

Determining Oil and Grease In Seawater by Automated Solid Phase Extraction

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Introduction

Oftentimes it becomes necessary to test sea water for oil and grease analytes. Whether it is due to a controlled release of effluent water from a waste water treatment plant (usually during large rain storms) or a large oil spill, there is a need generated for a reliable, automated extraction and evaporation system.

The purpose of this Application Note is to highlight the use of Horizon Technology's (now part of Biotage) SPE-DEX 1000/3000XL Automated Oil and Grease Extractor Systems and Speed-Vap III Evaporation System to test for oil and grease within a seawater matrix. It will prove that the background seen within this matrix is negligible, and will not interfere with test results.

EPA Method 1664¹ is a performance based method which is used to find the total n-Hexanes extractable material (HEM) and the silica-gel treated n-Hexanes extractable material (SGT-HEM) within a sample. This method specifically outlines the steps needed to determine the amount of oil and grease analytes and will be used as the basis for the all extractions within this Application Note.

The SPE-DEX 1000 and 3000XL Automated Extraction Systems were specifically designed to automatically extract oil and grease from a wide range of clean and dirty aqueous samples for EPA Method 1664A or 1664B. The Speed-Vap[®] III Solvent Evaporation System allows for consistent and gentle heating and air flow over the sample to prevent the loss of volatile compounds. The Solvent Trap Recovery System is designed to recover up to 70% of the n-Hexanes solvent vapors for reuse or disposal. Pacific[®] Premium disks are designed to obtain the accuracy and consistency of data needed to comply with method 1664A/ B QC specifications.

In software version 2.2, four extraction methods come preloaded: 26, 27, 28, and 29 along with one purge method: 30. Extraction methods 26 and 27 were developed for use with a 47 mm Pacific Premium disk while methods 28 and 29 are for use with a 90 mm Pacific Premium disk.

Instrumentation

- » Horizon Technology (now part of Biotage)
 - » SPE-DEX[®] 3000XL with Controller v2.2
 - » Speed-Vap[®] III Evaporation System
 - » Solvent Trap[™] Recovery System
 - » Pacific[®] Premium SPE Disks (47 or 90 mm)
 - » Oil & Grease Standard Containing 4 mg/mL Hexadecane and 4 mg/mL Stearic Acid
- » Aluminum Weight Pans, 105 mm
- » Mettler AE 200 (Balance)



Horizon Technology (now part of Biotage) Speed-Vap[™] III Evaporation System, SPE-DEX[®] 3000XL Automated Extractor System, Controller v2.2, and Solvent Trap[™] Solvent Recovery System.

Method Summary

1. 4 L of seawater was sampled from the Atlantic Ocean.
2. Homogenize and split sea water sample into 1 L aliquots.
3. Acidify each bottle with 1:1 Hydrochloric Acid (until pH \leq 2).
4. For matrix spikes, add 5 mL of Oil and Grease Standard to each bottle (total concentration of 40 ppm).
5. Do not add any standard to the water blank sample bottle.
6. Ensure that the vacuum source is set to -25 in. Hg and that the nitrogen source is set to 70 psi.
7. Purge each station to be used on the SPE-DEX 3000XL to ensure the proper flow of solvents.
8. Extract samples using the SPE-DEX 3000XL with 47 mm Pacific Premium SPE Disks and the method in Table 1; or with 90 mm Pacific Premium SPE Disks and the method in Table 2.
9. Pre-weigh one aluminum pan for each sample.
10. Add one extract to each of the aluminum pans.
11. Use the Speed-Vap III Evaporation System to evaporate each extract at a temperature of no more than 40 °C.
12. Weigh each extract's pan and calculate HEM recovery (nominally 40 mg).

Table 1. Controller Method 26 for 47 mm Pacific® Premium Disks.

Prewet Hexane	Time	Prewet Methanol	Time
Dispense	6 sec.	Dispense	6 sec.
Saturate	1 sec.	Saturate	1 sec.
Soak	30 sec.	Soak	30 sec.
Drain	30 sec.	Drain	3 sec.
Air Dry	3 min.		
Solvent	Rinse	Soak	Elute
1. Hexane	4 sec.	5 sec.	15 sec.
2. Hexane	2 sec.	45 sec.	45 sec.
3. Hexane Methanol	2 sec.	45 sec.	30 sec.
4. Discard	2 sec.	20 sec.	1 min.
5. Hexane	2 sec.	45 sec.	45 sec.
6. Hexane	2 sec.	45 sec.	30 sec.
7. Hexane	2 sec.	45 sec.	30 sec.
8. Hexane	2 sec.	45 sec.	1 min.

Table 2. Controller Method 28 for 90 mm Pacific® Premium Disks.

Prewet Hexane	Time	Prewet Methanol	Time
Dispense	10 sec.	Dispense	10 sec.
Saturate	1 sec.	Saturate	1 sec.
Soak	1 min.	Soak	1 min.
Drain	1 min.	Drain	3 sec.
Air Dry	3 min.		
Solvent	Rinse	Soak	Elute
1. Hexane	8 sec.	10 sec.	15 sec.
2. Hexane	7 sec.	45 sec.	45 sec.
3. Hexane Methanol	7 sec.	45 sec.	30 sec.
4. Discard	4 sec.	20 sec.	1 min.
5. Hexane	4 sec.	45 sec.	45 sec.
6. Hexane	4 sec.	45 sec.	30 sec.
7. Hexane	4 sec.	45 sec.	30 sec.
8. Hexane	4 sec.	45 sec.	1 min.

Results

Table 3. Results of 40 ppm spikes and sea water blank for 90 mm Pacific® Premium Disks.

	Initial Wt. (g)	Initial Wt. (g)	Recovery (mg or %)
Matrix Blank 1	6.4490	6.4492	0.2
Matrix Blank 2	6.4707	6.4718	1.1
Matrix Blank 3	6.4423	6.4432	0.9
		Average	0.7
		Deviation	0.5
Sample 1	6.4487	6.4870	95.8%
Sample 2	6.4653	6.5034	95.3%
Sample 3	6.4159	6.4556	99.2%
		Average	96.8%
		Deviation	2.2%

Conclusions

This study utilized seawater from the Atlantic Ocean to highlight the use of Horizon Technology's (now part of Biotage) SPE-DEX 3000XL automated oil and grease extractor system for the testing of oil and grease analytes. The results prove that interferences from typical seawater matrices containing upwards of 35 g/L of salt are negligible and do not effect the precision or accuracy of a spiked matrix sample. In fact, the HEM recoveries shown above fall well within both IPR and OPR acceptance ranges for EPA Method 1664¹.

References

1. Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry; EPA-821-R-98-002; United States Environmental Protection Agency, Office of Water: Washington, DC, February 1999.

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