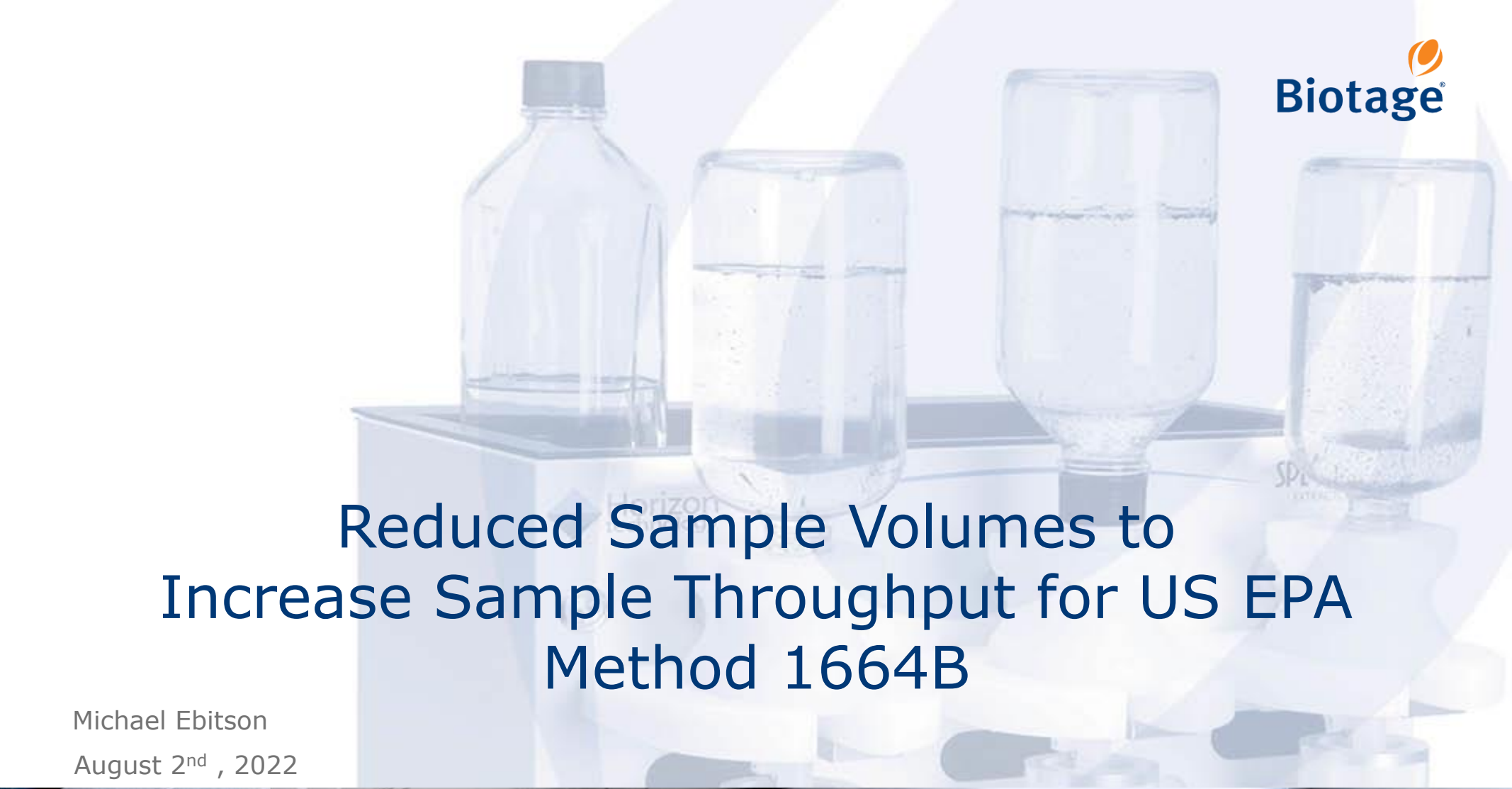

# Reduced Sample Volumes to Increase Sample Throughput for US EPA Method 1664B

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August 2nd , 2022

# Todays Topics

1. What is oil and grease testing?
2. Brief History of EPA method 1664
    1. Oil and Grease around the world
3. EPA 1664
    1. Why consider EPA 1664
        1. US- only method approved for oil and grease
4. Workflow for oil and grease
    1. What is best to implement in your lab?
5. Oil and grease SPE disk sizes
    1. Q&A
    2. Sediment capacity
6. Reduced sample volume study
7. How low can you go?
8. Conclusions
9. Q&A

# Definition of Oil and Grease Testing

Oil and Grease is a common way to describe **all soluble materiel in Hexane that is extracted from wastewater.**

It is technically referred to as **Hexane Extractable Material [HEM]** in all official methods but is referred to commonly and in trade as “Oil and Grease”

Referred to in many ways in many countries some for example:

- **[Oil and Grease],[TPH], [EPH], [soluble Aliphatic and Aromatics],[grease portion]**

Hexane Extractable Material is measured by many different techniques most common measurements:

- IR
- Gravimetric
- Gas Chromatograph

# Why is it important to humankind?

Clean Water Act's **N**ational **P**ollution **D**ischarge **E**limination **S**ystem (NPDES)

Examples of businesses that are required to perform Oil and Grease Measurements

- Restaurants
- Oil Refineries
- Chemical Plants
- Power Utilities
- Manufacturing/Engineering companies
- Wastewater treatment plants

✓ Leaking Underground Storage Tank regulatory program

✓ Petroleum exploration

# Global Concerns - “Fatberg”

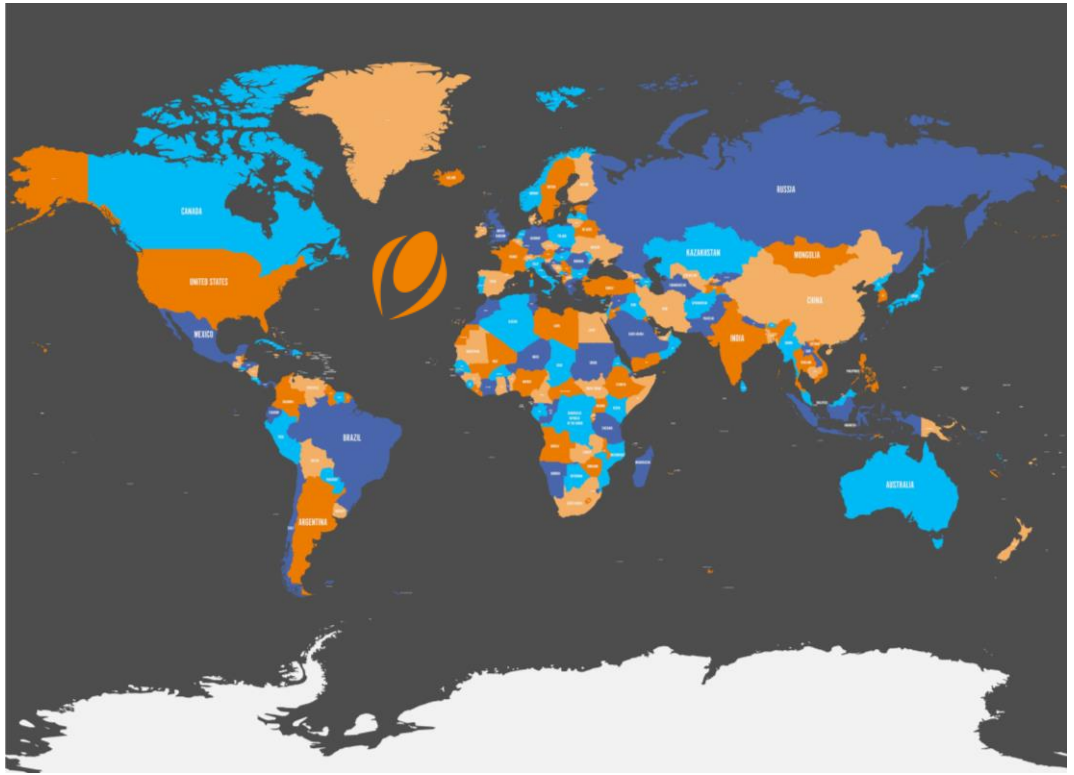
- The term fatberg what is it?
- How big can they get?
- What damage can they cause?
- How do can they become unblocked?
- How can they be prevented?



https://www.independent.co.uk/news/uk/home-news/fatberg-london-belgravia-cadogan-place-sewer-thames-water-b1444222

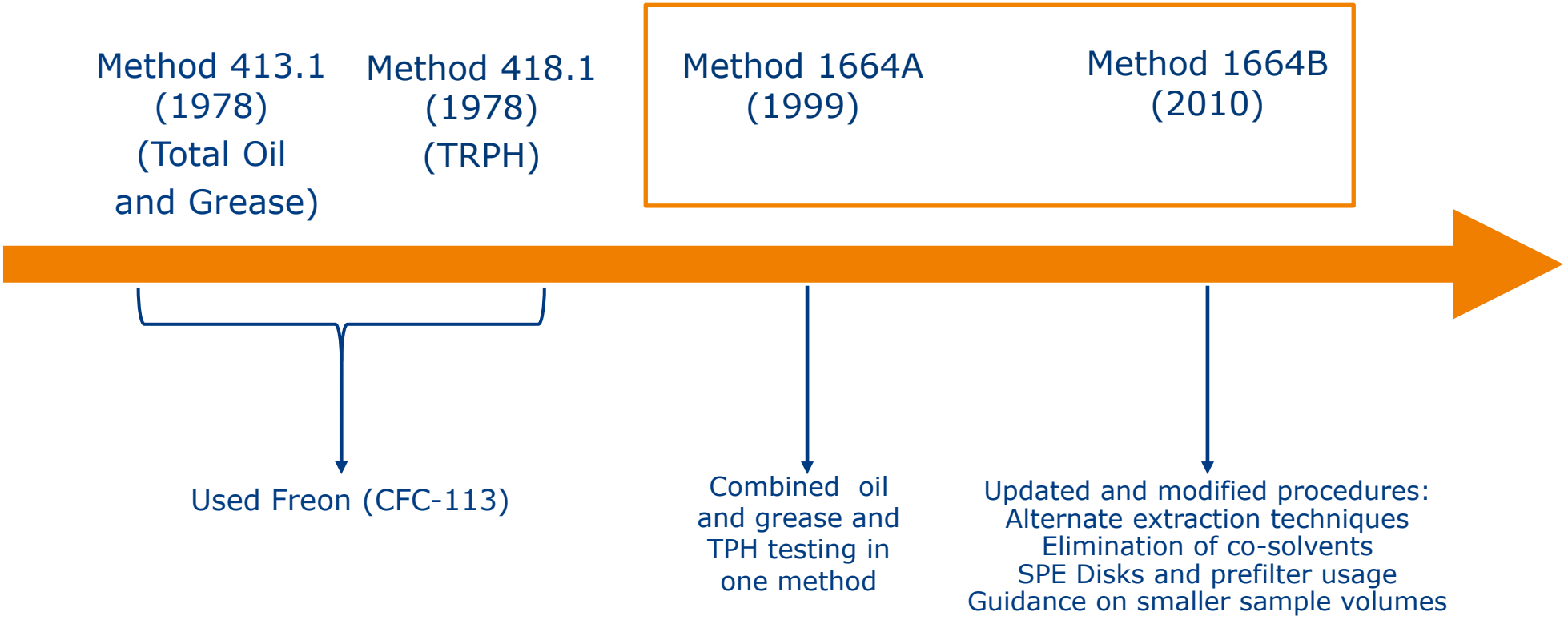
# Global Methods for Oil and Grease

- Standard Method 5520C Partition-Infrared method: Standard method for the examination of Water and Wastewater, 1999
- ASTM® D7066-04: Standard test method for dimer/trimer of chlorotrifluoroethylene (S-316) recoverable Oil and Grease and Nonpolar material by Infrared determination, 2004
- EPA 1664B: The gravimetric method measures the weight of the oil that is removed from the sample with hexane and is evaporated off.



# History of Oil and Grease Testing

Biotage



# EPA Method 1664B

There is a continued need for a simple, easy to follow method to assess water quality for treatment process performance and environmental protection

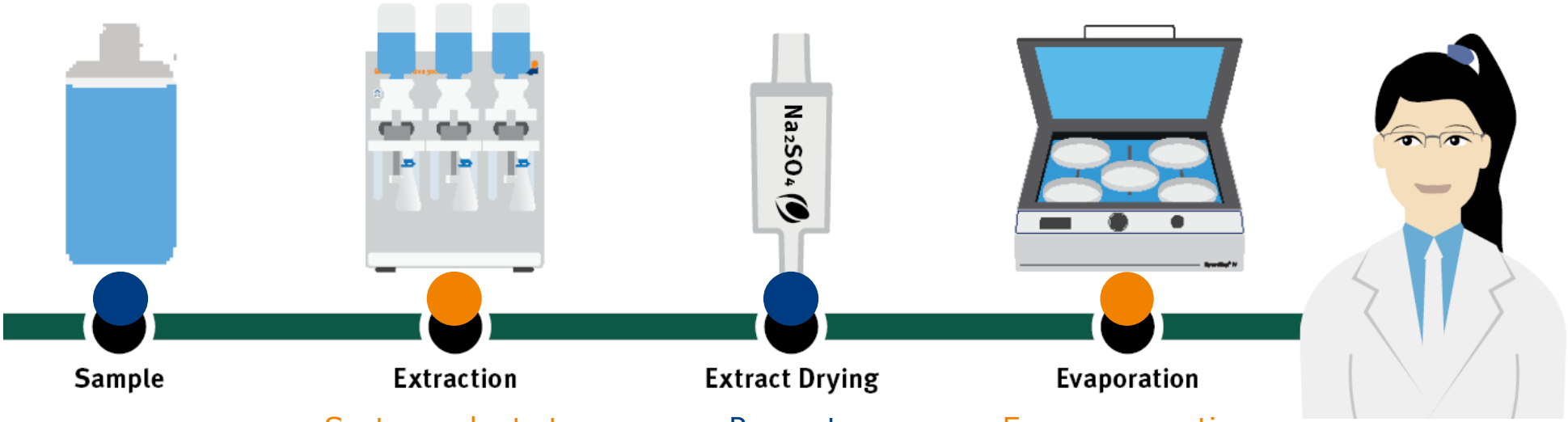
Oil and Grease testing can provide a method to do this with the following advantages

- Simple
- Inexpensive
- Can be automated

The US methods have become adopted in many places around the world and provide a good starting point for more detailed assessment

# 1664 Workflow Solution

## Oil & Grease Extraction Workflow




| Sample | Extraction | Extract Drying | Evaporation |
|---|---|---|---|
| 100mL-1L | System adapts to any sample bottle types | Prevents False positives | Even evaporation No hot or cool spots |

# Automated Extraction Platforms

Biotage

**Biotage Horizon 3100**

**Biotage Horizon 5000**

# How to Choose the Right Extraction System

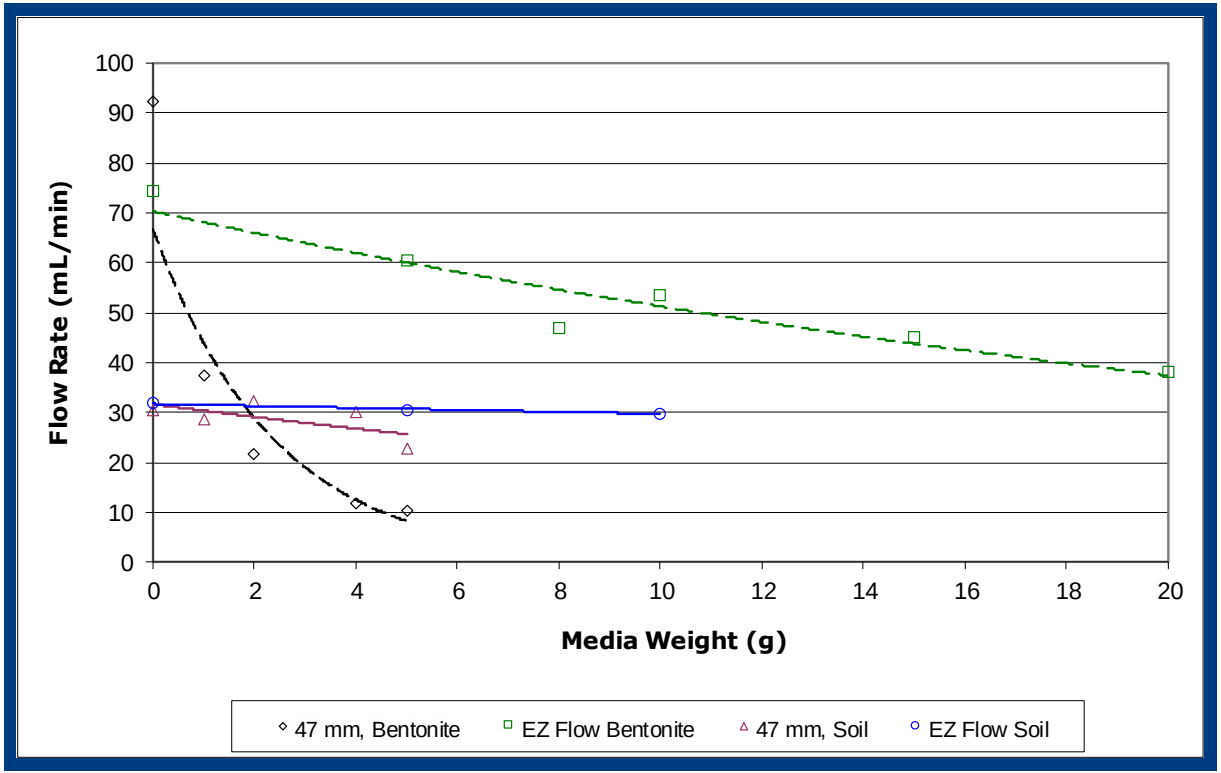
| | 5000 | 3100 |
|---|---|---|
| EPA 1664 | ✓ | ✓ |
| Extract other EPA Aqueous Methods | ✓ | |
| In-line Na2SO4 Cartridge | | ✓ |
| Interactive User Training | ✓ | ✓ |

# Oil and Grease SPE Disks- What to Know

# What Disk Size Should You Use?

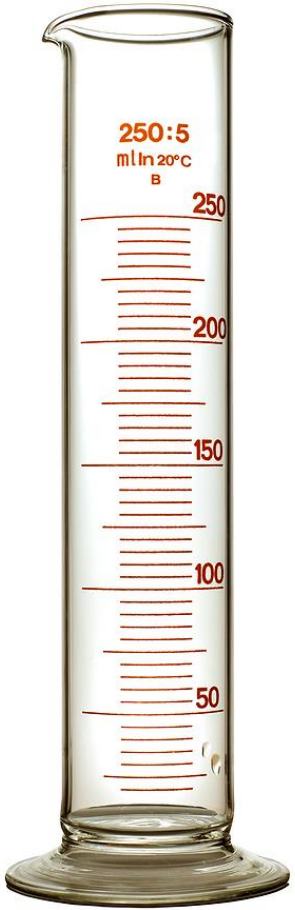
- How much sediment can I run?
  - Matrix Type
  - Particle Size/Shape
  - Disk Area (diameter)

- Limits:
  - Volume of Disk Holder
  - Geometry of particles



# Reduced Sample Volume Study

# Benefits of Reducing Sample Volumes




- Reduced shipping costs
- Reduced sample footprint (storage)
- Decrease solvent consumption
- Reduced water waste
- Improve sample throughput
- Helpful with dirty samples
- Smaller disks and less filter aid required
- More free time to complete other tasks

# Extraction Procedure on the Horizon 5000


- Obtain the 250 mL samples of DI water and acidify to pH 2
- Use a 47 mm Pacific® Premium disk for extraction
- Spike the samples with the proper amount of hexane extractable material (HEM) using Oil and Grease QC Standard (8mg/mL)
  - MDL: 125 µL of standard
  - LCS: 1250 µL of standard
- Attach the 19/22 tapered 125 mL separatory funnel or Erlenmeyer flask onto the collection adapter
- Complete extraction with programmed method

# Biotage 5000 Initial Demonstration of Capability Study

## IPR Results

| Sample | HEM (mg/L) | Percent Recovery (%) |
|---|---|---|
| 1 | 39.6 | 99.0 |
| 2 | 38.4 | 96.0 |
| 3 | 39.6 | 99.0 |
| 4 | 39.6 | 99.0 |
| Average Percent Recovery (X) | | 98.2 |
| Precision (s) | | 1.50 |

# Biotage 5000 Method Detection Limit Study

| MDL Results | |
|---|---|
| Sample | HEM (mg/L) |
| 1 | 3.2 |
| 2 | 3.2 |
| 3 | 2.8 |
| 4 | 3.6 |
| 5 | 3.2 |
| 6 | 3.6 |
| 7 | 2.8 |
| Precision (s) | 0.33 |
| MDL Value < 1.4 mg/L | 1.03 |

# Biotage 5000 Method Blank Data

| Blank Results | |
|---|---|
| Sample | HEM (mg/L) |
| 1 (1 L Blank) | 0.2 |
| 2 (250 mL Blank) | 0.0 |
| 3 (250 mL Blank) | 0.0 |
| 4 (250 mL Blank) | 0.0 |

All blanks < MDL  1.4 mg/L

# Extraction Procedure on the Horizon 3100

- Obtain 250 mL samples of DI water and acidify to pH 2
- Use a 47 mm Pacific® Premium disk for extraction
- Spike the samples with the proper amount of hexane extractable material (HEM) using Oil and Grease QC Standard (8mg/mL)
  - MDL: 125 µL of standard
  - LCS: 1250 µL of standard
- Attach the 24/40 tapered 125 mL separatory funnel or Erlenmeyer flask onto the collection adapter
- Complete extraction with programmed method

# Biotage 3100 Initial Demonstration of Capability Study

| IPR Results | | |
|---|---|---|
| Sample | HEM (mg/L) | Percent Recovery (%) |
| 1 | 37.2 | 93.0 |
| 2 | 35.2 | 88.0 |
| 3 | 36.0 | 90.0 |
| 4 | 36.8 | 92.0 |
| Average Percent Recovery (X) | | 90.7 |
| Precision (s) | | 2.22 |

# Biotage 3100 Method Detection Limit Study

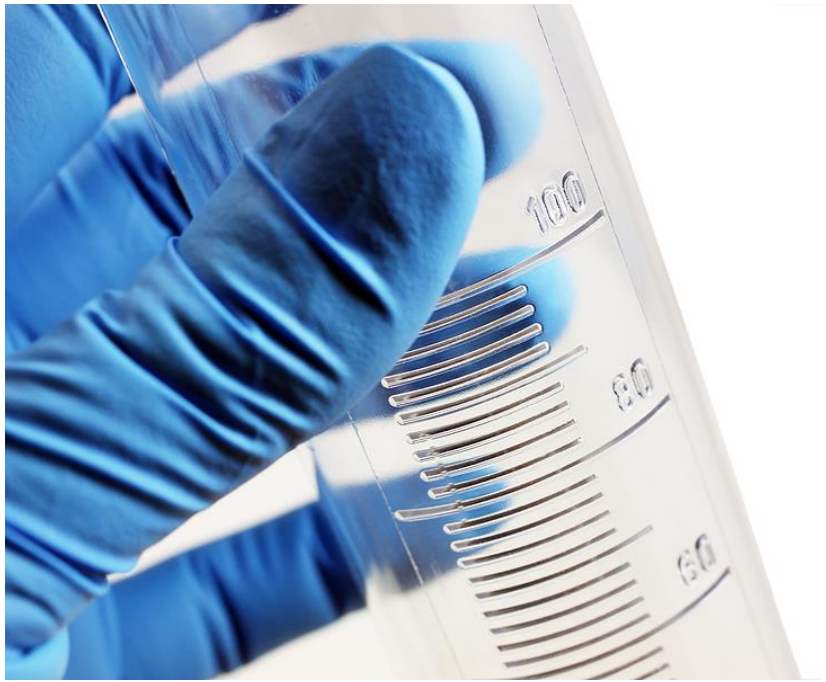
| MDL Results | |
|---|---|
| Sample | HEM (mg/L) |
| 1 | 2.8 |
| 2 | 2.8 |
| 3 | 3.2 |
| 4 | 3.2 |
| 5 | 3.2 |
| 6 | 2.8 |
| 7 | 3.2 |
| Precision (s) | 0.21 |
| MDL Value < 1.4 mg/L | 0.67 |

# Biotage 3100 Method Blank Data

| Blank Results | |
|---|---|
| Sample | HEM (mg/L) |
| 1 (1 L Blank) | 0.4 |
| 2 (250 mL Blank) | 1.2 |
| 3 (250 mL Blank) | 1.2 |
| 4 (250 mL Blank) | 1.2 |

All blanks < MDL 1.4mg/L

# Can I Reduce Sample Size to 100 mL?



- Section 1.7.1.5 of US EPA Method 1664B outlines the use of 100 mL samples
- A balance capable of measuring 0.00001 g is required for 100 mL samples

# 100mL Method Blanks

| Blank | Initial Wt (g) | Final Wt (g) | Diff Wt (mg) | Dilution Factor (x10) |
|---|---|---|---|---|
| 1 | 6.0855 | 6.0857 | 0.2 | 2 |
| 2 | 6.1874 | 6.1876 | 0.2 | 2 |
| 3 | 6.1563 | 6.1565 | 0.2 | 2 |

| Acceptance Criterion | mg/L |
|---|---|
| Average | 0.2 |
| Standard Deviation | 0.0000 |
| Below the Calculated MDL<br>MDL Value < 1.4 mg/L | 2.0 |

# 100mL IPR Samples

| IPR | Initial Wt (g) | Final Wt (g) | Diff Wt (mg) | Dilution Factor (x10) | Subtract Blank | % Rec |
|---|---|---|---|---|---|---|
| #1 | 6.1560 | 6.1599 | 3.9 | 39.0 | 38.8 | 97 |
| #2 | 6.1630 | 6.1670 | 4.0 | 40.0 | 39.8 | 99.5 |
| #3 | 2.4314 | 2.4352 | 3.8 | 38.0 | 37.8 | 94.5 |
| #4 | 2.4398 | 2.4436 | 3.8 | 38.0 | 37.8 | 94.5 |

| HEM Ave % Rec | HEM Precision |
|---|---|
| 96.4 | 0.96 |

# MDL Results: 100mL samples

| MDL | Initial Wt (g) | Final Wt (g) | Diff Wt (mg) | Dilution Factor (x10) | % Rec |
|---|---|---|---|---|---|
| #1 | 2.4368 | 2.4372 | 0.4 | 4 | 100.0 |
| #2 | 2.4365 | 2.4370 | 0.5 | 5 | 125.0 |
| #3 | 2.4344 | 2.4348 | 0.4 | 4 | 100.0 |
| #4 | 2.4315 | 2.4319 | 0.4 | 4 | 100.0 |
| #5 | 2.4143 | 2.4147 | 0.4 | 4 | 100.0 |
| #6 | 2.4394 | 2.4398 | 0.4 | 4 | 100.0 |
| #7 | 2.4226 | 2.4231 | 0.5 | 5 | 125.0 |

# What Do the Results Mean?

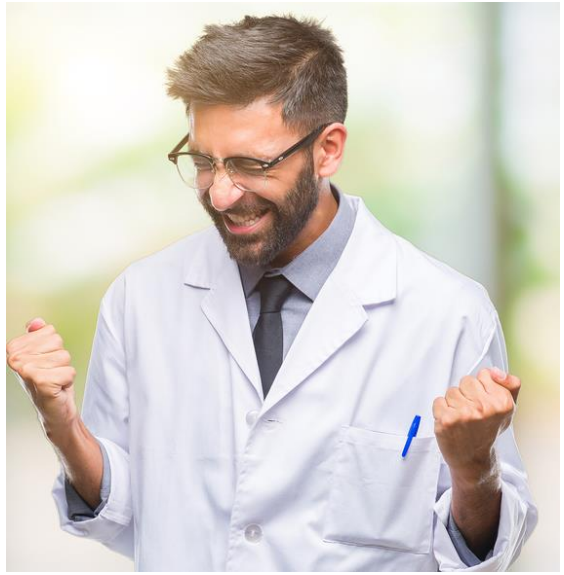
- **Acceptance Criteria is achieving 1.4 mg/L or less for MDL**
- **Achieved a standard deviation of 0.0488**
- **MDL for seven samples = 0.1533mg**
- **Add the times ten (X10) dilution factor MDL= 1.5331**
  - **Does not pass method criteria.**
  - **All mg results were rounded up decreasing the accuracy**
  - **A semi micro scale is needed to detect to 0.01mg**

# Scales: Increase sensitivity

| Balance Type | Readability | Sample Volume | Estimated Price ($USD) | Balance Table |
|---|---|---|---|---|
| Ultra-Micro | 0.1 ug | 1- 10 mL | >$35,000 | Yes |
| Micro | 1 ug | 10 – 100 mL | $10,000 - $35,000 | Yes |
| Semi-Micro | 0.01 mg | 100 - 250 mL | $5,000-$10,000 | Yes |
| Analytical | 0.1 mg | 250 mL - 1L | $2,000-$5,000 | No |
| Precision | 1 mg | >1L | $1000-$2,000 | No |

# Conclusions

- Globally there is a need for oil and grease testing
  - Gravimetric analysis is more robust for heavier fats oil and grease (FOG)
  - IR methods are not able to accommodate those higher levels mg/L
- Automated solutions will be more consistent, leading to lower MDLs and higher quality of results.
  - Having the right solutions make it easier for workflows and all levels of employee knowledge. Turnkey at any level!
- Key points- Reducing Sample Volumes
  - Reduced shipping costs
  - Reduced sample footprint (storage)
  - Decrease solvent consumption
  - Reduced water waste
  - Improve sample throughput
  - Helpful with dirty samples
  - Smaller disks and less filter aid required
  - More free time to complete other tasks



# Where to Find Additional Information

- **Blogs**
  - Environmental Blogs
- **www.Biotage.com**
  - **Application Notes**
    - EPA 1664B Reduce Sample Volume
    - EPA 1664B Extraction with In-Line Sodium Sulfate
    - EPA 1664B Extraction on VacMaster Disk Manual Manifold
- **Products**
  - **Extraction**
    - Manual Systems
    - Automated Systems
    - Pacific® Oil & Grease Disks and Pre-Filters
  - **Extract Drying**
    - ISOLUTE® Sodium Sulfate Drying Cartridge
    - DryDisk® Solvent Drying System (SDS)
  - **Evaporation**
    - Speed-Vap®

# Questions and Answers

Thank you for attending
For more information, please visit
Biotage.com

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