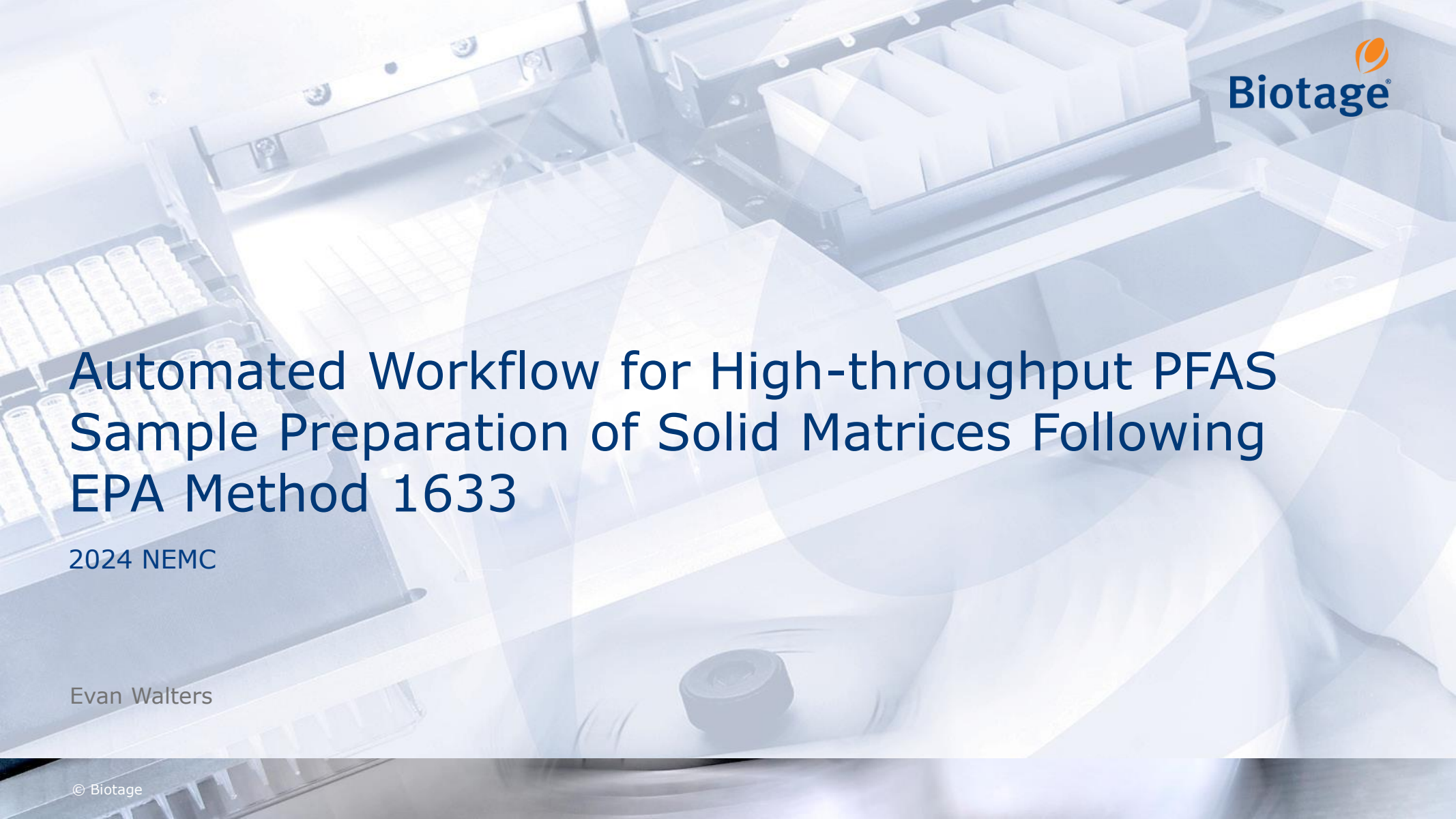


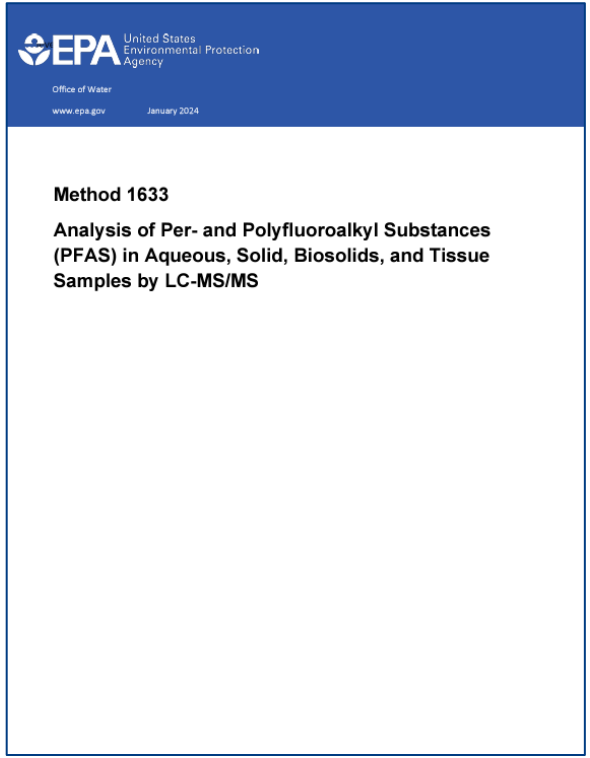
# Automated Workflow for High-throughput PFAS Sample Preparation of Solid Matrices Following EPA Method 1633

2024 NEMC

Evan Walters

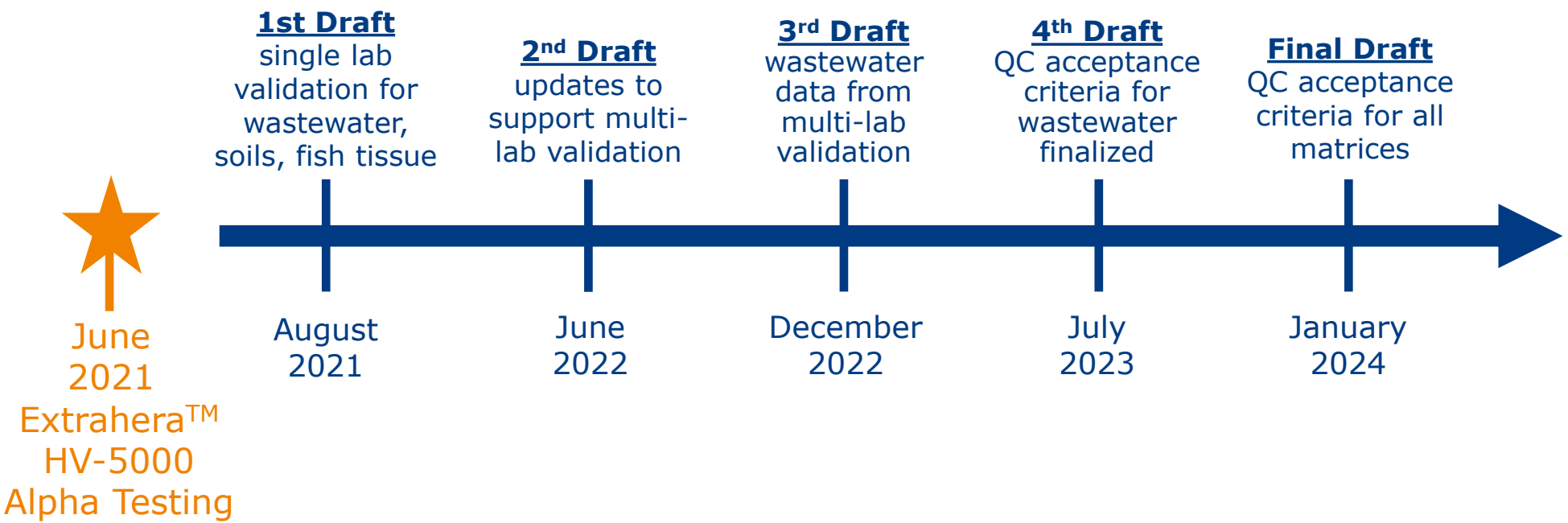
# Outline
## Automated Preparation of Solid Matrices for EPA 1633

- Overview of Workflow
- Workflow Performance
- Workflow Efficiency
- Summary



# USEPA 1633 Method

## History of Method



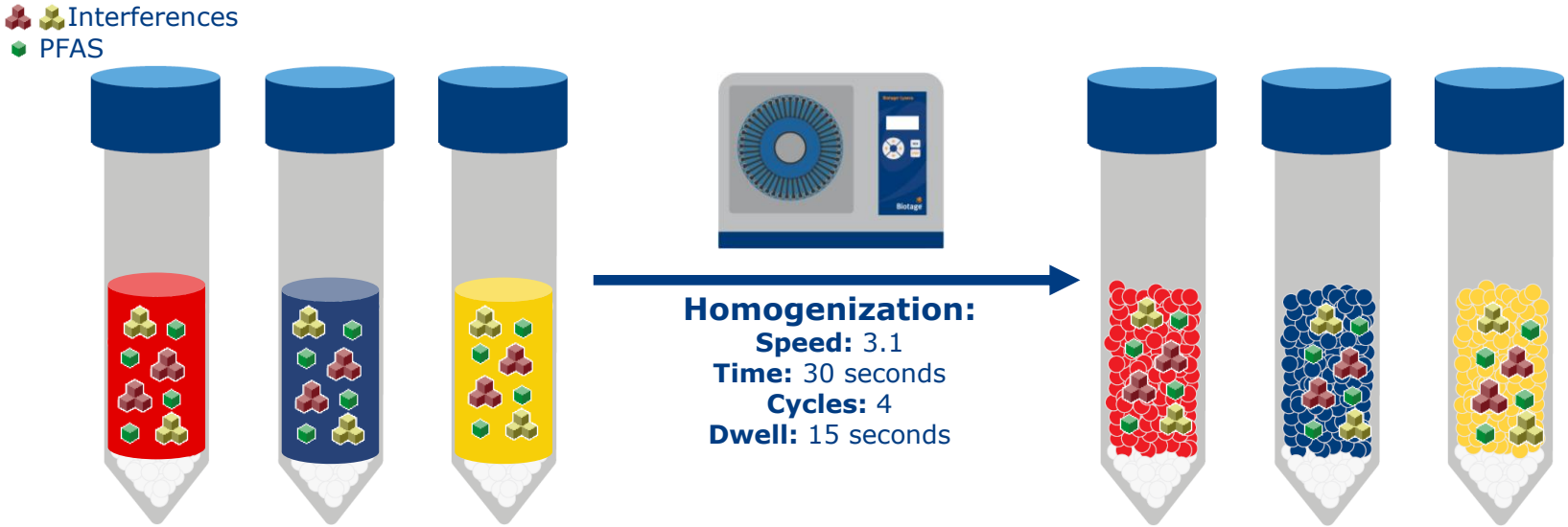
# EPA Method 1633 Sample Matrices

## Aqueous, Solids & Tissue Samples

| Matrix | Description | Volume or Mass | Homogenization & Extraction | Solid Phase Extraction | Solvent Evaporation |
|---|---|---|---|---|---|
| **Aqueous** | Water, sludges, and similar materials containing < 50mg solids/sample | Water (125 - 250mL) | | X | |
| | | Leachate (100mL) | | | |
| **Solids** | Soils, sediments, and biosolids that contain > 50mg solids | Soil & Sediment (5g) | X | X | X |
| | | Biosolids (0.5g) | | | |
| **Tissues** | Whole fish, fish fillets, and other tissues | Tissue (1-2g) | X | X | X |

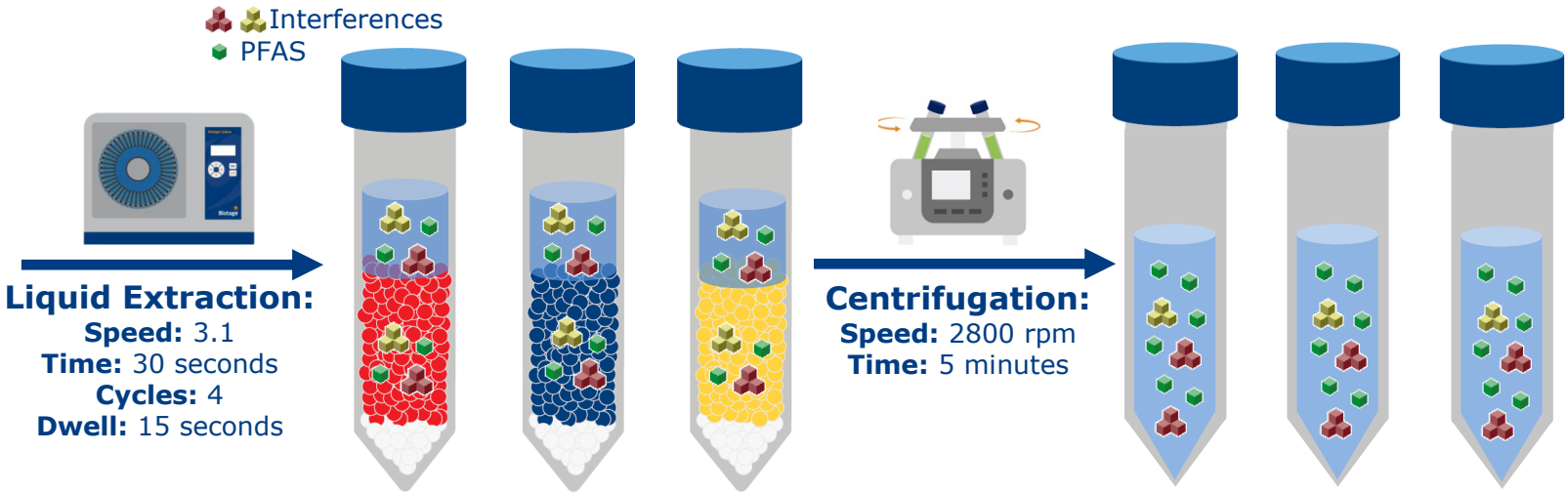
# Homogenization

## Biotage® Lysera Bead Mill



# Liquid Extraction

## Biotage® Lysera Bead Mill Processing

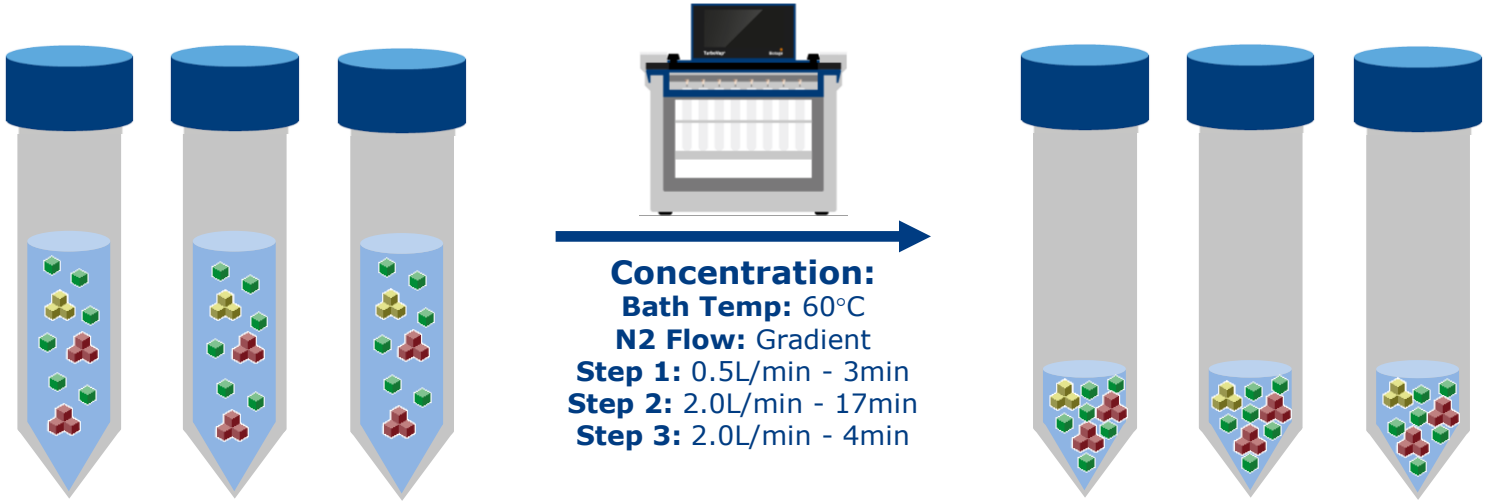



| Matrix Solvent | Soils & Biosolids | Tissue |
|---|---|---|
| First | 0.3% $NH_4OH$ in MeOH | 0.05M KOH in MeOH |
| Second | 0.3% $NH_4OH$ in MeOH | Acetonitrile |
| Third | 0.3% $NH_4OH$ in MeOH | 0.05M KOH in MeOH |

# Solvent Evaporation: Pre-Cleanup

## TurboVap® LV Heated Bath, Ramped $N_2$ Blowdown

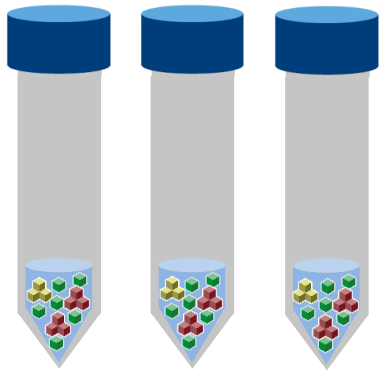
Interferences
PFAS



**Concentration:**
**Bath Temp:** 60°C
**N2 Flow:** Gradient
**Step 1:** 0.5L/min - 3min
**Step 2:** 2.0L/min - 17min
**Step 3:** 2.0L/min - 4min

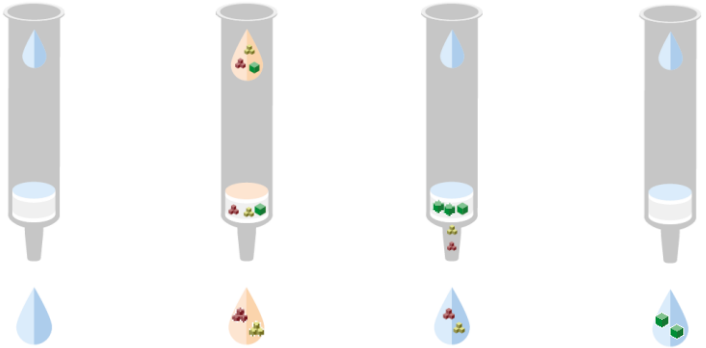
# Cleanup: Solid Phase Extraction

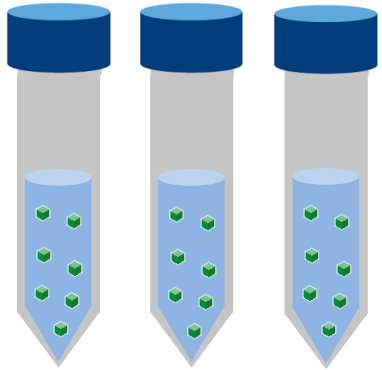
## Extrahera™ HV-5000 Automated SPE Workstation



**Option A:** Catch & Release

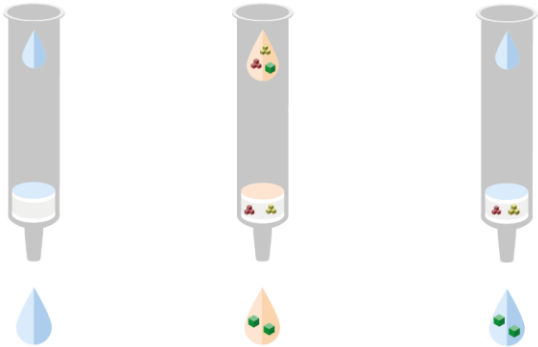
1. Dilute sample
2. Condition SPE
3. Equilibrate SPE
4. Load Sample
5. Wash SPE x 3
6. Dry SPE
7. Rinse Container & Fluid Path
8. Elute SPE & Collect





**Option B:** Pass-through

1. Condition SPE
2. Equilibrate SPE
3. Load Sample
4. Rinse Container & Fluid Path
5. Elute SPE & Collect

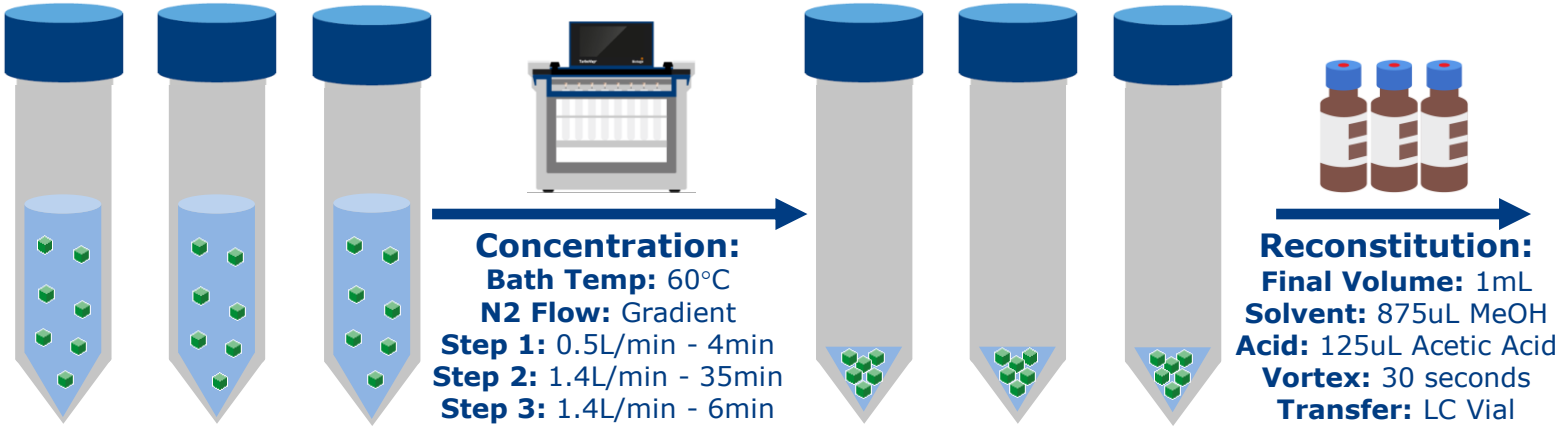


**Solid Phase Extraction:**
**Option A:** Catch & Release
**Option B:** Pass-through

# Extract Concentration: Post-Cleanup

## TurboVap® LV Heated Bath, Ramped N2 Blowdown

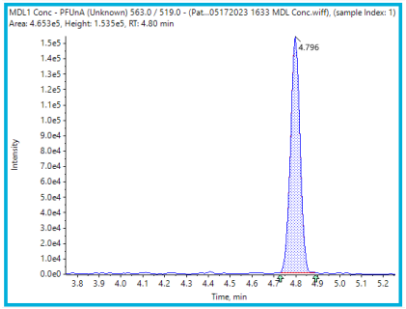
Interferences
PFAS



**Concentration:**
**Bath Temp:** 60°C
**N2 Flow:** Gradient
**Step 1:** 0.5L/min - 4min
**Step 2:** 1.4L/min - 35min
**Step 3:** 1.4L/min - 6min

**Reconstitution:**
**Final Volume:** 1mL
**Solvent:** 875uL MeOH
**Acid:** 125uL Acetic Acid
**Vortex:** 30 seconds
**Transfer:** LC Vial

**PFUnA MDL Spike Peak Intensity**
5mL Final Volume ~2.5e4
1mL Final Volume ~1.5e5



# Workflow Summary

## High-throughput PFAS Sample Preparation of Solid Matrices

Homogenize & Liquid Extraction

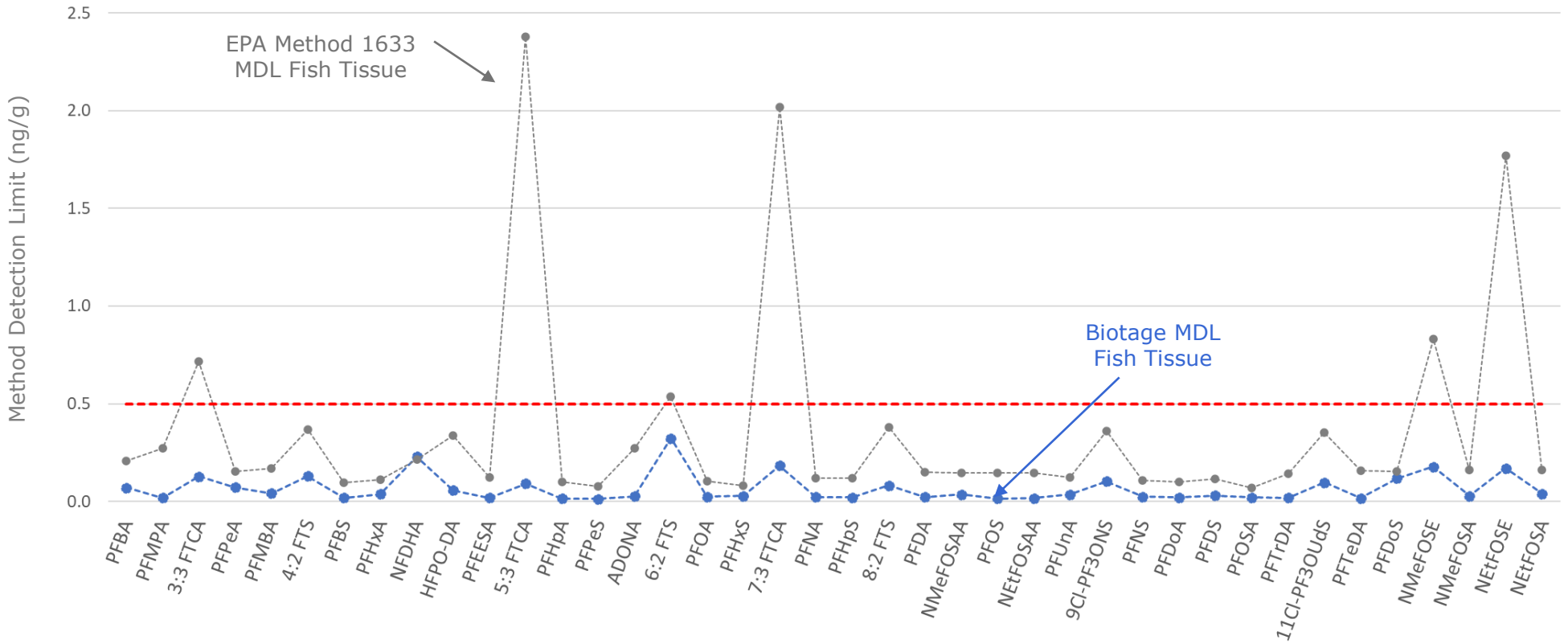
Solvent Evaporation


Solid Phase Extraction

Concentration & Reconstitution

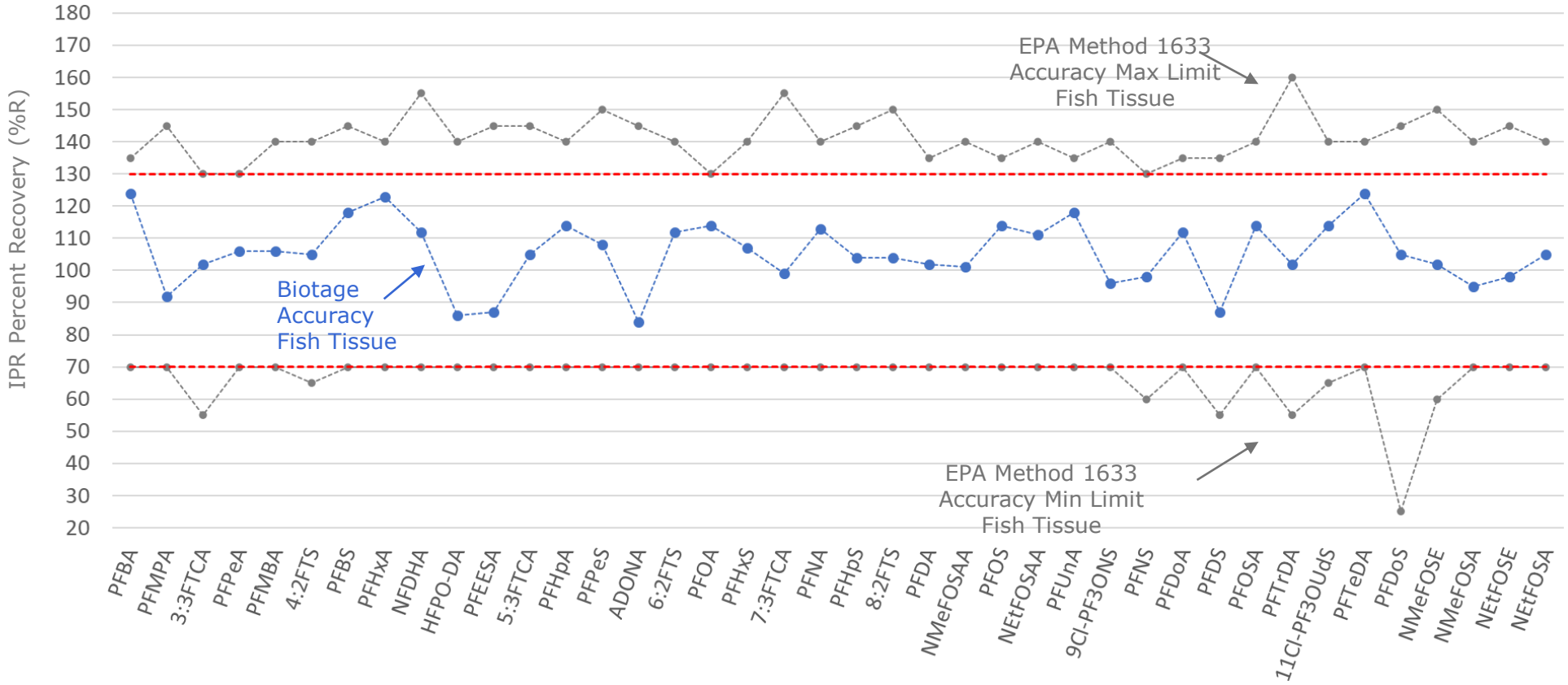
# Method Detection Limits

## Demonstrated MDL Compared to EPA 1633



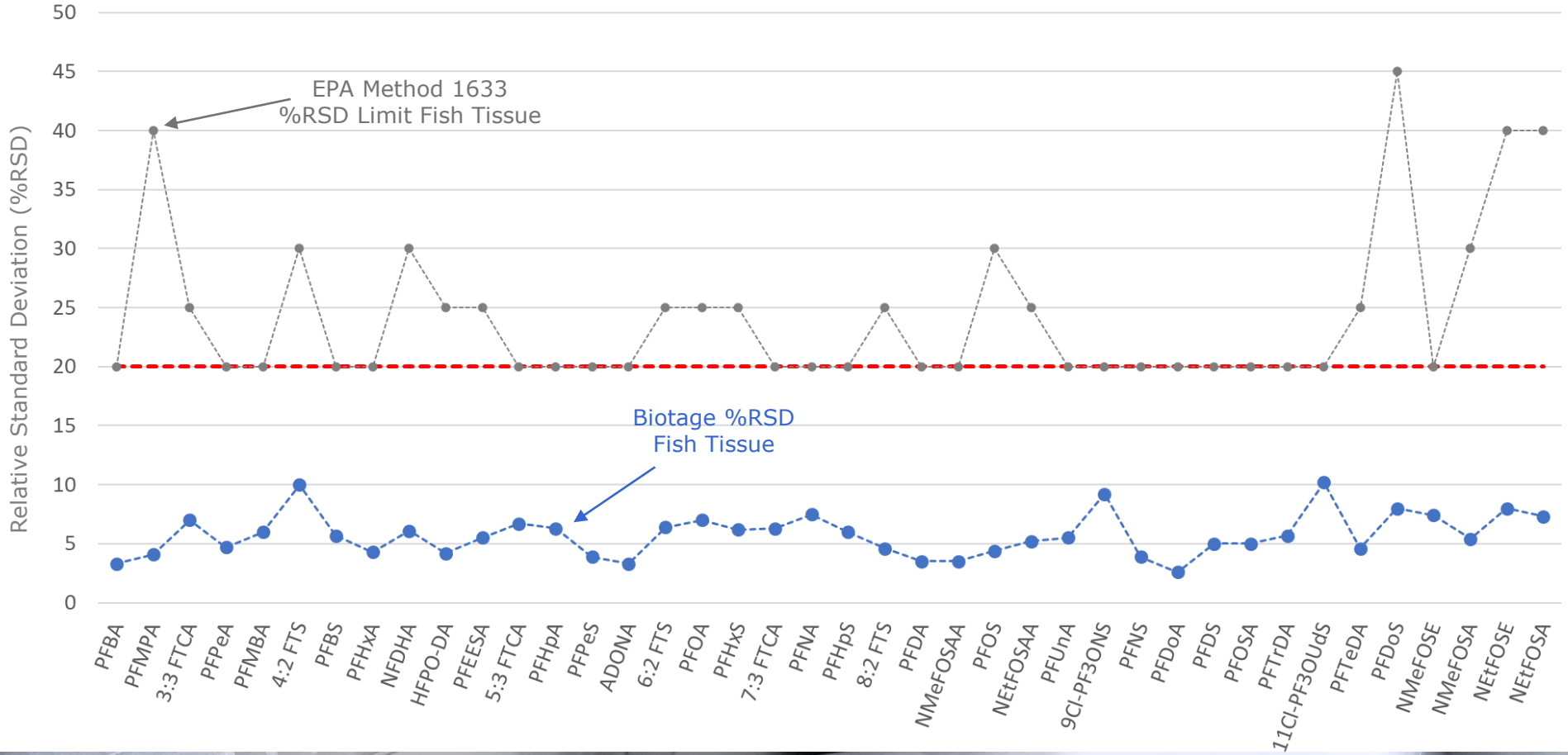
# Accuracy

## Demonstrated Accuracy Compared to EPA 1633



# Precision

## Demonstrated Precision Compared to EPA 1633



# Workflow Success in the Field

## PFAS in Soils (EPA 1633)

Biotage®

> Sometimes we get busy with PFAS samples and it's all hands-on deck in the lab. The Biotage workflow makes it possible to process multiple batches of samples in a day because it's so simple and fast.


> The Biotage 1633 PFAS in soils workflow has been extremely reliable. Since implementing these products in our lab, we rarely, if ever, have had to re-extract samples.

# Batch (24 Extractions) Processing Efficiency

## Homogenization, Extraction, Extract Cleanup & Concentration

**Figure 4.** Tissue Matrices

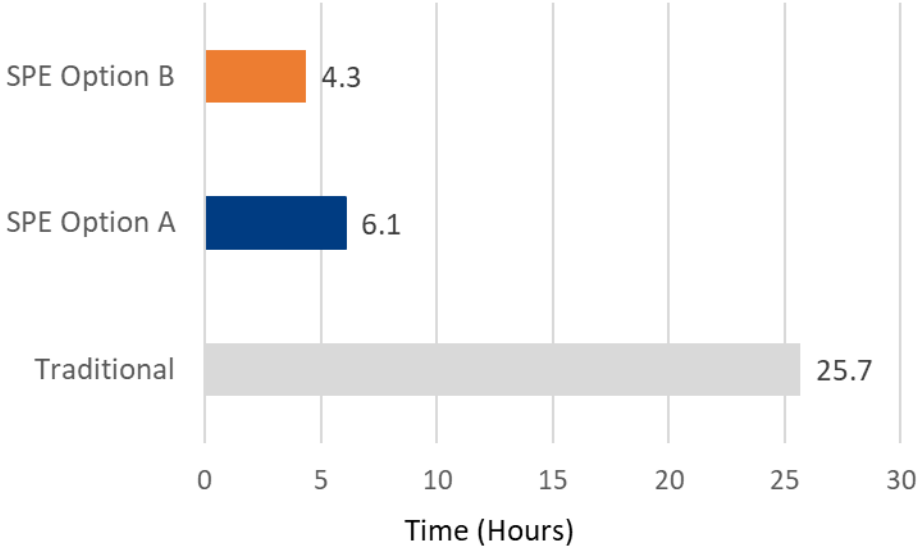
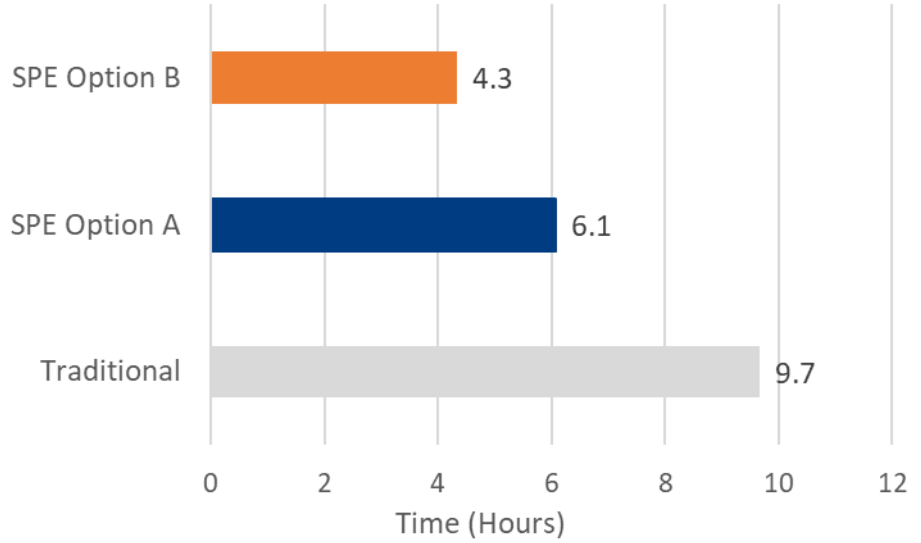



**Figure 5.** Soils & Biosolids



# Summary
## Automated Preparation of Solid Matrices for EPA 1633

- Biotage® Lysera
  - Homogenize Samples in <5min
  - Performed Liquid Extraction directly in homogenization tube
    - Eliminate transfer step
  - Liquid Extraction in <15min
    - Eliminate 16-hour extraction for fish tissue
- TurboVap® LV
  - Enhanced Detection with Final Concentration step
  - Control of Final Extract Composition
  - Rapid concentration of various tube sizes
- Extrahera™ HV-5000
  - Automated Batch processing of 24 samples in < 1 hour
  - Virtually no cleaning required from batch-to-batch processing
  - Minimal sample fluid path eliminates risk of carry over

More Information

# Thank You

## 2024 NEMC

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