How Solid Phase Extraction (SPE) Disks Differ for Hexane Extractable Material

Michael Ebitson, Biotage.

Introduction

The quality of the solid phase disk used for extraction is key to the success and ease of an analysis. Pacific® Premium Oil & Grease SPE disks from Biotage for Method 1664/ISO 11349 meet all Initial Precision and Recovery (IPR), Method Detection Limit (MDL) and Ongoing Precision and Recovery (OPR) limits specified by the US EPA, while saving the lab extraction time and solvent volume. Both 47 and 90 mm disks are fast-flowing and have a high-retention capability, which increases efficiency without compromising recoveries.

A number of tests were performed, comparing oil and grease disks from Biotage (Pacific® Premium and Pacific®) with a variety of other competitor's disks, available commercially. Twelve disks of each type from a single batch were tested and to make the comparison as valid as possible, the disks were run using the same method conditions on the same equipment. Automation of the SPE process using the SPE-DEX® 3000XL (Horizon Technologies, now part of Biotage) to apply the sample and elute the hexane extractable material provided the most consistent results.

Experimental

The experimental conditions used for all the disks are shown in Table 1. The larger diameter disks were used in all cases, except when the brand did not offer the larger disk. In that case, the smaller diameter disk was used. The larger diameter disks are more useful for samples with particulates, whereas the smaller diameter disks are useful for samples with less particulate. The disks were evaluated for background, recovery of a standard, and flow rate.

Table 1. Experimental Conditions for Evaluation Experiments

Parameter	
Sample	1 L Purified Water
Vacuum	-20 inches of mercury
90 mm Method	Number 27, stored in controller
47 mm Method	Number 24, stored in controller
Speed-Vap Evaporation	40 °C
Rinse Elution Funnels	3 times w/ ~ 3 mL each
Sample pH	~2
Extractor	SPE-DEX 3000XL Oil & Grease System
Balance	Mettler AE 200

Results and Discussion

Since these methods use a gravimetric determinative step, background contributed by the disk may cause an error, biasing the measurement high. Biotage Pacific® disks yield very little background material and are carefully made to ensure consistency. For hexane-extractable material (HEM) and silica-gel treated material (SGT-HEM) in this method, the method detection limit (MDL) is 1.4 mg/L and the minimum level of quantitation (ML) is 5.0 mg/L, specified in Method 1664B, dated 2010.[1]

The results, shown in Table 2, indicate the background varies widely among the different brands. The MDL could be estimated from the t statistic for 12 replicates (11 degrees of freedom) and standard deviation and would be acceptable for most of the brands. However, the ISO 11349 method is very specific and specifies that a blank value greater than 3 mg/L is unacceptable, ruling out two of the brands.[2]

Table 2. Background for a Variety of Competitive Disks Compared to Pacific® Disks

Disk Brand	Replicates	Background (mg/L)	Standard Deviation (mg/L)
Pacific® Premium	12	1.11	0.49
Pacific®	12	0.03	0.17
Brand 2	12	0.63	0.76
Brand 3	12	4.84	1.58
Brand 4	12	0.78	0.42
Brand 5	12	4.29	0.97
Brand 6	12	0.77	0.38
Brand 7	12	0.66	0.38

Recovery of a standard is a quality control demonstration that helps in evaluating method performance and capability. Table 3 shows recovery of a 40 mg spike into the 1 L purified water sample before the extraction step. The spike consists of a mix of 20 mg n-hexadecane and 20 mg stearic acid. The high recovery and low standard deviation seen with the Pacific® disks was excellent. The ISO 11349 method specifies that recoveries must fall between 90-105% for 100 mg of vegetable oil added to 500 mL of water. The sample used here is more dilute, providing a challenge to the lower concentration values to be determined and providing an excellent demonstration of performance.

Table 3. Recovery of a 40 mg Standard Material

Disk Brand	Replicates	Recovery (%)	Standard Deviation (%)
Pacific® Premium	12	97.7	1.2
Pacific®	12	90.5	2.0
Brand 2	12	90.4	2.3
Brand 3	9	88.7	7.8
Brand 4	12	94.2	2.2
Brand 5	12	93.4	7.9
Brand 6	12	91.5	3.1
Brand 7	12	89.4	2.6

Flow rates are also important in an efficient analysis. With the complex matrices encountered when performing this method it is critical that the samples process to completion in a timely manner. The use of the larger diameter Pacific disk with prefilters and glass wool can preserve the flow of most any sample through the disk. Flow of the blank and standard through the disk is shown in Table 4 and illustrated in Figure 1 to give an indication of the fastest time expected for a sample with few particulates.

Table 4. Processing Time for 1L of Water With and Without Spike

Disk Brand	Replicates	Processing Time (min) Standard	Processing Time (min) Blank
Pacific® Premium	12	1.3	1.3
Pacific®	12	1.9	1.7
Brand 2	12	1.9	1.8
Brand 3	9	12.4	7.4
Brand 4	12	2.5	2.2
Brand 5	12	2.0	2.0
Brand 6	12	4.4	4.6
Brand 7	12	1.8	2.1

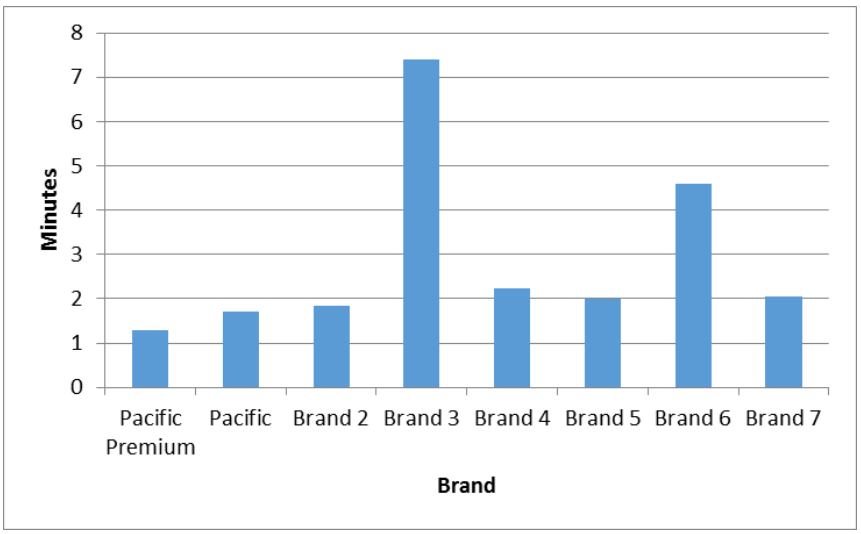

Figure 1. Processing Time of 1 L Water through the Disk in Minutes.

Conclusion

Several different brands of disks were tested against Pacific® Premium and Pacific® disks for blank background, recovery of a standard and speed of processing through the disk. The Pacific Premium and Pacific disks are manufactured with strict quality control to ensure consistency and performance, which was demonstrated with these tests.

References

1. 1664B, US EPA http://water.epa.gov/scitech/methods/cwa/upload/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.pdf (2010).
2. ISO method 11349, Water Quality-determination of low-volatility lipophilic substances-gravimetric method (2010).

EUROPE
Main Office: +46 18 565900
Toll Free: +800 18 565710
Fax: +46 18 591922
Order Tel: +46 18 565710
Order Fax: +46 18 565705
order@biotage.com
Support Tel: +46 18 56 59 11
Support Fax: + 46 18 56 57 11
eu-1-pointsupport@biotage.com

NORTH & LATIN AMERICA
Main Office: +1 704 654 4900
Toll Free: +1 800 446 4752
Fax: +1 704 654 4917
Order Tel: +1 704 654 4900
Order Fax: +1 434 296 8217
ordermailbox@biotage.com
Support Tel: +1 800 446 4752
Outside US: +1 704 654 4900
us-1-pointsupport@biotage.com

JAPAN
Tel: +81 3 5627 3123
Fax: +81 3 5627 3121
jp_order@biotage.com
jp-1-pointsupport@biotage.com

CHINA
Tel: +86 21 68162810
Fax: +86 21 68162829
cn_order@biotage.com
cn-1-pointsupport@biotage.com

KOREA
Tel: + 82 31 706 8500
Fax:+ 82 31 706 8510
korea_info@biotage.com
kr-1-pointsupport@biotage.com

INDIA
Tel: +91 22 4005 3712
india@biotage.com

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