



Reagent-free Solid Phase Extraction Disk for Method 1664

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From EPA 413.1 to EPA 1664B



- Replaced Freon with n-hexane
- 1664 is now the only method approved for testing wastewater for oil & grease
- Use of solid-phase extraction (SPE) has been allowed without a demonstration of equivalency
- Oil & Grease still a “method-defined analyte” though is now considered Hexane Extractable Material (HEM)

Method-Defined Analyte



- HEM vs. Oil & Grease
- Method modifications are extremely limited
- Modifications cannot alter the chemistry of the method
- Modifications cannot adversely affect HEM recoveries

Solid Phase Extraction (SPE)



- SPE allowed in Revision A of Method 1664 without a demonstration of equivalency
- Utilizes an extraction disk impregnated with C-18 as the absorbent resin
- The C-18 in the disk is “activated” with a polar solvent (methanol) prior to sample filtration
- Sample is filtered through the disk which retains the HEM
- HEM is then eluted off of the disk with n-hexane
- Hexane + HEM collected in a pre-weighed flask or dish and the n-hexane driven off by heat to leave the HEM behind

Potential Problems with SPE

- Method 1664 does allow for methanol or another polar solvent to be used to activate the extraction disk, however “at no time will residual polar solvent introduce the target analyte into the sample, collocate with or be collected with the extraction solvent, n-hexane.”



Potential Problems with SPE

- Filter disk activation – how to avoid the use of “co-solvents”?
- Active sites on C-18 resin are typically closed and hydrophobic. Must be activated by methanol to open these sites and allow water to filter through.



UltraPrep™ SPE Filter Disk

- C-18 resin works for both hydrophilic and hydrophobic retention.
- No activation step required.
- C-18 phase remains active even when dry.



UltraPrep SPE Filter Disk



- Means the elimination of methanol
- No risk of co-solvent contact with sample or hexane
- Guaranteed method compliance

Does UltraPrep Work?

Study #1

- MDL study performed*
- 5 Method Blanks
- 5 LCS (40 mg/L) stds
- 10 “Real World” samples*
- 2 MS*
- 2 MSD*
- Performed on StepSaver

*Compared results to
UltraFlow filter results



MDL Study Results

UltraFlow Disks	UltraPrep Disks
Rep 1 = 5.0 mg/L	Rep 1 = 4.6 mg/L
Rep 2 = 4.8 mg/L	Rep 2 = 4.6 mg/L
Rep 3 = 5.2 mg/L	Rep 3 = 4.4 mg/L
Rep 4 = 4.8 mg/L	Rep 4 = 4.7 mg/L
Rep 5 = 4.8 mg/L	Rep 5 = 4.9 mg/L
Rep 6 = 4.9 mg/L	Rep 6 = 4.7 mg/L
Rep 7 = 4.9 mg/L	Rep 7 = 4.5 mg/L
Calculated MDL = 0.46 mg/L	Calculated MDL = 0.50 mg/L

Minimum MDL
required by Method
1664 is ≤ 1.4 mg/L.

Method Blanks and LCS Results



Method Blank	Laboratory Control Standard
Rep 1 = 1.3 mg/L	Rep 1 = 35.8 mg/L
Rep 2 = 1.1 mg/L	Rep 2 = 37.9 mg/L
Rep 3 = 1.6 mg/L	Rep 3 = 35.0 mg/L
Rep 4 = 2.3 mg/L	Rep 4 = 35.8 mg/L
Rep 5 = 0.5 mg/L	Rep 5 = 39.2 mg/L
RL = 5.0 mg/L	LCS TV = 40 mg/L
	Avg % recovery = 91.9%

Real World Sample Comparisons



Sample ID	UltraFlow	UltraPrep	%RPD
WW Sample #1	8.5 mg/L	8.8 mg/L	3.5%
WW Sample #2	17.2 mg/L	16.2 mg/L	6.0 %
WW Sample #3	60.0 mg/L	58.8 mg/L	2.0 %
WW Sample #4	22.4 mg/L	23.8 mg/L	6.1 %
WW Sample #5	9.30 mg/L	9.60 mg/L	3.2 %
WW Sample #6	29.1 mg/L	30.9 mg/L	6.0 %
WW Sample #7	22.3 mg/L	21.8 mg/L	2.3 %
WW Sample #8	19.4 mg/L	17.7 mg/L	9.2 %
WW Sample #9	104 mg/L	107 mg/L	2.8 %
WW Sample #10	26.5 mg/L	23.9 mg/L	10.3 %

MS / MSD Result Comparisons

Sample ID	UltraFlow	% Recovery	UltraPrep	% Recovery
MS #1	72.7 mg/L	43%	82.0 mg/L	52%
MSD #1	73.5 mg/L	45%	81.2 mg/L	50%
MS #2	48.5 mg/L	97%	44.9 mg/L	91%
MSD #2	49.6 mg/L	100%	48.8 mg/L	100%

- A 40 mg/L spike was added to each sample.
- MS#1 and MSD#1 exhibited matrix interferences in spike recoveries for both the UltraFlow and UltraPrep filters.
- MS#2 and MSD#2 showed acceptable recoveries in both the UltraFlow and UltraPrep filters.

Conclusions



- MDL passed Method 1664 requirements.
- Method Blanks showed consistent cleanliness and passed laboratory requirements.
- LCS recoveries were well within the method defined limits of 78 – 114%.
- Duplication of samples between UltraFlow disks and UltraPrep disks were all < 11%.
- Matrix Spike recoveries were both consistent.

Applications for Field Filtration

Study #2

- 10 x 40mg/L (LCS) standards, 10 x 5mg/L (MDL) standards, and 10 x Method Blanks (MB) filtered through UltraPrep filters
- After filtration filter and apparatus stored along with sample bottle at 4°C
- Every few days 2 of each filter removed and HEM eluted
- Results recorded and checked for HEM stability on UltraPrep filter



Stability Results



# of days held for extraction	MB	MDL Std (5mg/L)	% Rec	LCS Std (40mg/L)	% Rec
5 days	0.7 mg/L	5.3 mg/L	106%	38.3 mg/L	96%
5 days	0.5 mg/L	4.7 mg/L	94%	37.8 mg/L	95%
9 days	3.9 mg/L	5.5 mg/L	110%	41.5 mg/L	104%
9 days	3.4 mg/L	5.4 mg/L	108%	39.7 mg/L	99%
13 days	-0.3 mg/L	5.2 mg/L	104%	38.7 mg/L	97%
13 days	0.3 mg/L	5.3 mg/L	106%	38.7 mg/L	97%
21 days	0.7 mg/L	4.1 mg/L	82%	32.7 mg/L	82%
21 days	0.4 mg/L	5.1 mg/L	102%	34.2 mg/L	86%
30 days	1.2 mg/L	6.1 mg/L	122%	32.9 mg/L	82%
30 days	2.2 mg/L	6.1 mg/L	122%	34.1 mg/L	85%

Conclusions



- LCS recoveries all showed stability over a 30 day period.
- Began to see the MB and MDL standard results creep up over time.
- Could have some implications for field filtration and save laboratories money on sample shipping costs.

Credit Where Credit is Due



A big thank you to Davis & Brown Laboratories of Florence, SC who performed the analyses of these two studies, especially Charles Green (chemist) and Zachary Ward (college student).

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QUESTIONS?

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