

# Improving the 90 mm Disk Oil & Grease Method Using the Horizon 5000 (EPA 1664B)

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## Introduction

The treatment and removal of oil and grease from wastewater is imperative because it can negatively affect the biological and aquatic life that encounter it. Not only that, but since the oil and grease are not miscible with water, it leaves an unappealing layer on top of the water. Even before the oil and grease can reach the aquatic life, it will solidify on the inner walls of pipes, causing blockage over time.

Oil and grease analysis is routinely performed in many labs that also extract and analyze a variety of analyte suites using different EPA methods, so time management and flexibility are a necessity. The Biotage® Horizon 5000 offers these labs a flexible solution that can extract a wide variety of analytes as well as oil and grease. In these circumstances it is imperative to have a streamlined solution with the minimum amount of manual operation required.

The purpose of this application note is to optimize the solution for the removal and testing of total n-hexane extractable material (HEM) using solid phase extraction in combination with the Biotage® Horizon 5000 automated extraction system. Using the SPEED-VAP® IV Solvent Evaporation System, the extracts will be evaporated with gentle heat and consistent air flow through precisely drilled holes.

## Instrumentation

### Biotage Products:

- » Biotage® Horizon 5000 (P/N SPE-DEX 5000)
- » SPEED-VAP® IV – Solvent Evaporation System (P/N 200-1000-04)
- » Pacific® Premium Oil & Grease Disk, 90 mm (P/N 1664-100-PHT)
- » Pacific® O&G Fast Flow Pre-Filter, 90 mm (P/N FFP-90-HT)
- » Oil and Grease Standards, 40 mg (P/N 50-021-HT)
- » Oil and Grease Standards, 26 mL (P/N 50-003-HT)
- » Oil & Grease Aluminum Weighing Pans, 105 mm, 125 mL (P/N 50-002-02-HT)

### Other Instruments:

- » Analytical Balance

## Method Summary

- Obtain 1L DI water samples and acidify to  $\text{pH} \leq 2$  using concentrated HCl.
- Spike any relevant IPR samples with the 40 mg Oil & Grease Standard and any MDL samples with 500  $\mu\text{L}$  of the 26 mL Oil & Grease Standard vial for a final concentration of 4.0 mg.
- Attach the sample bottle to the water inlet valve using cap adapters as necessary, then place them onto the extractor.
- Load the disk holder with a 90 mm Pacific® Premium disk and place onto the disk holder platform.
  - If needed, 90 mm Pacific O&G Fast Flow Pre-Filters may be used when working with high particulate samples, however the entire study must be completed using prefilters.
- Place a clean 125 mL separatory funnel or equivalent onto the collection vessel adapter, securing it with a clip.
- Extract the sample using extraction method found in Table 2.
- Dry the final extract with phase separation paper (DryDisk® from Biotage) or  $\text{Na}_2\text{SO}_4$  and thoroughly rinse the collection vessel with hexane to collect any residual HEM.
- Pre-weigh 105 mm pans and transfer the dried extract into each pan rinsing collection flask three times.
- Using the SPEED-VAP® IV Solvent Evaporation System, evaporate the extracts utilizing the parameters in Table 1.
  - Be sure to remove the sample from the SPEED-VAP as soon as the extract evaporates. If needed complete further evaporation under a hood and then transfer to a desiccator.
- Weigh each pan and calculate the HEM recovery in mg.
- To complete the IPR study, extract four replicates using steps one through ten. To pass this study, average % recovery should be between 83–101% and precision should be  $< 11\%$ .
- To complete the MDL study, extract seven replicates using steps one through ten over three separate days. To pass this study, the calculated MDL must be  $< 1.4 \text{ mg/L}$ .

Table 1. SPEED-VAP® IV Parameters.

Step	Operation
Temperature (°C)	40
Compressed Air Inlet Pressure (psi)	80

Table 2. 90 mm Pacific No-Pre-filter 1664 Extraction.

Step	Select Solvent	Volume (mL)	Purge (s)	Vacuum	Saturate (s)	Soak (s)	Drain/Elute (s)	Sample Delay (s)	
Condition SPE Disk	Hexane	16	60	2	1	60	60		
Condition SPE Disk	Methanol	16	60	2	1	60	2		
Load Sample				5					45
Air Dry Disk				6			180		
Elute Sample Container	Hexane	21	35	5	1	10	15		
Elute Sample Container	Hexane	15	35	5	1	45	45		
Elute Sample Container	Hexane	15	60	6	1	45	60		
Wash Sample Container	Methanol	8	60	6	1	20	60		
Elute Sample Container	Hexane	9	35	5	1	45	45		
Elute Sample Container	Hexane	9	35	5	1	45	45		
Elute Sample Container	Hexane	9	60	6	1	45	60		

**Table 4.** IPR Results.

Sample	Recovery (mg)	Recovery (%)
1	39.2	98.0
2	39.7	99.2
3	39.7	99.2
4	39.0	97.5
<b>Average % Recovery</b>		98.5
<b>Standard Deviation</b>		0.89

**Table 5.** Sample Blank Results.

Sample	Initial Weight (g)	Final Weight (g)
1	6.4548	6.4549
2	6.4416	6.4415
3	6.4626	6.4626
<b>All &lt; Calculated MDL of 0.3360 mg/L</b>		

**Table 6.** MDL Results.

Sample	Recovery (mg)	Recovery (%)
1	3.7	92.5
2	3.5	87.5
3	3.7	92.5
4	3.6	90.0
5	3.6	90.0
6	3.6	90.0
7	3.4	85.0
<b>Standard Deviation</b>		0.1069
<b>Calculated MDL (mg/L)</b>		0.3360

## Discussion

The acceptable recovery limit for an IPR study outlined in EPA Method 1664B is 83–101%. As you can see in table 4 the recoveries all fall within that range, confirming that this method passes the IPR guidelines.

Table 5 shows that there is no background contamination that would skew any of the results.

Furthermore, Method 1664B sets the acceptance criteria for an MDL study to be 1.4 mg/L or less, where the calculated value in table 6 concludes that our study passes.

## Conclusion

This application note demonstrates that the final extract volume has been optimized, in turn shortening the extraction and evaporation times. Previous methods for this application on the Horizon 5000 resulted in a 45-minute extraction time and a final extract volume close to 120 mL. Unfortunately, because the final volume was greater, it exceeded the capacity of the 105mm pan, resulting in a two-step and longer evaporation process. The newly optimized method produces a final extract volume of roughly 78 mL with a 35-minute extraction time. The most important conclusion is that the entire volume of the sample extract, as well as any rinses, fit into a single 105 mm aluminum weighing pan, reducing the manual labor associated with the previous method. The end goal was achieved by implementing the optimizations for method 1664B using the Biotage® 5000 and 90 mm disk. This workflow will reduce extraction times, solvent usage and evaporation times which all leads to improved data quality and sample throughput.

## References

Method 1664, Revision B: n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry, available at [www.epa.gov](http://www.epa.gov), (2010).

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