



New Triple Layer SPE Filter for EPA Method 1664A

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EPA has proposed to withdraw two chlorofluorocarbon-based Oil and Grease liquid liquid extraction methods and replace them with approved EPA Method 1664A which employs n-hexane as the extraction solvent. Liquid liquid extraction (LLE) is an allowable extraction technique but the lower density of n-hexane creates a cumbersome procedure prone to emulsions and poor recovery/reproducibility. Solid phase extraction (SPE), also an allowable extraction technique offering the greatest cost savings potential has not yet lived up to expectations due to slow (> 10 min) and/or incomplete filtration of the QC samples.

Below we demonstrate Xenosep® SPE Filters provide superior EPA Method 1664A recovery, precision and MDL data with QC Sample filtration times of approximately 10 s.

Apparatus & Reagents

SPE Filter: Xenosep® 47 mm O&G Filter

Glassware: Xenosep® 47 mm SPE System

Reagents and other apparatus per EPA 1664A (1).

Experimental Conditions

The PAR (Precision and Recovery) standard (4 mg/mL 1:1 hexadecane:stearic acid) was prepared according to the method. The IPR (Initial Precision and Recovery) study was determined with 4 samples at 40 mg/L concentration. The MDL (Method Detection Limit) study was determined with 7 samples at 8 mg/L concentration.

Results

IPR Recovery	94.8%
IPR Precision	2.7%
MDL	0.65 mg
Filtration Time	10 s

Discussion

Filtration of the QC sample has been typically slow and/or incomplete when using SPE filters. Stearic acid particulates reform when the PAR standard contacts the preacidified water sample. During filtration, these particles are large enough (1–10 µm) in size and quantity to prematurely clog and significantly reduce sample flow through the SPE filter.

Xenosep® SPE Filters with triple layer construction are optimized for filtration and extraction of EPA Method 1664A samples. As can be seen in Figure 1, Oil and Grease analytes are retained by the chemically treated fibers of the Adsorbent Layer. The Intermediate Prefilter Layer traps particulates in the 1–5 µm range while the Coarse Prefilter Layer traps 5–25 µm particulates. The combination of these two prefilter layers effective

ly suspends the stearic acid particulates throughout the SPE filter matrix and enables filtration of the QC sample in approximately 10 s. Trapping particulates at each layer also improves SPE recovery and precision performance. This is due to increased air flow through and more efficient removal of residual water from the SPE filter prior to Oil and Grease analyte extraction with n-hexane.

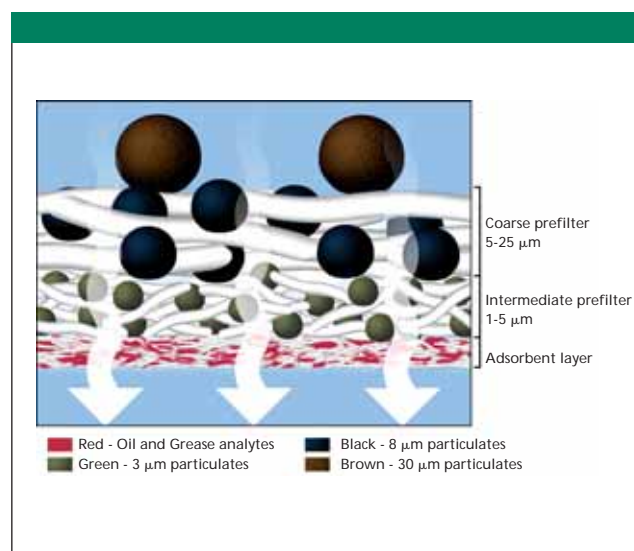


Figure 1: Cut away view of the triple layer Xenosep® SPE Filter.

Conclusion

Xenosep® SPE Filters with Triple Layer Construction offer significant advantages for EPA Method 1664A. Besides superior recovery, precision and MDL data, QC sample filtration times that were previously measured in minutes and/or hours can now be measured in seconds. Xenosep® SPE Filters with high loading capacity and fast flow rates should enable the widespread use of SPE filters with EPA Method 1664A.

References

- (1) EPA Method 1664, Revision A: 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. EPA-821-R-98-002, PB99-121949, February 1999.

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