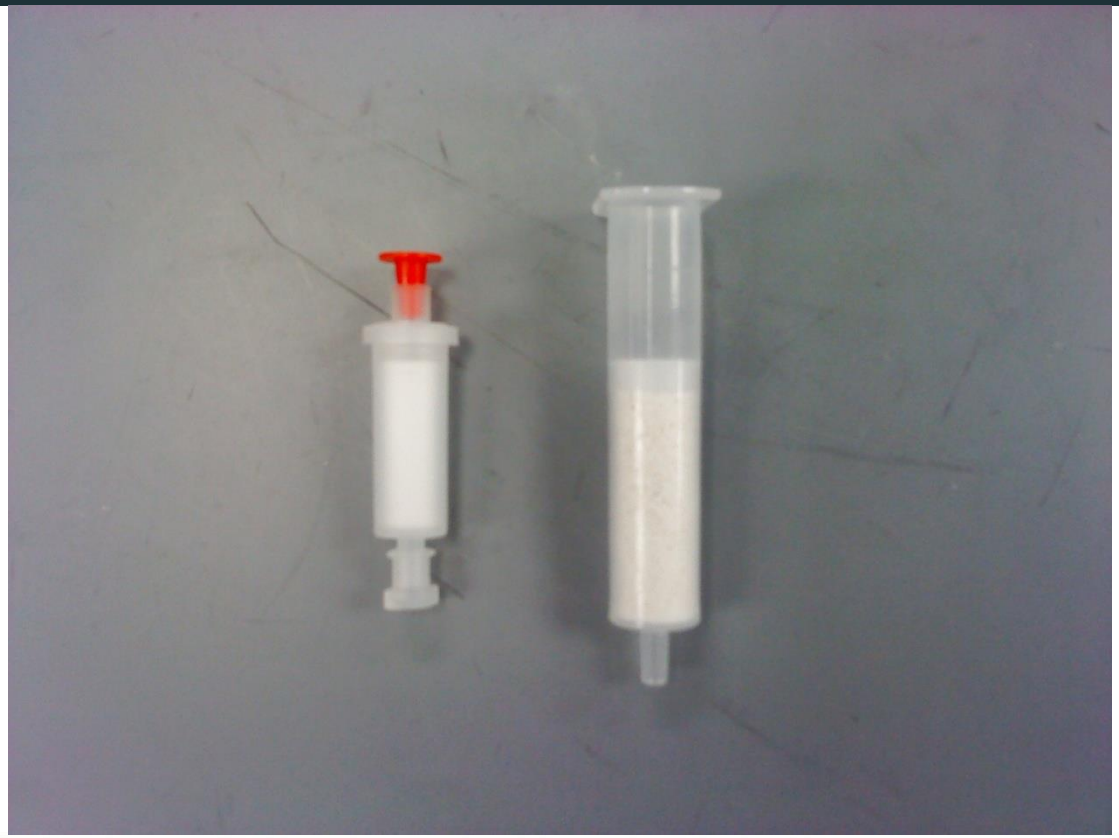
SPE Cartridges



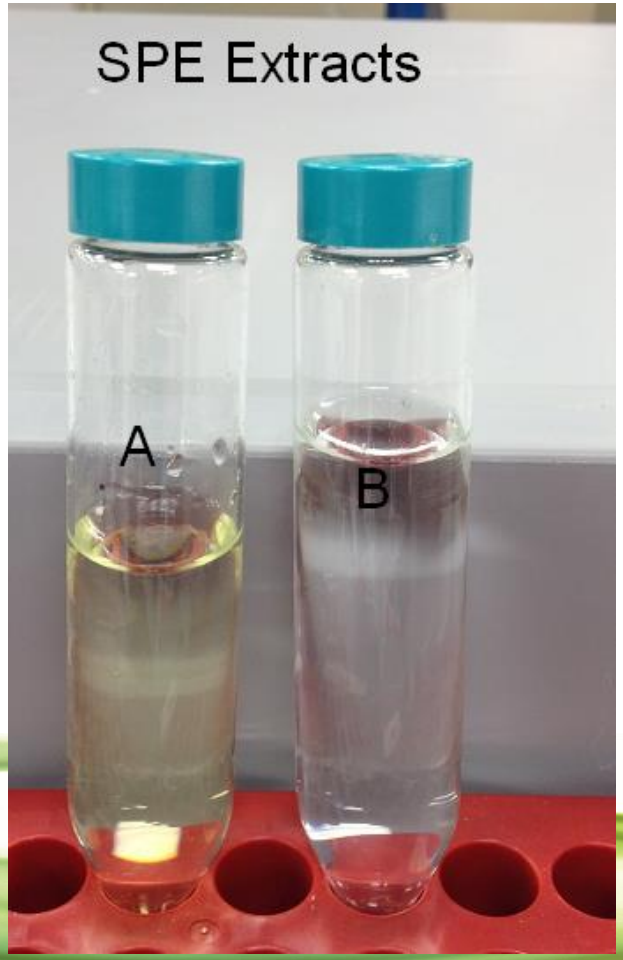
Drying Cartridges



Vacuum and Nitrogen Drying

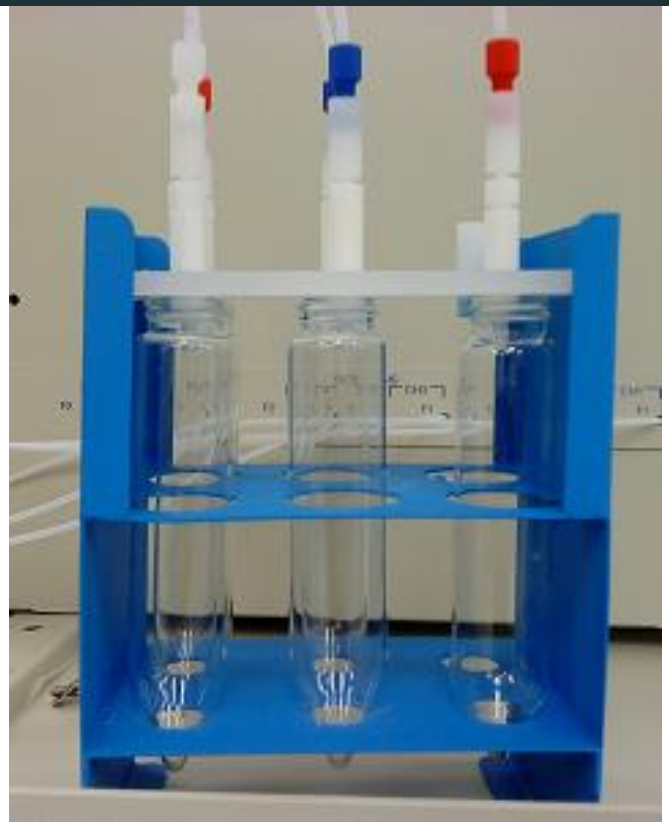
No Formation of Emulsions

No subsequent Emulsions in Extracts to deal with

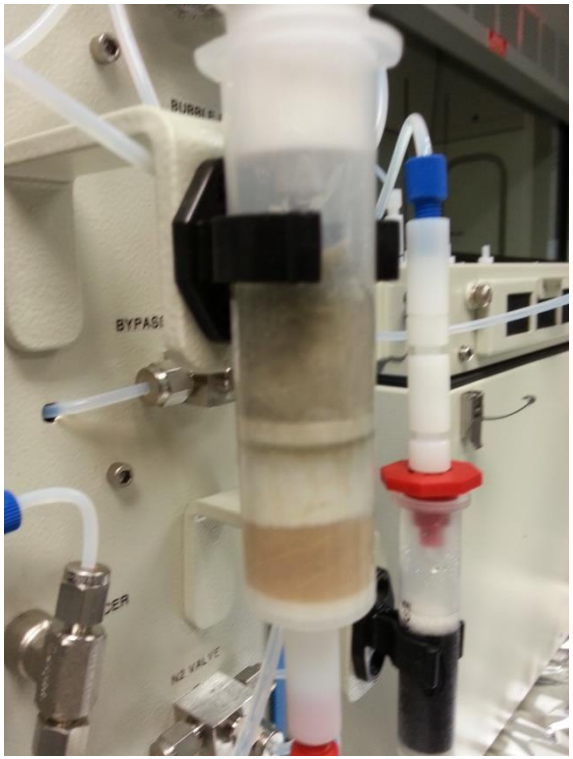


Reduced Solvent Usage

Reduced Exposure to Laboratory Background



Dual SPE Cartridge Extraction



Direct to Vial Concentration

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Direct to Vial Concentration

SuperVap Concentrator

- 12 position
- 50ml vessel

SuperVap Features

- **6 (250ml), 12 (50ml) 24 (15ml) position models for extractions.**
- **Dry bath heating element**
- **Independent secondary heater for extract nipple (Can be disabled).**
- **Sensor controlled**
- **Savable temperature log**

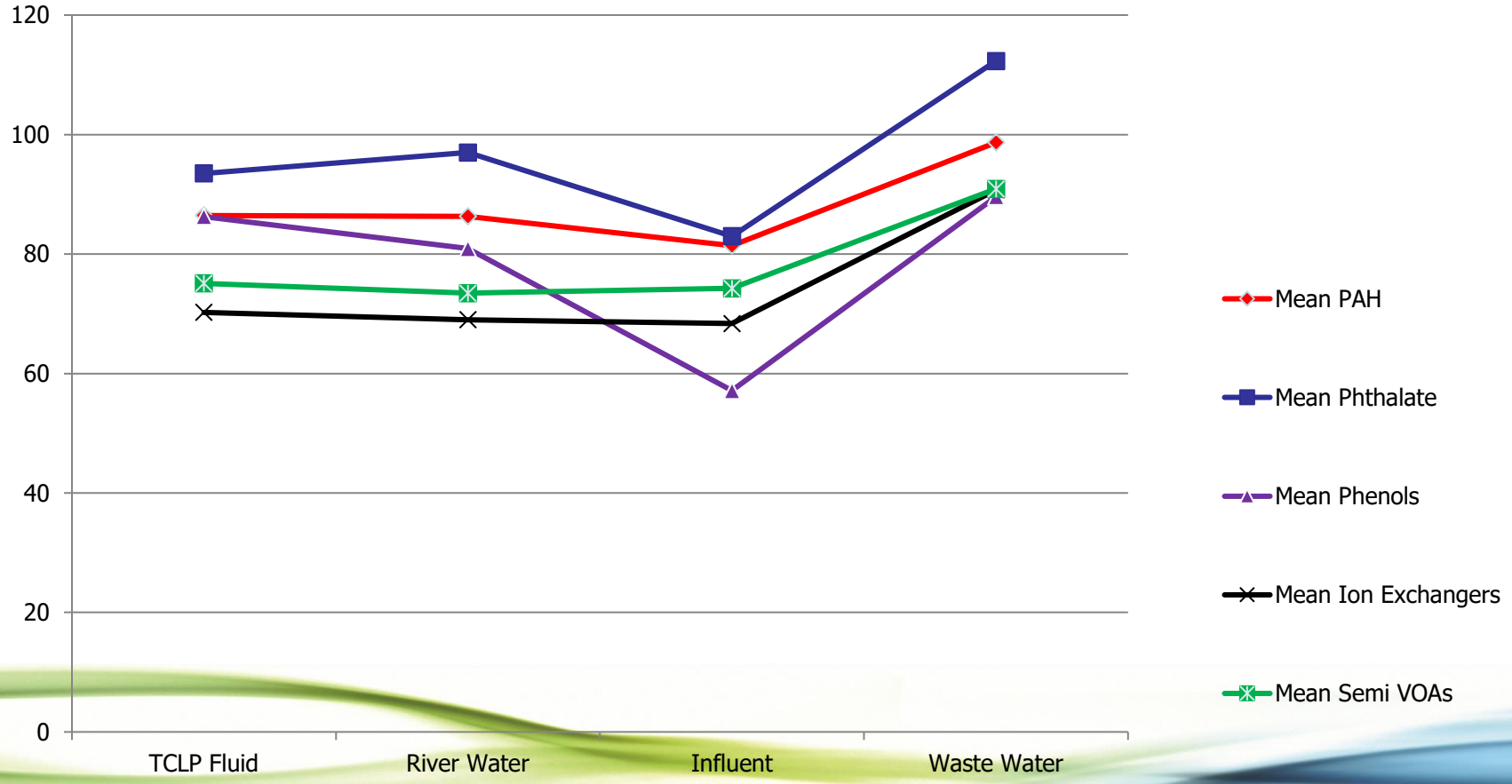
SPE Procedure (1)

- 1 L water samples, pH < 2, spike with standards
- Condition with DCM, MeOH, water
- Cartridges: mixed bed and coconut charcoal
- Load samples across cartridges under vacuum and dry
- Bottle rinse, elute DCM across mixed bed and collect (Fraction # 1)
- Recondition mixed bed (MeOH); 1% NaOH across both cartridges

SPE Procedure (2)

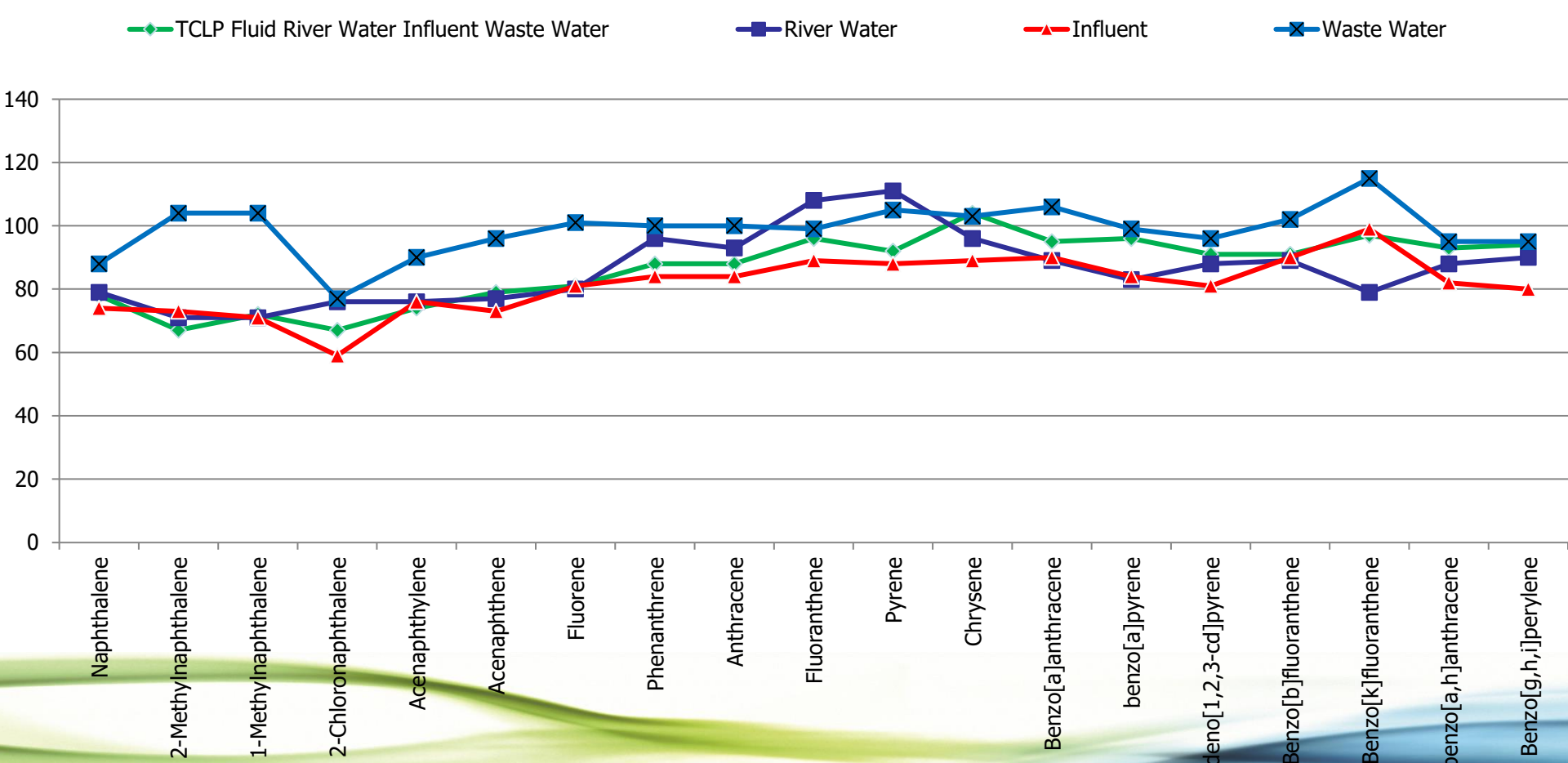
- **Dry, elute both cartridges sequentially with DCM (Fraction # 2)**
- **Purge system with nitrogen to collect any analytes**
- **Dry Fraction with sodium sulfate**
- **Evaporation and low res GC/MS**

Recoveries by Analyte Class

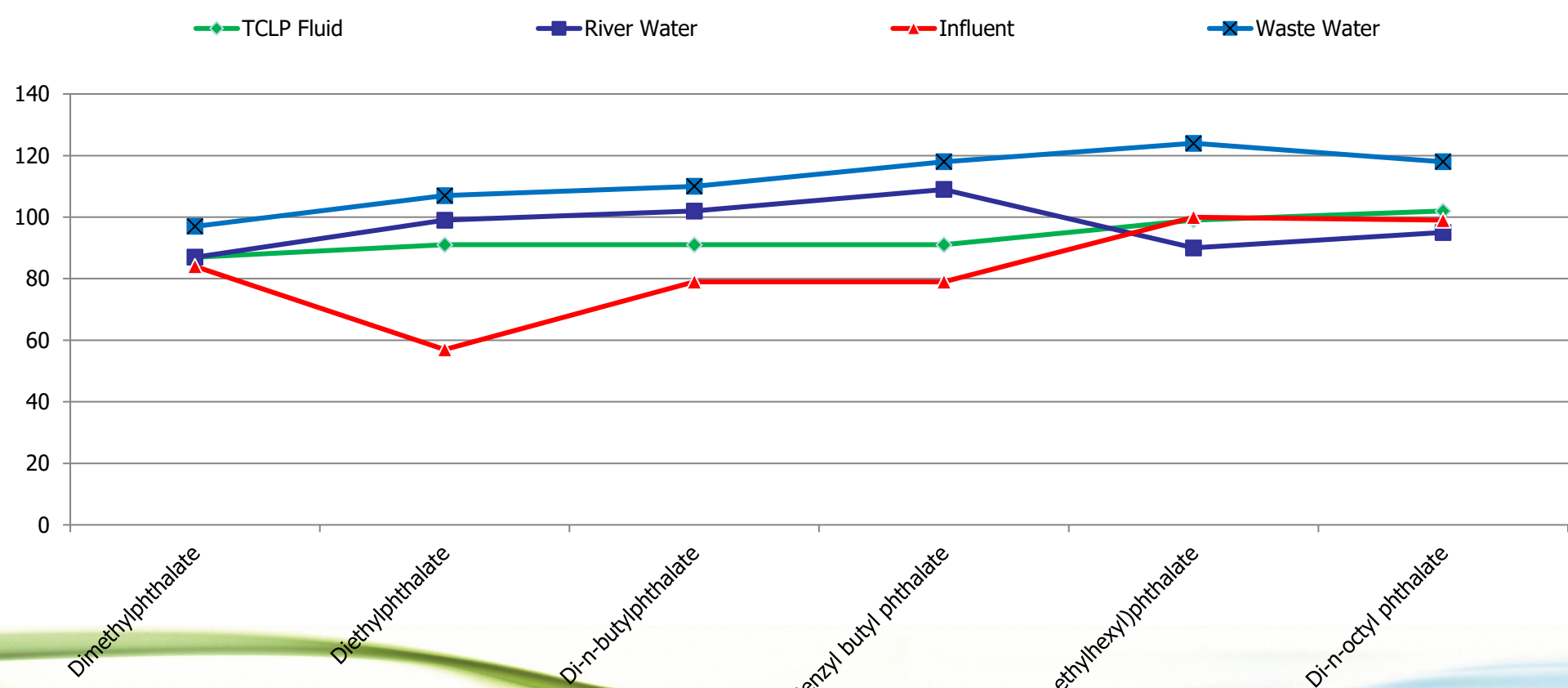


Recoveries for Individual Compounds with specific Analyte Classes

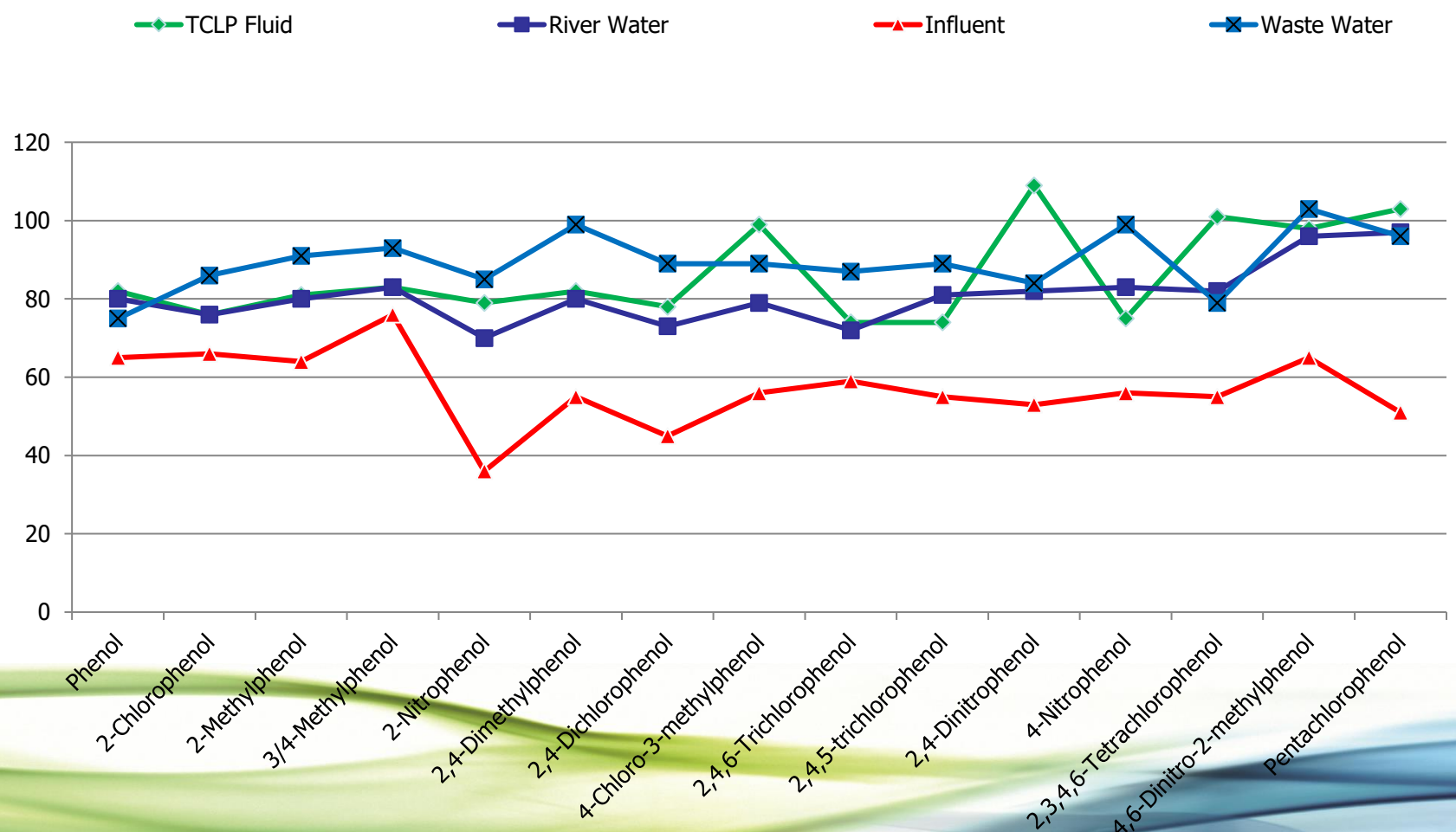
PAHs


TCLP Fluid River Water Influent Waste Water
River Water
Influent
Waste Water
140
120
100
80
60
40
20
0
Naphthalene
2-Methylnaphthalene
1-Methylnaphthalene
2-Chloronaphthalene
Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene
Chrysene
Benzo[a]anthracene
benzo[a]pyrene
Indeno[1,2,3-cd]pyrene
Benzo[b]fluoranthene
Benzo[k]fluoranthene
Dibenzo[a,h]anthracene
Benzo[g,h,i]perylene

Phthalates


TCLP Fluid
River Water
Influent
Waste Water
140
120
100
80
60
40
20
0
Dimethylphthalate
Diethylphthalate
Di-n-butylphthalate
Benzyl butyl phthalate
Bis(2-ethylhexyl)phthalate
Di-n-octyl phthalate

Phenols


TCLP Fluid
River Water
Influent
Waste Water
120
100
80
60
40
20
0
Phenol
2-Chlorophenol
2-Methylphenol
3/4-Methylphenol
2-Nitrophenol
2,4-Dimethylphenol
2,4-Dichlorophenol
4-Chloro-3-methylphenol
2,4,6-Trichlorophenol
2,4,5-trichlorophenol
2,4-Dinitrophenol
4-Nitrophenol
2,3,4,6-Tetrachlorophenol
4,6-Dinitro-2-methylphenol
Pentachlorophenol

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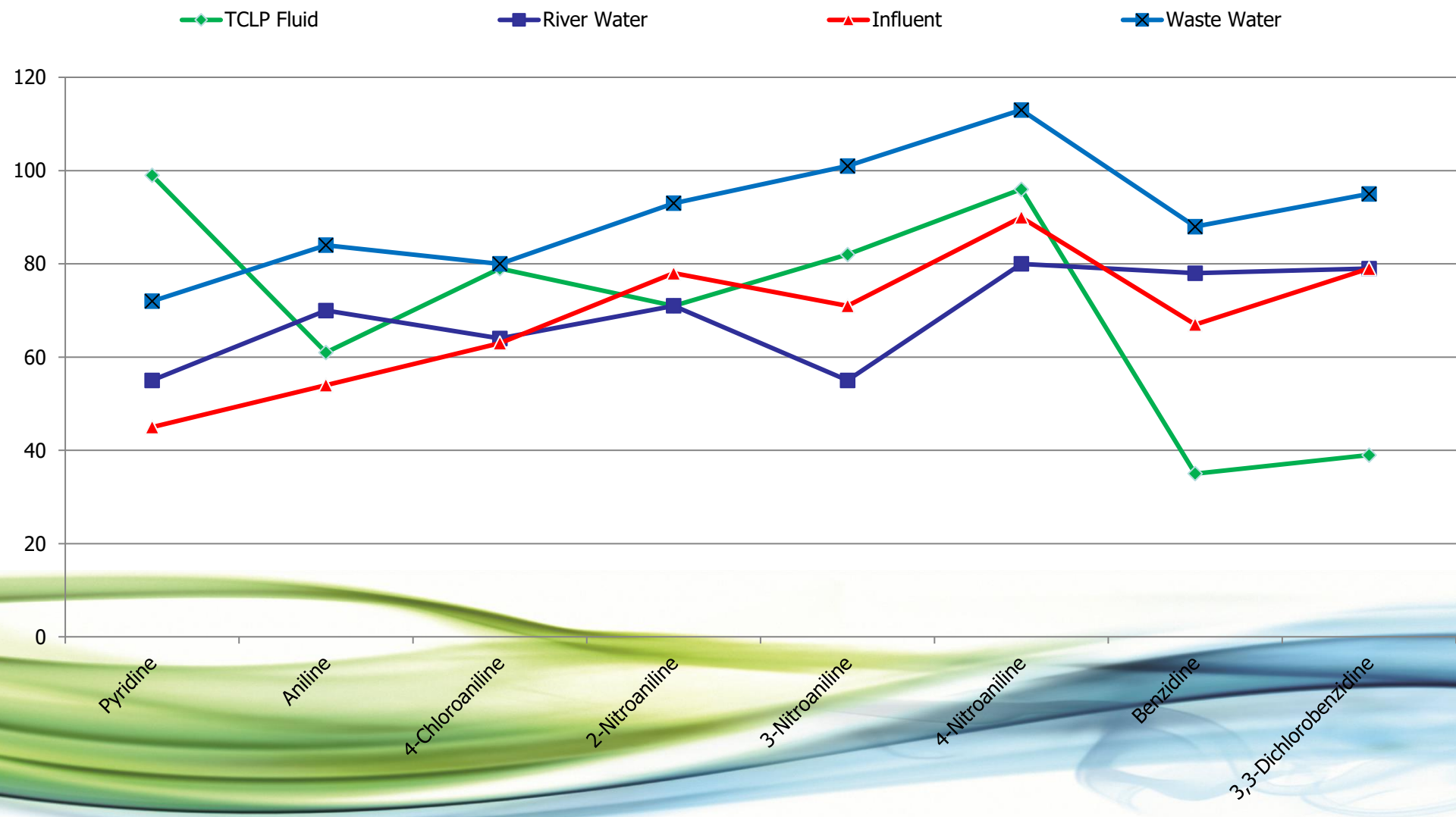
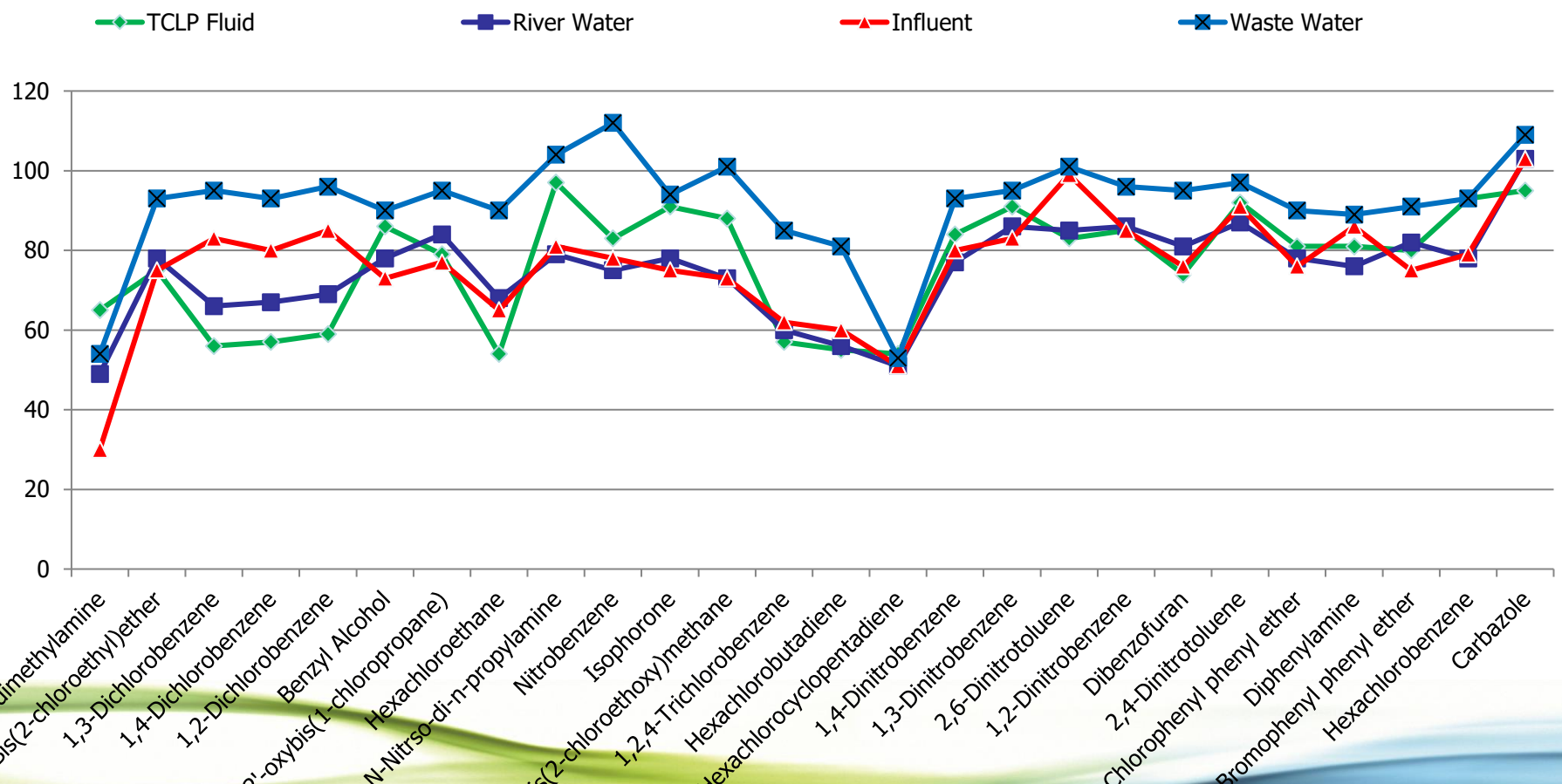
Ion Exchangers

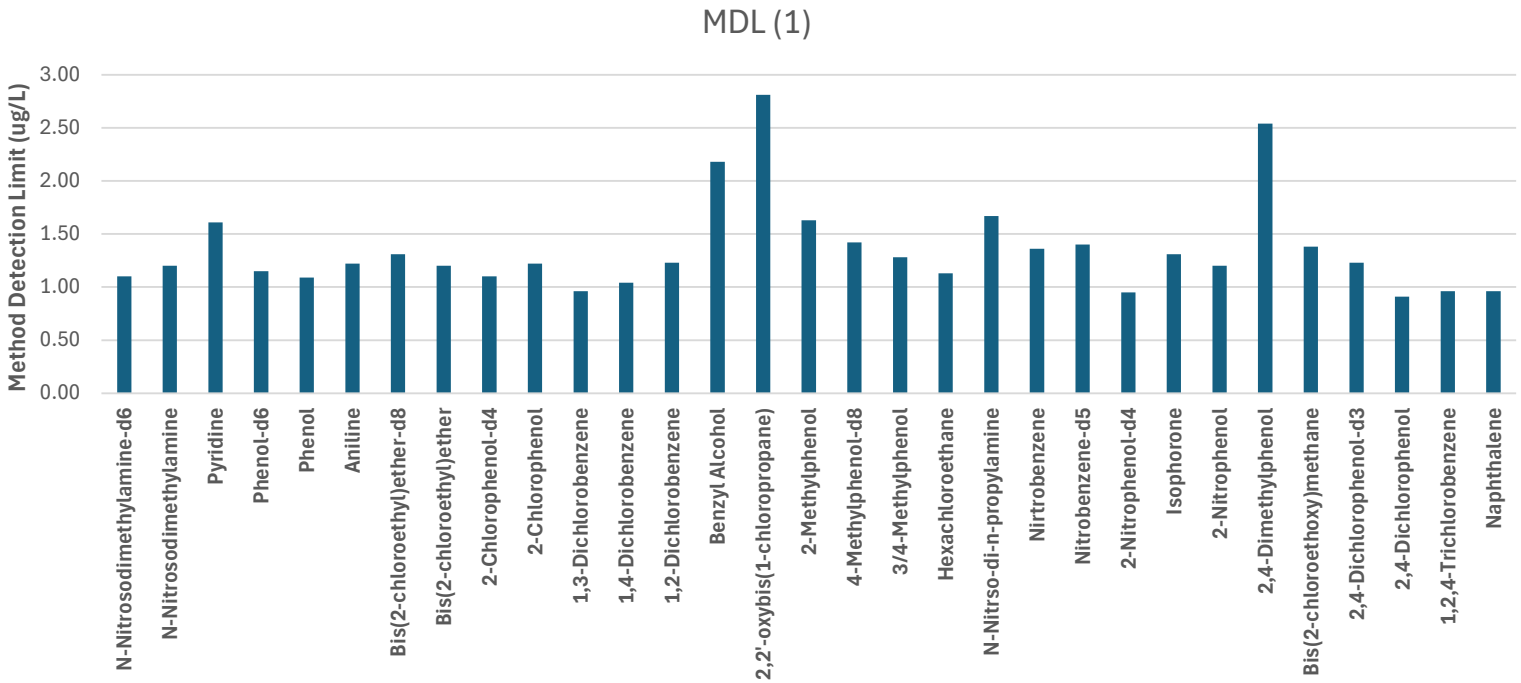


Semi-Volatiles

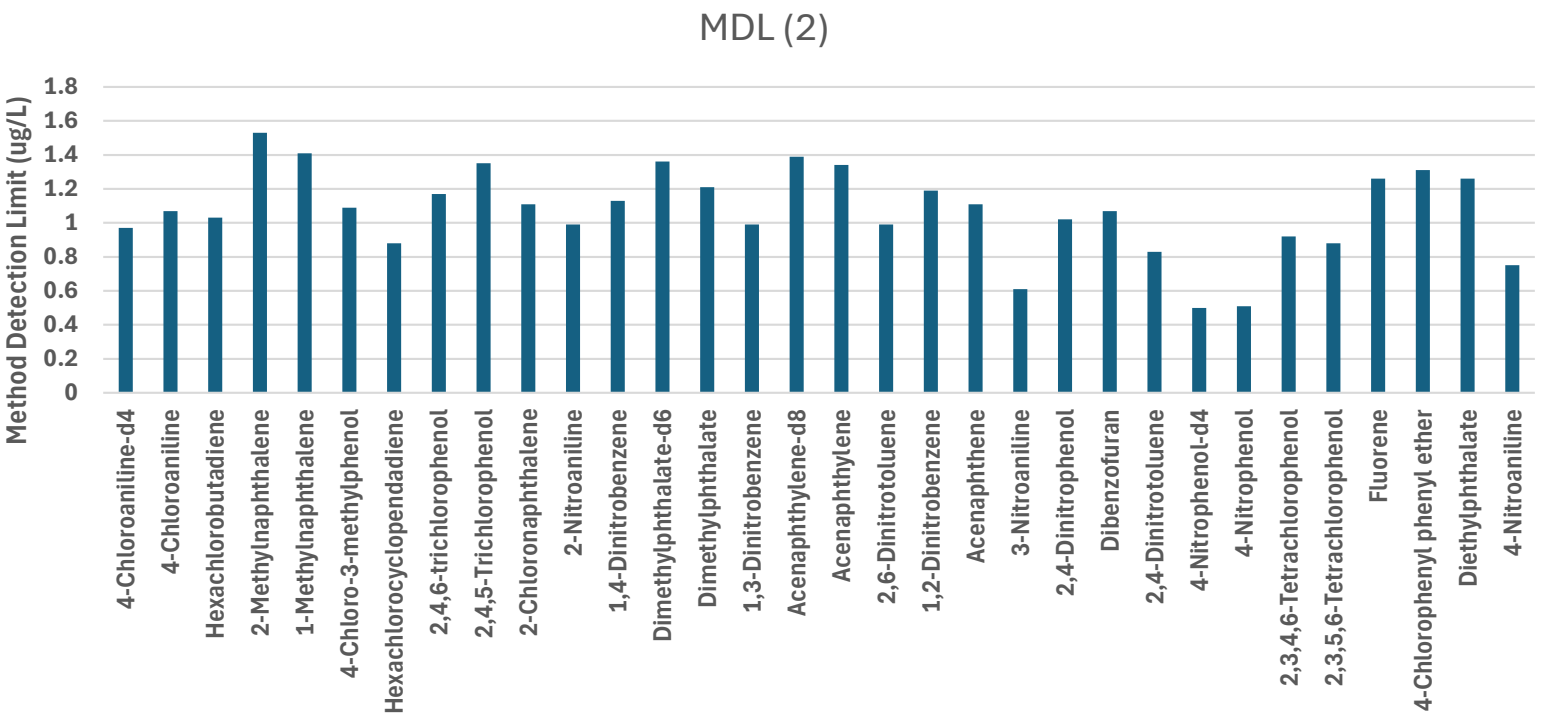


Method Detection Limit

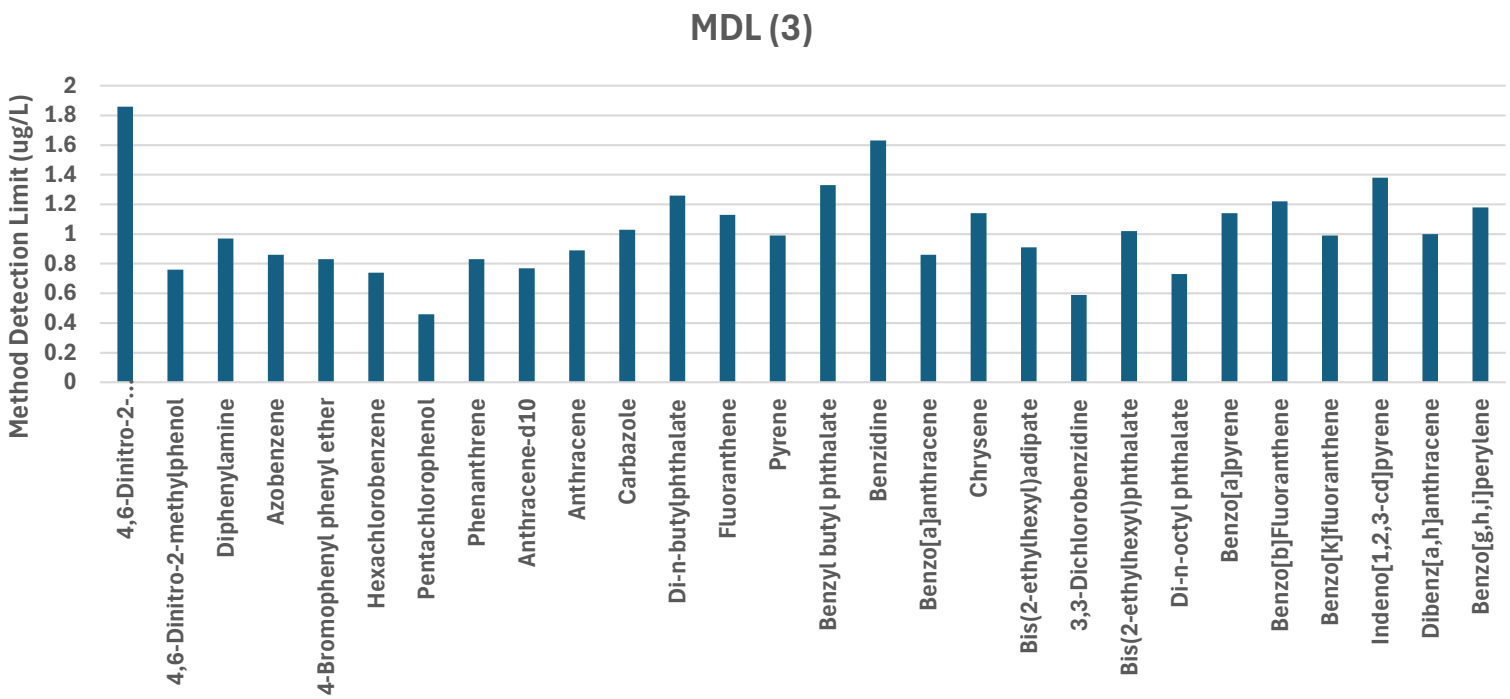
MDL (1)


MDL (1)
Method Detection Limit (ug/L)
3.00
2.50
2.00
1.50
1.00
0.50
0.00
N-Nitrosodimethylamine-d6
N-Nitrosodimethylamine
Pyridine
Phenol-d6
Phenol
Aniline
Bis(2-chloroethyl)ether-d8
Bis(2-chloroethyl)ether
2-Chlorophenol-d4
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
Benzyl Alcohol
2,2'-oxybis(1-chloropropane)
2-Methylphenol
4-Methylphenol-d8
3/4-Methylphenol
Hexachloroethane
N-Nitrso-di-n-propylamine
Nirtrobenzene
Nitrobenzene-d5
2-Nitrophenol-d4
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
Bis(2-chloroethoxy)methane
2,4-Dichlorophenol-d3
2,4-Dichlorophenol
1,2,4-Trichlorobenzene
Naphthalene

MDL (2)


MDL (2)
Method Detection Limit (ug/L)
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
4-Chloroaniline-d4
4-Chloroaniline
Hexachlorobutadiene
2-Methylnaphthalene
1-Methylnaphthalene
4-Chloro-3-methylphenol
Hexachlorocyclopendadiene
2,4,6-trichlorophenol
2,4,5-Trichlorophenol
2-Chloronaphthalene
2-Nitroaniline
1,4-Dinitrobenzene
Dimethylphthalate-d6
Dimethylphthalate
1,3-Dinitrobenzene
Acenaphthylene-d8
Acenaphthylene
2,6-Dinitrotoluene
1,2-Dinitrobenzene
Acenaphthene
3-Nitroaniline
2,4-Dinitrophenol
Dibenzofuran
2,4-Dinitrotoluene
4-Nitrophenol-d4
4-Nitrophenol
2,3,4,6-Tetrachlorophenol
2,3,5,6-Tetrachlorophenol
Fluorene
4-Chlorophenyl phenyl ether
Diethylphthalate
4-Nitroaniline

MDL (3)


MDL (3)
Method Detection Limit (ug/L)
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
4,6-Dinitro-2-...
4,6-Dinitro-2-methylphenol
Diphenylamine
Azobenzene
4-Bromophenyl phenyl ether
Hexachlorobenzene
Pentachlorophenol
Phenanthrene
Anthracene-d10
Anthracene
Carbazole
Di-n-butylphthalate
Fluoranthene
Pyrene
Benzyl butyl phthalate
Benzidine
Benzo[a]anthracene
Chrysene
Bis(2-ethylhexyl)adipate
3,3-Dichlorobenzidine
Bis(2-ethylhexyl)phthalate
Di-n-octyl phthalate
Benzo[a]pyrene
Benzo[b]Fluoranthene
Benzo[k]fluoranthene
Indeno[1,2,3-cd]pyrene
Dibenz[a,h]anthracene
Benzo[g,h,i]perylene

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 six samples simultaneously
  - Vacuum for fast loading of large volume samples
  - Unattended Sample loading walkaway time
- **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

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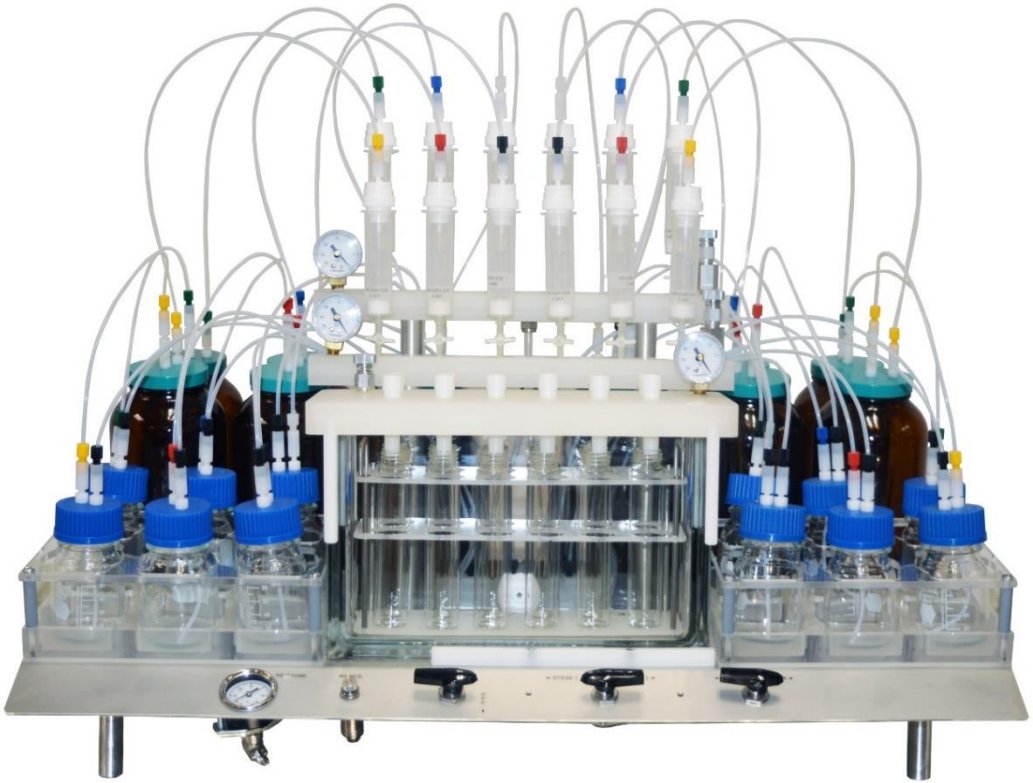
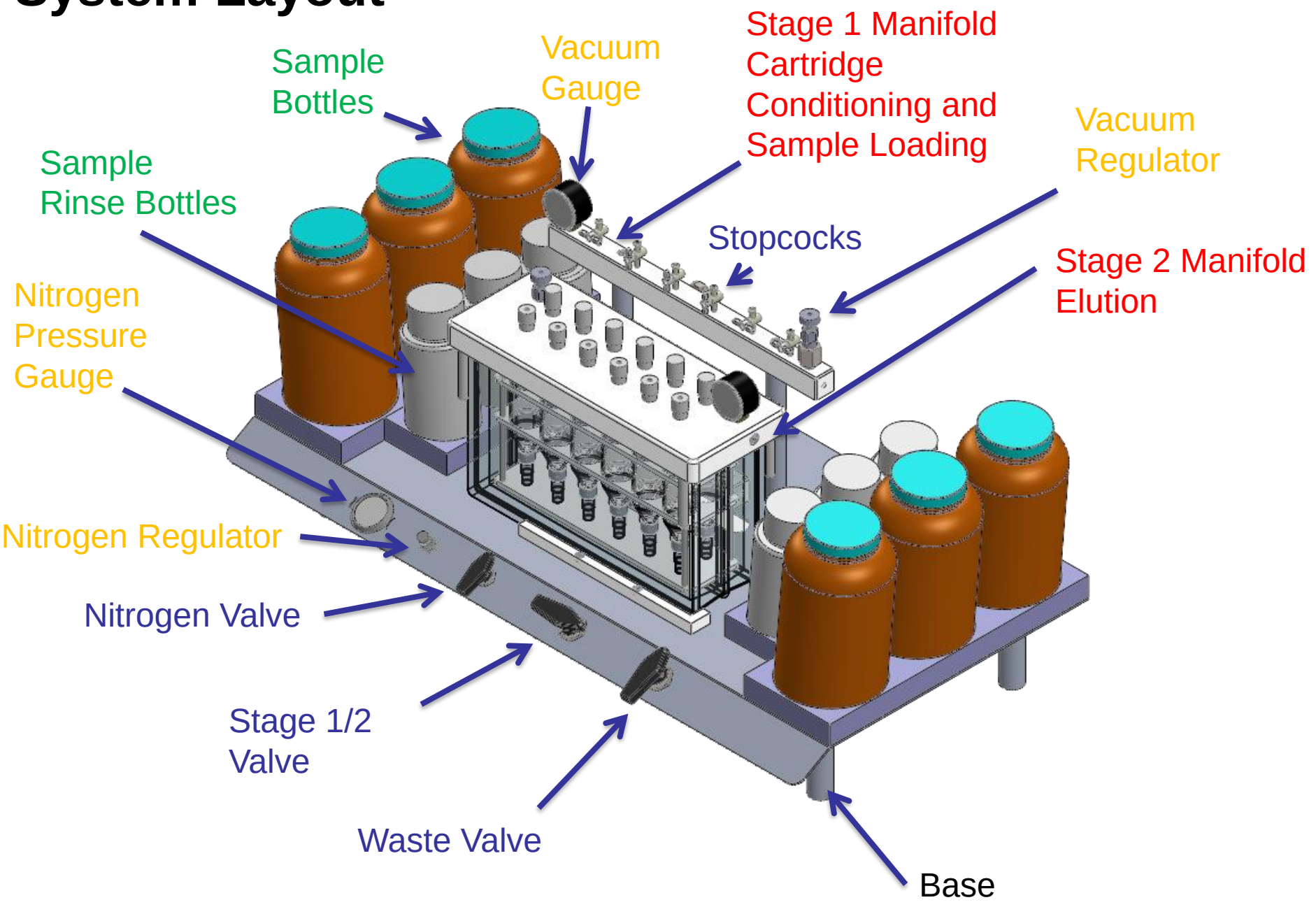
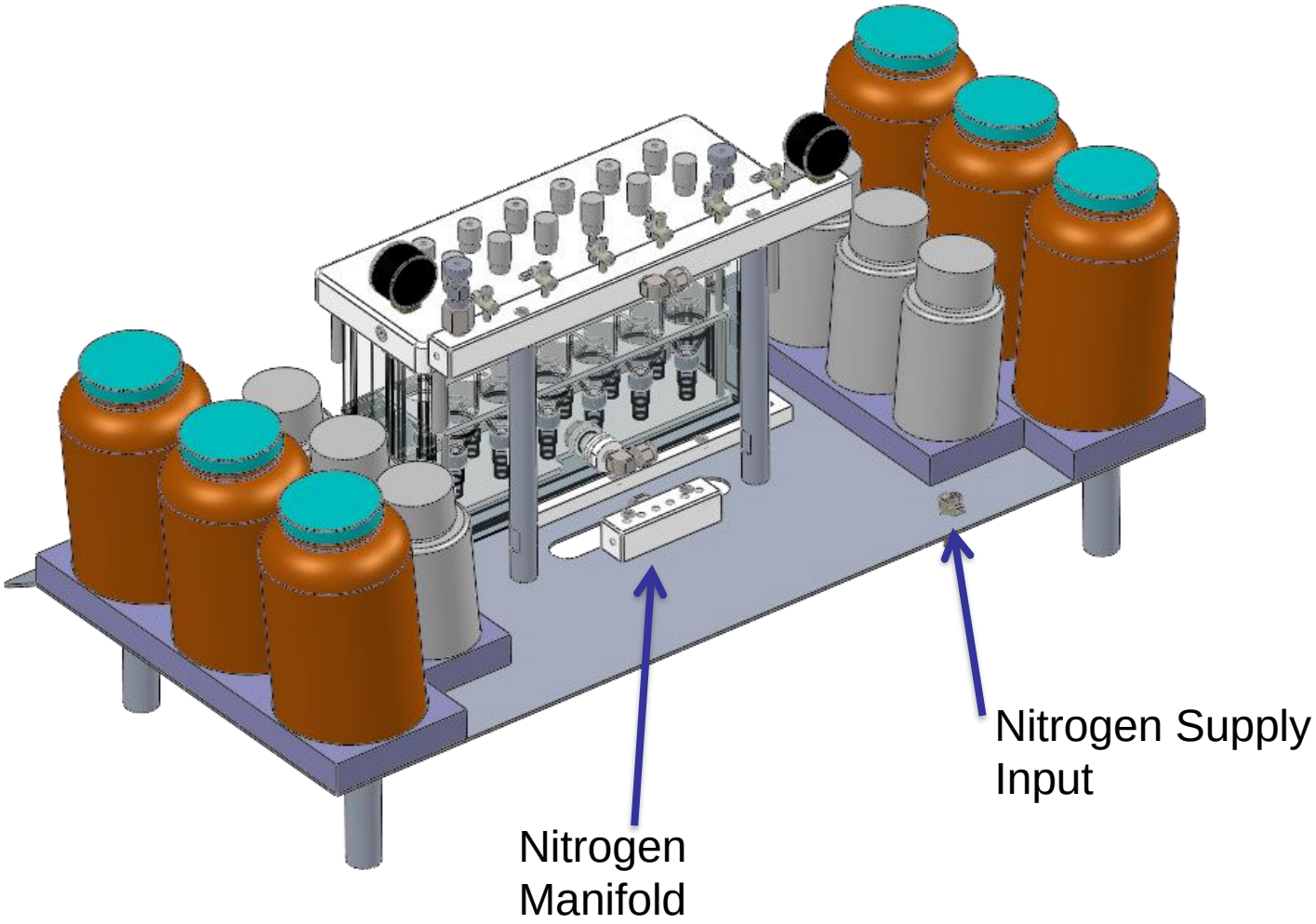
EZSpe®



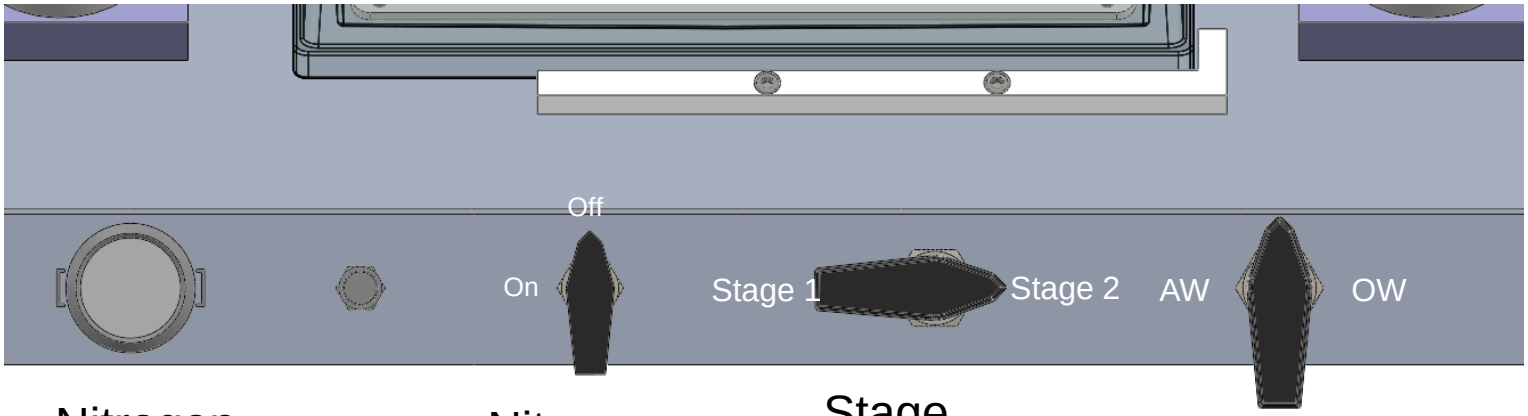
EZSpe 12 sample

System Layout




Nitrogen Supply Input
Nitrogen Manifold

Control Valve Layout



Nitrogen Gauge

Nitrogen Valve

Stage 1/2 Valve

Waste Valve

Summary

- **Fully Automated Semi-Volatile extractions for various compound classes**
- **Excellent recoveries**
- **Method validated (five year US-based study), can be used internationally**
- **Can use many brands of cartridges**
- **Excellent MDL results**

Summary

- **Solid Phase Extraction is a well accepted technology**
- **New Solid Phase Extraction Chemistries and Sorbents are being developed**
- **Drinking Water and Waste Water Extractions**
  - **625/8270**
  - **608**
  - **Validation data package is available**
- **Capable of performing in-line extract drying and/or Cartridge extract clean-ups**
- **Reduce Solvent, Labor, and Time**

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

- **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, and precise delivery of samples and solvents**

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Questions?