Advancements in high matrix neutralization SPE automation for preparation of surface, ground, and wastewater samples.

2025 NEMC

Biotage®

Outline

Advancements in High Matrix Neutralization SPE Automation

- Automated SPE processing for Surface, Ground, & Wastewater samples
- Novel SPE Format for High Matrix Neutralization
- Sample Processing Performance & Data Quality SVOC Analysis
- Final Conclusions

Sample Bottle Adaption
Processing Surface, Ground, & Wastewater

33x400mm
Thread

Water **I**nlet **V**alve
WIV

- Glass Bottles
- VOA Vials
- Plastic Bottles

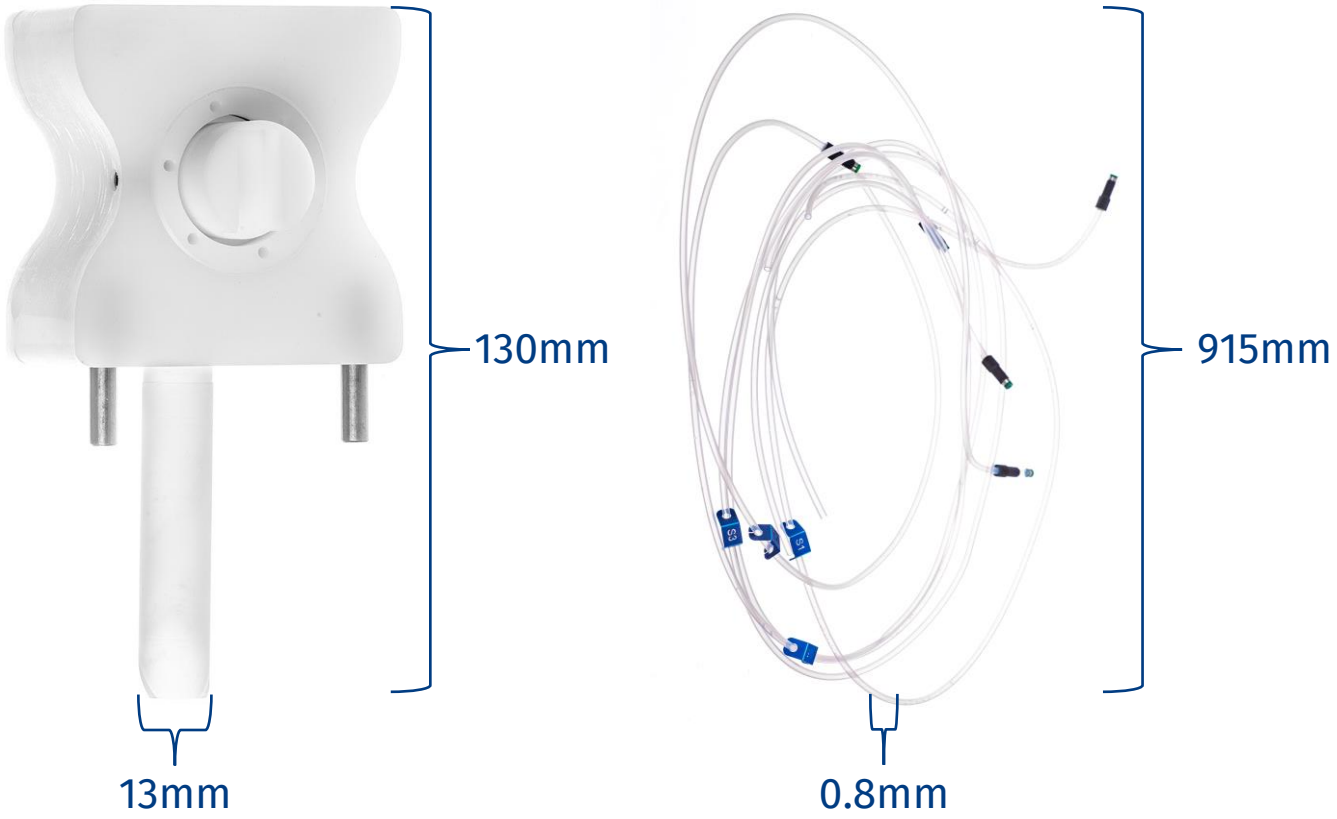
1 Liter
(33x400mm)

1 Liter
(89x400mm)

250 mL
(58x400mm)

WIV Sample Processing
Streamlined Fluid Path

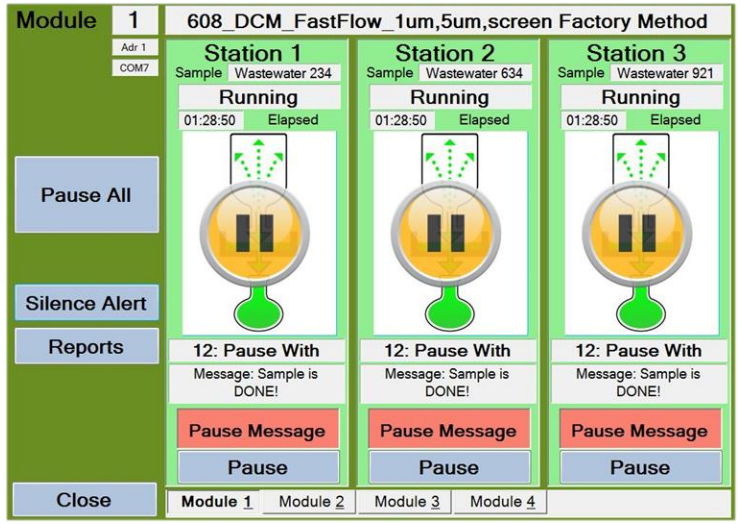
Water Inlet Valve (WIV) **VS** Sample Tubing Systems

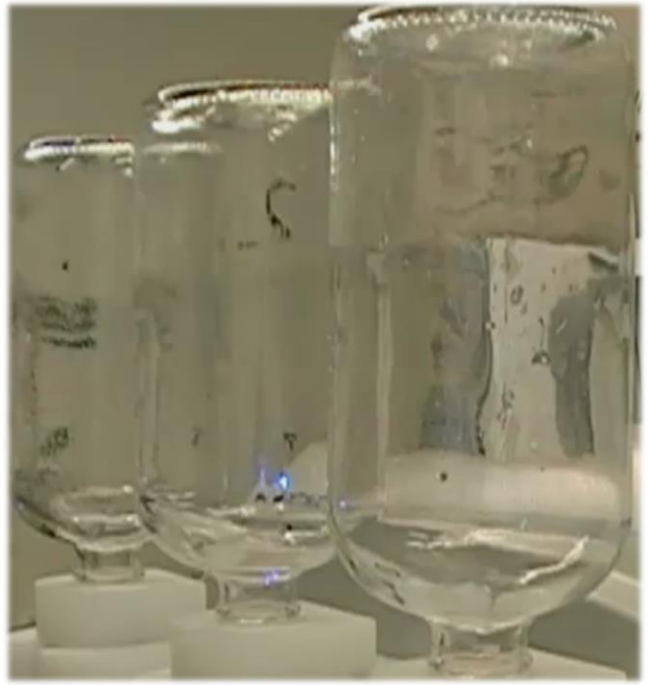


- Short Fluid Path
  - **~7x shorter** than tubing systems
- Wide Fluid Path
  - **~16x wider** than tubing systems

WIV Sample Processing
Extracting the Entire Sample

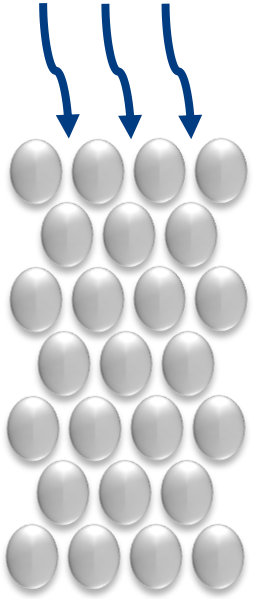
- **Direct** Load of Entire Sample
- **Powerful** Bottle Rinsing
- **Quick & Easy** Cleaning



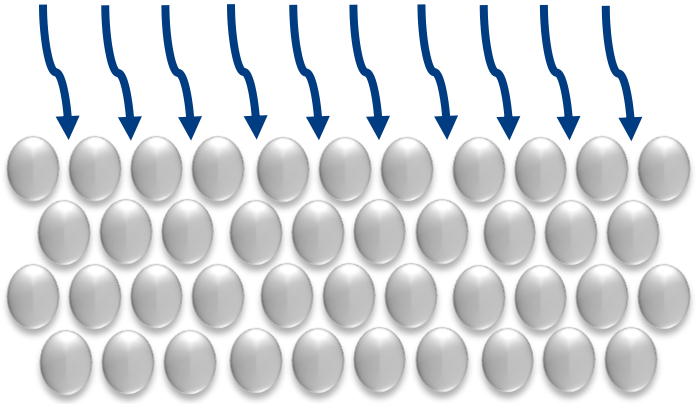


SPE Formats
Sample Volume Consideration

- Determined by Sample Volume
  - **Cartridge Volumes:** ≤ 250mL
    - Load Rate: 5 - 30mL/min
  - **Disk Volumes:** 100mL – 4 L
    - Load Rate: 30 – 500mL/min



Cartridge

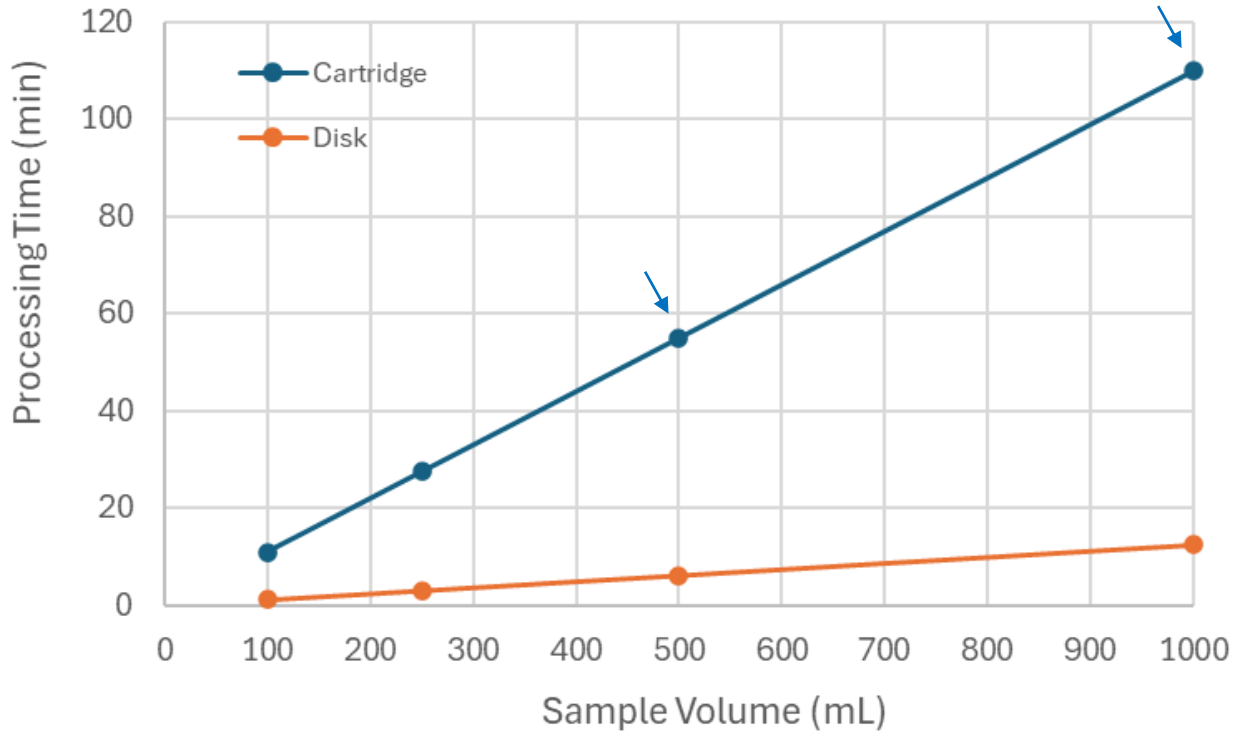


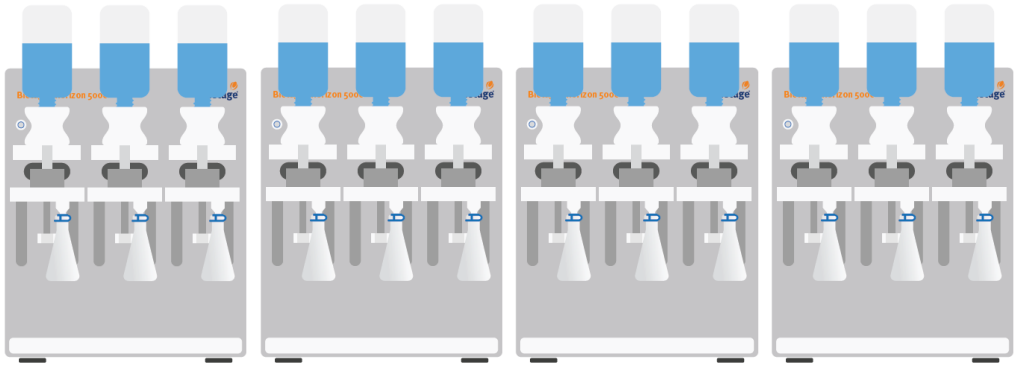
Disk

Samples can be loaded
~10x Faster on SPE disks

SPE Formats
Sample Volume Consideration

- Determined by Sample Volume



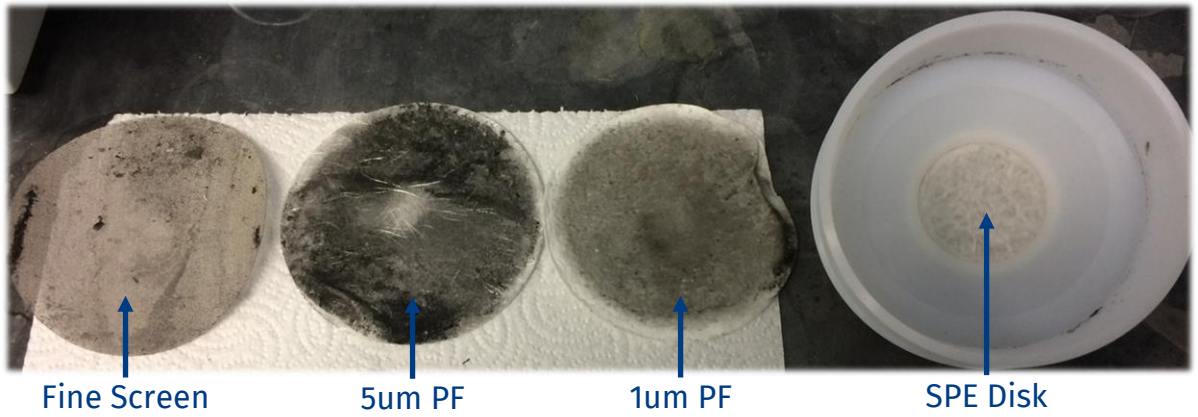


1L Sample Load Time*	Cartridge (Hours)	Disk (Hours)
12 Samples	1.9	0.3
24 Samples	3.9	0.6
48 Samples	7.8	1.3
96 Samples	15.6	2.6

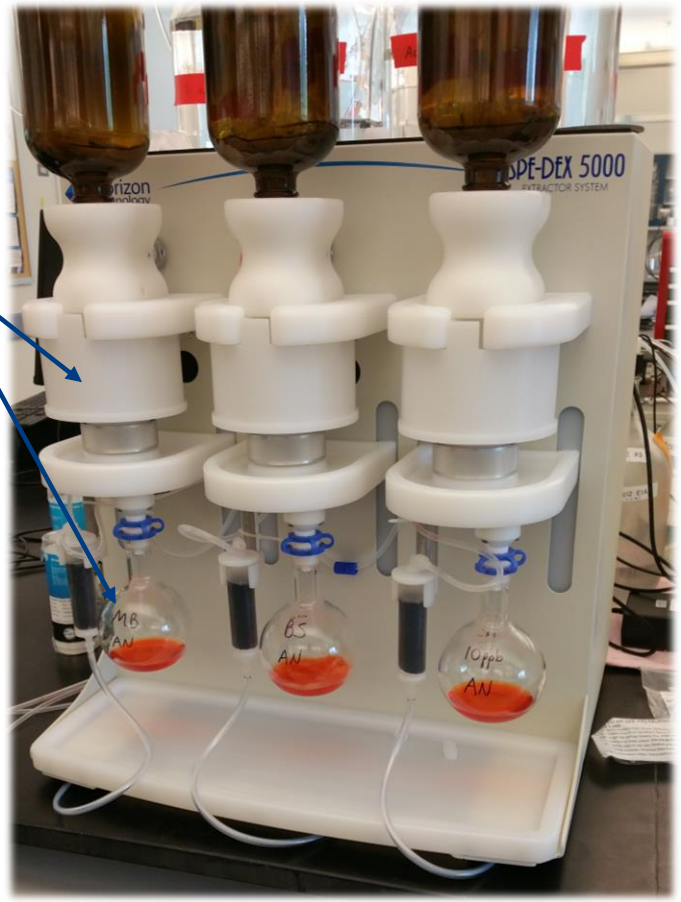
***Note:** Estimates for sample load time only. Additional SPE steps not included

SPE Formats
Sample Type Considerations

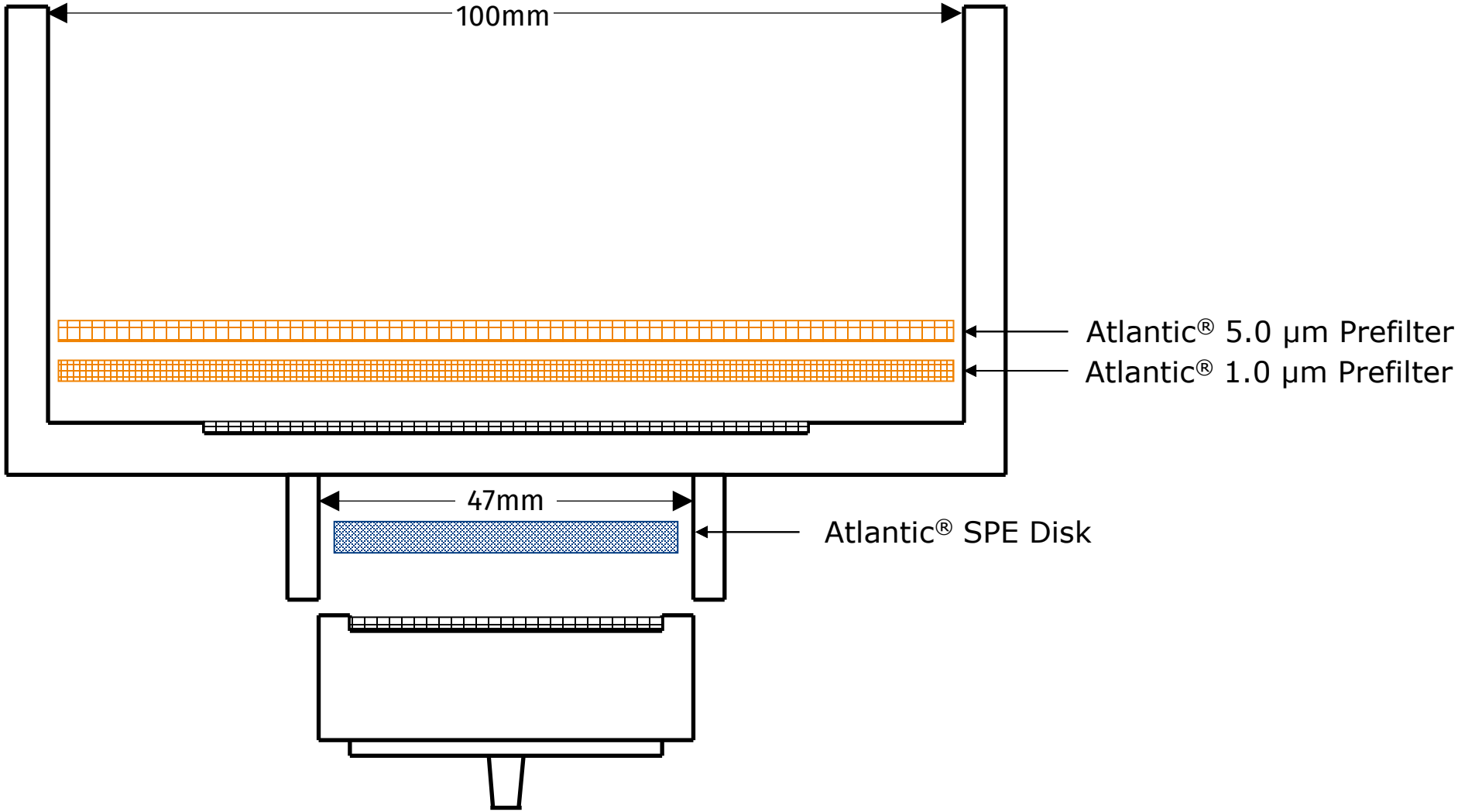
- Determined by Sample Type
  - **Cartridge:** Low Particulate
    - Drinking Water
  - **Disk:** Low & High Particulate
    - Drinking, Ground, Surface, & Wastewater



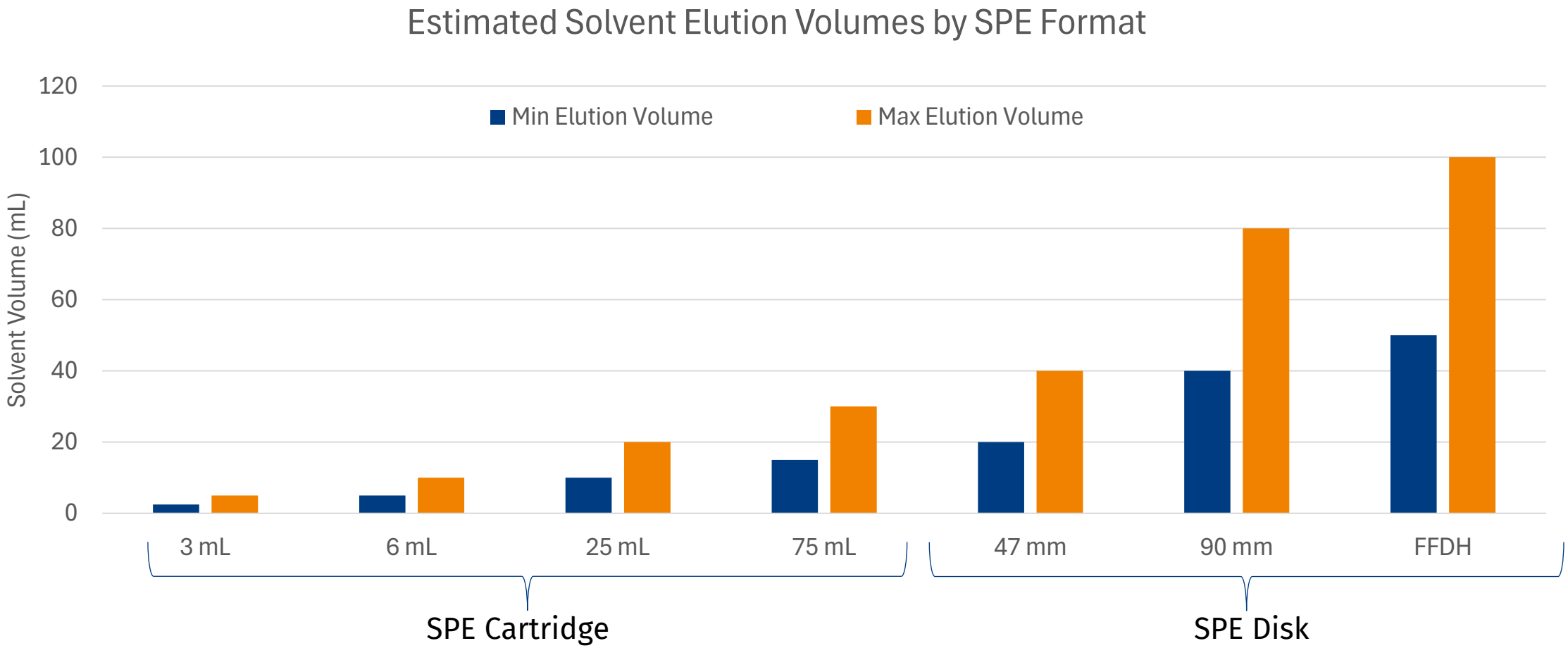
Fast Flow Disk Holder (FFDH)



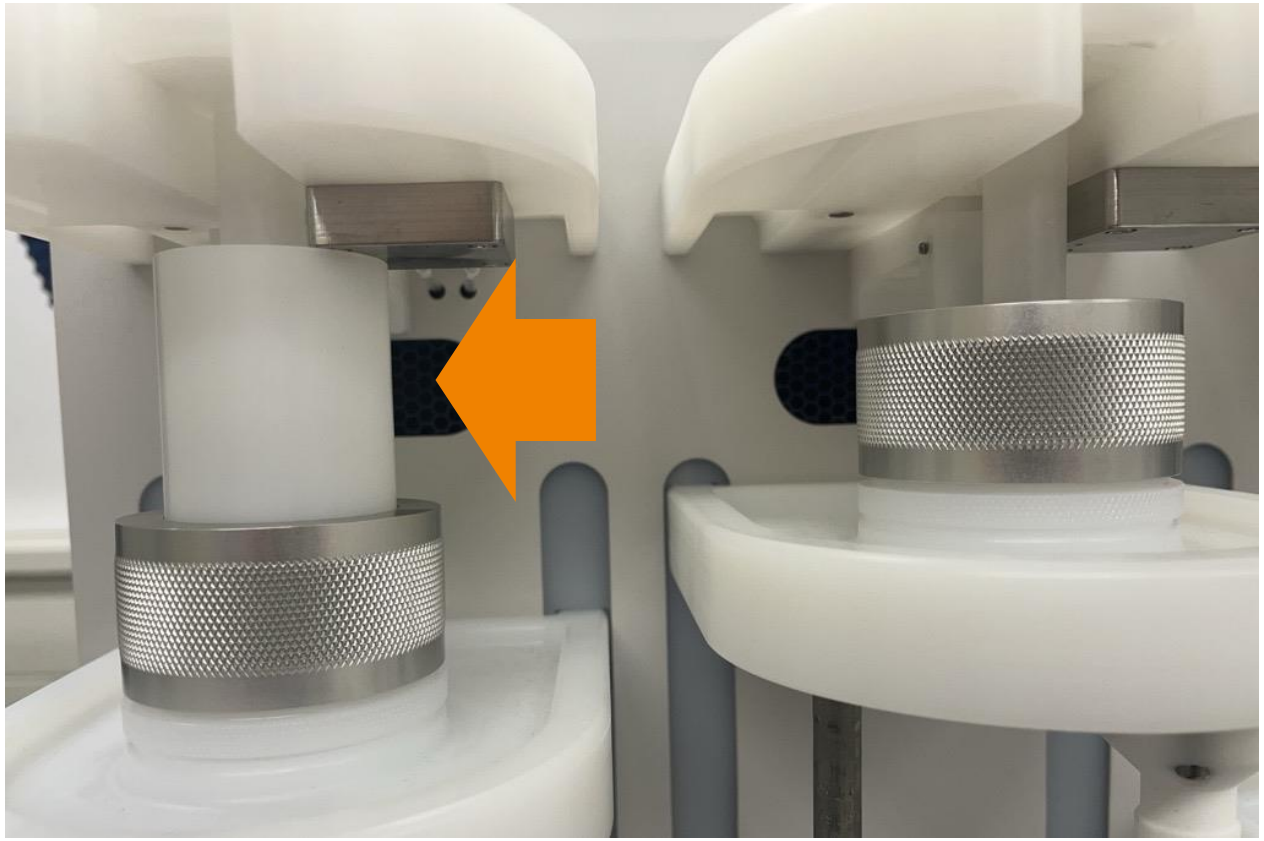
Fast Flow Disk Holder
SPE & Filtration Components



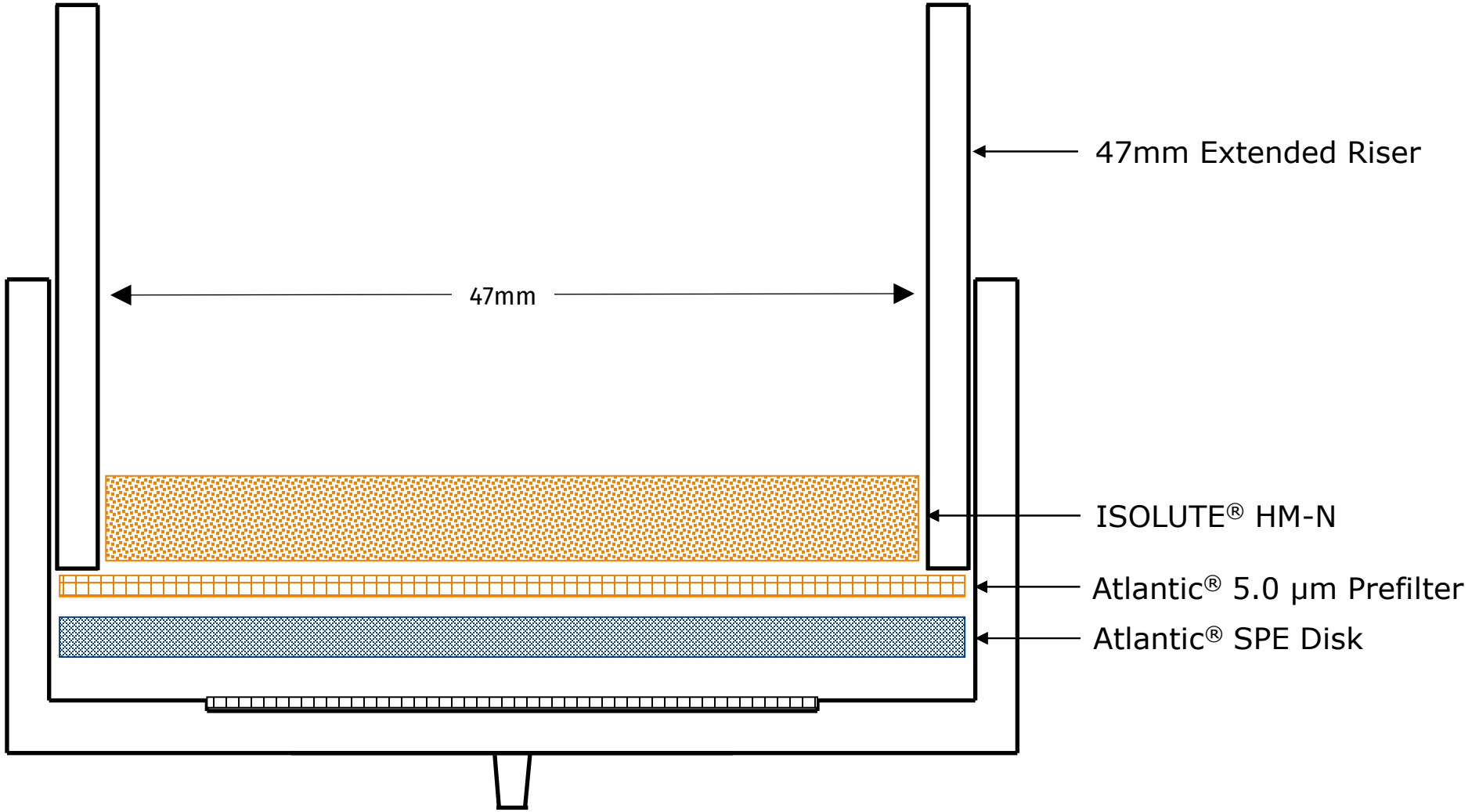
SPE Formats
Downside of Disk Based SPE



Novel SPE Format
47mm Extended Disk Holder



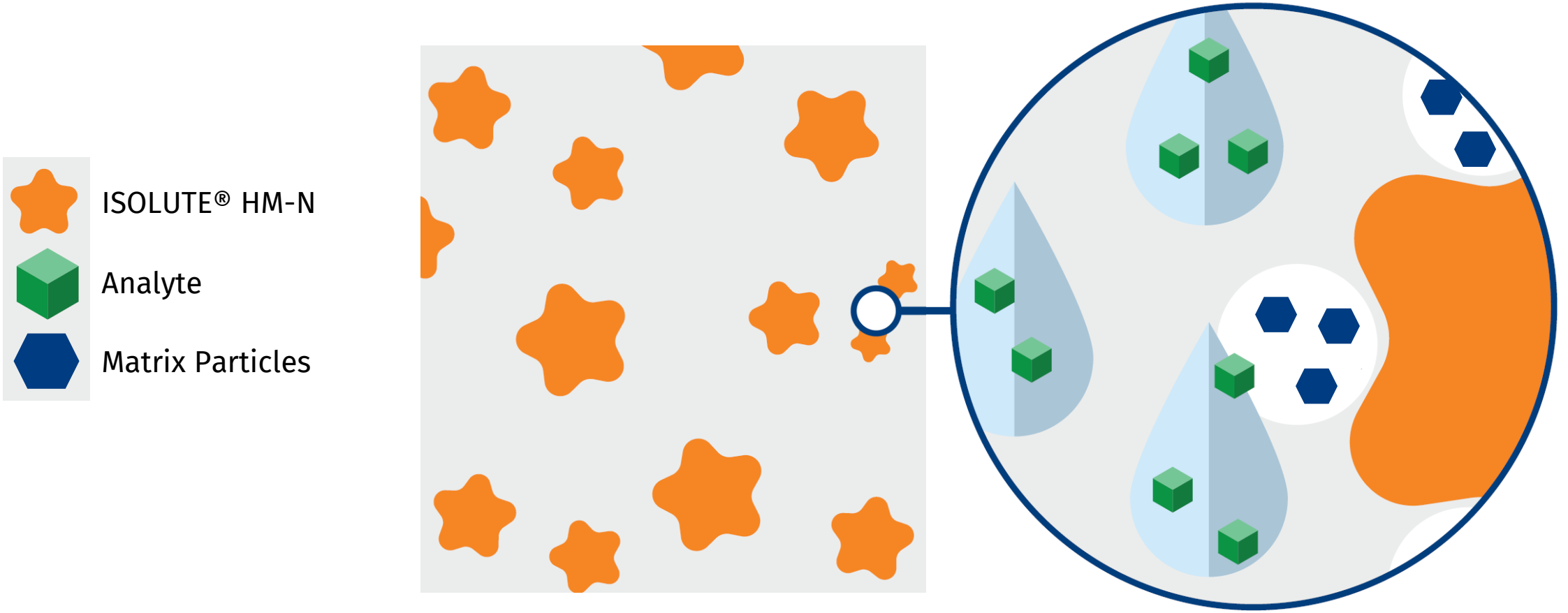
47mm Extended Disk Holder
ISOLUTE® HM-N (High Matrix - Neutralization)



ISOLUTE® HM-N
Chemical & Physical Properties

ISOLUTE® Product	HM-N	SLE+
Synonyms	Filter Agent, Celite®	
Diatomaceous Earth	≤ 50%	≥ 90%
Silicon Dioxide	50 – 100%	1 – 10%
Quartz	≤ 4%	1 – 10%
Average Particle Size	150 – 1400 Micron	≤ 180 Micron

ISOLUTE® HM-N
High Matrix – Neutralization

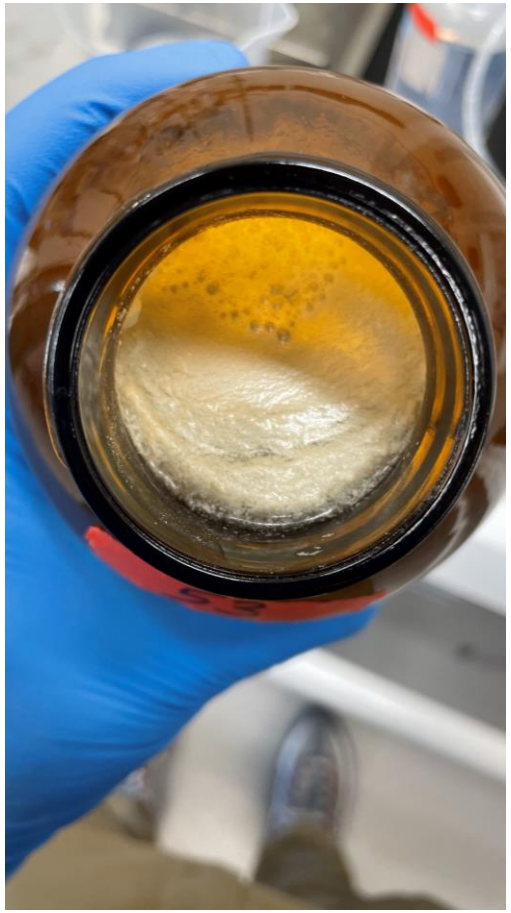


In-House Processing Performance
47mm Extended Disk Holder + ISOLUTE HM-N

- Samples Ran in Triplicate
- 10 g Soil Added to 1L RW
- Processing Time < 10 mins

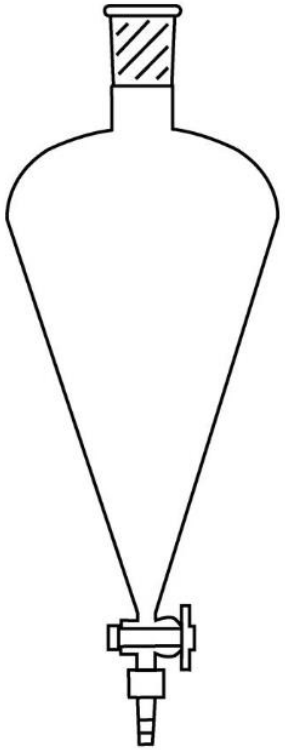
On-Site Processing Performance
EPA 625.1

- Laboratory transitioning from CLLE to SPE
- Significant Solvent Reduction
  - CLLE > 400mL DCM
  - SPE = 135 mL DCM
- Significant Time Savings
  - CLLE = 36 Hours
  - SPE < 1.5 Hours
- SPE Results Equivalent to CLLE Technique



EPA 625.1/8270 Surrogate Performance
Case Study LLE vs. Horizon 5000 SPE

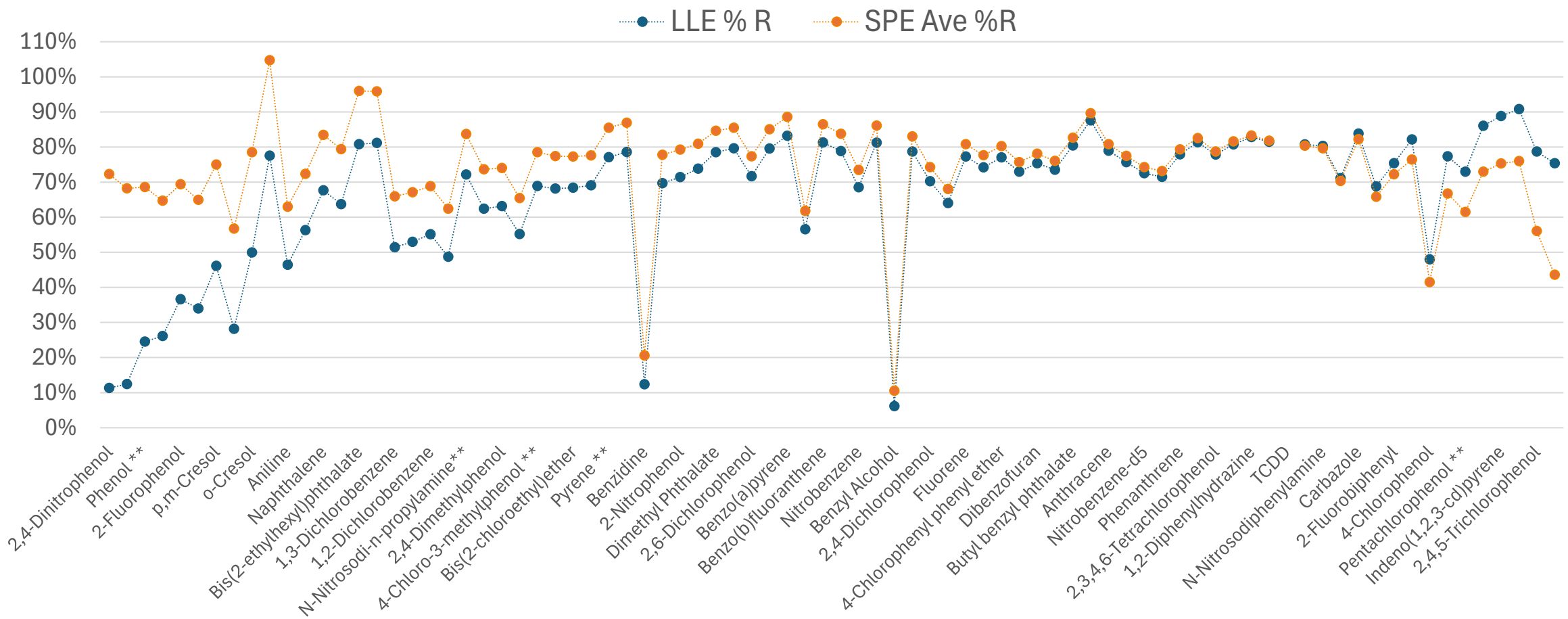
Liquid-Liquid Extraction (LLE) **vs** Horizon 5000 (SPE)



Surrogates*	Spike Concentration (ug/L)	LLE % Recovery	SPE % Recovery
2-FLUOREPHENOL	80	17	80
PHENOL-D5	80	10	69
NITROBENZENE-D5	40	35	80
2-FLUOROBIPHENYL	40	38	74
2,4,6-TRIBROMOPHENOL	80	55	99
TERPHENYL-D14	40	63	87

Surrogate – A compound unlikely to be found in a sample, and which is spiked into sample in a known amount before extraction or other processing, and is quantitated with the same procedures used to quantify other sample components. The purpose of the surrogate is to monitor method performance with each sample.

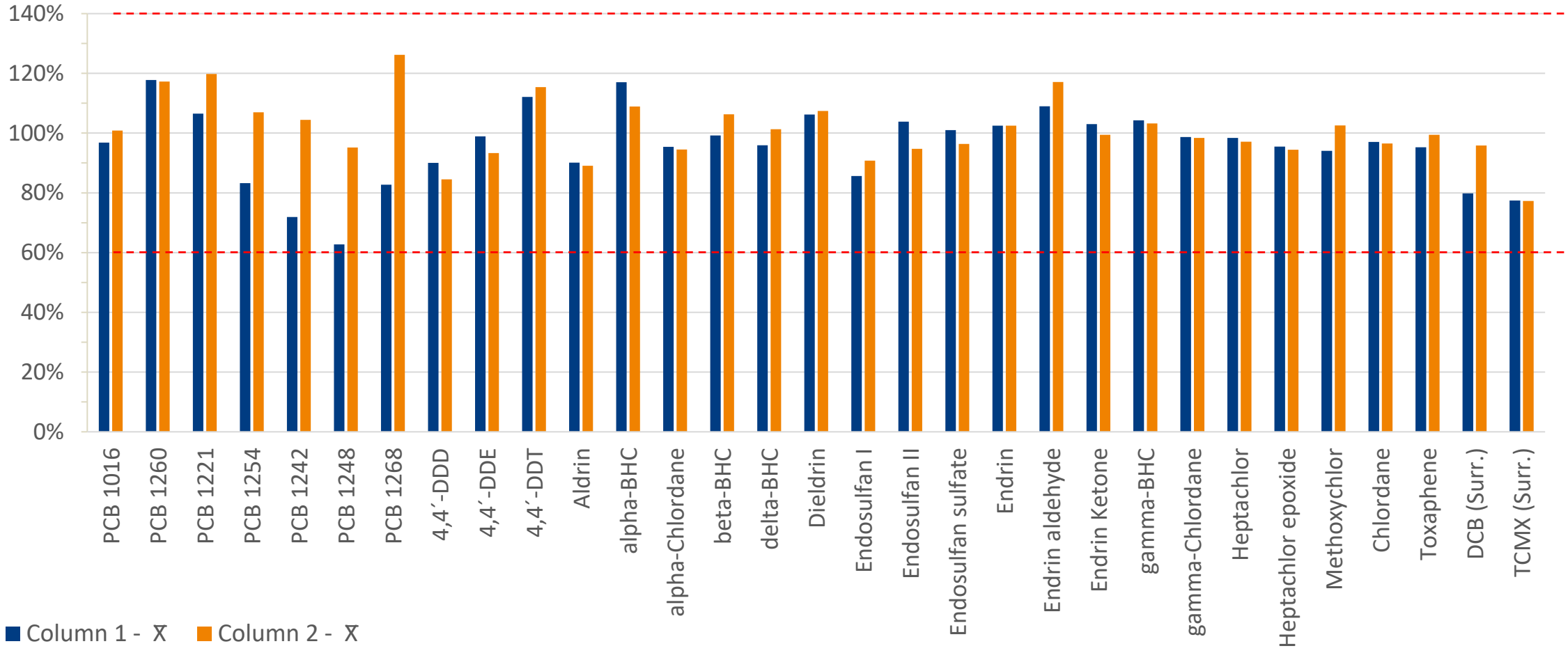
EPA 625.1/8270 Target Performance
Case Study LLE vs. Horizon 5000 SPE



On-Site Processing Performance
EPA 625.1 & 608.3

- Laboratory running SPE for both 625.1 & 608.3
- SPE Solvent Use by Method
  - EPA 625.1 = 135mL DCM
  - EPA 608.3 = 45 mL DCM
- Run Times by Method
  - EPA 625.1 = 1.5 hours
  - EPA 608.3 = 45 minutes
- New disk format worked for 90% of samples

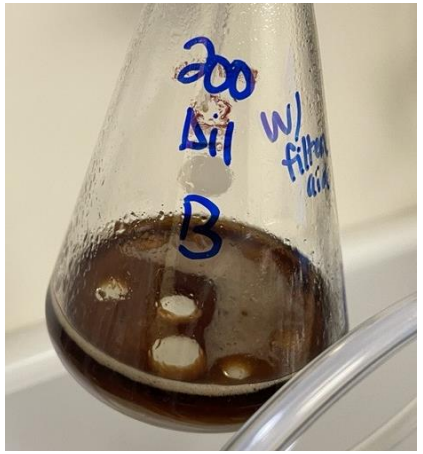
EPA 608.3 Target Performance
Horizon 5000 SPE



Extremely Challenging Samples
47mm Extended Disk Holder + ISOLUTE HM-N

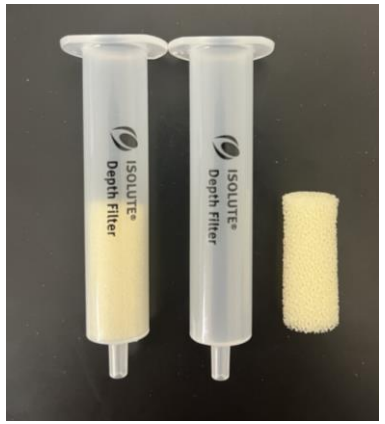
- A few cases of extreme samples from gas wells, leachate, & asphalt waste could not fully process at 1 L volumes
- New format could still process worst case sample volumes ranging from 125mL – 500 mL
- Horizon 5000 proved easy to clean, with no carry over on blanks ran after worst case samples
- Future work will evaluate two options for extreme sample processing
  - New 47mm Depth Filtration
  - Reduced Sample Volume Extraction

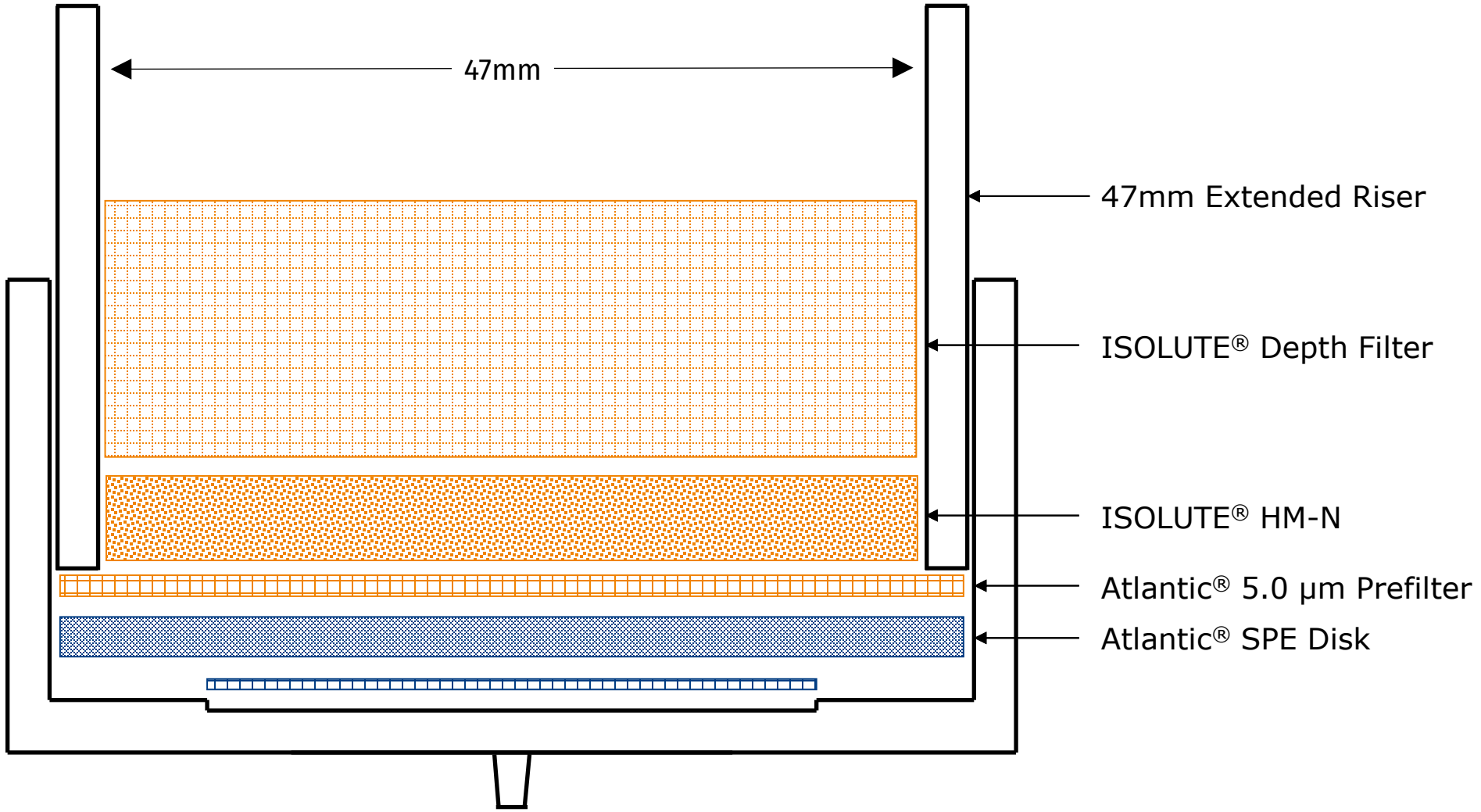






New 47mm Depth Filtration
Improve Processing Extreme Samples





Final Conclusions

Advancements in High Matrix Neutralization SPE Automation

- Horizon 5000 is an ideal SPE system for processing **large volume** aqueous samples & **challenging matrices**
  - Water Inlet Valve (WIV) Provides:
    - Flexible Sample Bottle Adaption
    - Direct SPE Sample Feed
    - Powerful Sample Bottle Rinsing
    - Quick & Easy Cleaning
    - Minimal Carryover Potential from Challenging Matrices
- Horizon 5000 station platform adapts to a **variety of SPE formats**
- New 47mm extended disk holder format **greatly reduces solvent** use while **increasing physical capacity** for processing large volumes of challenging matrices
  - Able to handle samples that were once only applicable to Fast Flow Disk Holder (FFDH)
  - Proven to meet performance criteria for EPA methods 625.1, 8270, 608.3, 8081, 8082
- Incorporation of new 47mm depth filter will expand processing capabilities for even the most extreme sample matrices

Thank you

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