Oil and Grease: Past, Present, and Future(?)

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June 20, 2012
What is Oil & Grease?
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
From EPA 413.1 to EPA 1664A

- Replaced Freon with n-hexane
- 1664 adopted in 1999 and we are now up to Revision B
- EPA withdrew all Freon methods from 40 CFR part 136 in 2007
- 1664 is now the only method approved for testing wastewater for oil & grease
What did Method Change Mean?

- Oil & Grease still a “method-defined analyte” though is now considered Hexane Extractable Material (HEM)
- The term "total petroleum hydrocarbons" (TPH) has been replaced by "non-polar material" (NPM) to avoid confusion with TPH measurements by other methods
- The requirement for a matrix spike duplicate (MSD) has been changed to a suggestion
- The size of an analytical batch has been increased to a maximum of 20 samples per discharge or waste stream
- Use of solid-phase extraction (SPE) has been allowed without a demonstration of equivalency
LLE vs. SPE

Liquid-Liquid Extraction
• 3 shakes of 30 mL of solvent
• Higher analyst exposure to n-hexane
• Prone to emulsions
• Relies on analyst precision and accuracy
• Hexane floats!

Solid-Phase Extraction
• Little solvent usage (as little as 40 – 50 mL)
• Less solvent = less exposure
• Less labor intensive
• Frees up analysts to do other things in the lab
• More consistent results
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
Manual SPE
Problems with SPE

• Turbid samples, turbid samples, turbid samples.
• How do you know how long it takes to filter a sample?
• Wasted time or voided results.
• Filter disk activation – how to avoid the use of “co-solvents”?
• How do you get residual HEM from the sample bottle?
• In the past it was easier to deal with these issues by running SPE manually rather than by automated instrumentation.
The SPE-XPress for Automation of 1664
Dealing with Turbid Samples and Extended Filtration Times

UltraFlow extraction disks

Fluid Sensor
Filter Activation and Eliminating Co-solvents

- 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.”
- SPE-XPress handles this by replacing the methanol with DI Water prior to the introduction of sample.
- Ensures the disk remains wet and the sample does not come into contact with methanol.
- All methanol is directed to waste.
Getting Residual HEM from Bottle

- SPE-XPress gives users flexibility on volume of n-hexane and total # of sprays to fully rinse the inside of the sample bottle
Reduction of Analyst Exposure to n-Hexane

- SPE-XPress software allows user to configure total amount of n-hexane used in each step of the process.
- There are minimums and maximums for each step giving a total n-hexane volume range of 46 – 170 mL.
- Evaporation takes place in the unit itself, eliminating a transfer step to a secondary evaporation device.
- Each chamber is equipped with it’s own exhaust fan to move hexane fumes out of the chamber. The fumes are then directed out of the chamber and out of the instrument via the exhaust port.
- No need to have the instrument in a fume hood.
How Long Does the Extraction Take?

- Pre-Rinse step = 2 – 4 minutes.
- Disk Activation step = 1 – 2 minutes.
- Sample Filtration = 15 – 20 minutes (will vary depending on sample type). This step includes a 10 minute drying of the filter step after filtration is complete.
- Sample Bottle Rinse step = 3 – 6 minutes.
- Sample Elution Step = 12 – 18 minutes.
- Evaporation Step = Ongoing during the Elution Step. Will be determined by total volume of n-hexane used.

- Total time is 45 – 60 minutes per sample on average.
Running Multiple Samples

• Once the elution step is complete the user can begin a second test on the same chamber.

• A new sample bottle and extraction disk are added and all disk preparation and sample filtration will begin.

• SPE-XPress will not begin the elution step until the previous pan is removed from the chamber.
SPE-XPress

Handle in Upright Position

Filter Assembly

Sample Bottle

Station Handle
What Does the Future Hold?

• Improvements to the SPE-XPress

• Improvements to the filter material

• Possible approval of a new solventless method
Questions?

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