Xenosep® SPE Filter Summary
EPA Method 1664A
Oil and Grease

- **Select Xenosep® SPE Filter Diameter**

Heavily contaminated samples may require a 90 mm filter, allowing the sample to settle out overnight and/or analysis of a smaller sample volume. Very clean samples may only need the 33 mm filter. The 47 mm filter is appropriate for most samples and is a good starting point to determine the need for the other sizes. A vacuum > 20" Hg with maximum free air flow > 3 SLPM should be used for all SPE filters unless otherwise indicated in a specific operating protocol.

- **Prepare Sample**

Bring the sample to room temperature. Either mark the sample at the water meniscus or weigh the bottle for later determination of sample volume. Weighing will be more accurate. Verify/acidify sample to pH < 2.

- **Prepare Xenosep® Apparatus and SPE Filter**

Thoroughly wash all components upon receipt. Insert the waste collection tube into the 1 L flask. Attach the glass holder and place the support and SPE filter into the recess so the fluffy side of the filter faces you. Tightly press the Xenosep® coupler (w/2 o-rings) onto the SPE filter and firmly attach funnel by squeezing the parts together. Secure filtration apparatus with clamp. Thoroughly rinse the side walls of the assembled apparatus with approximately 10 mL of n-hexane (30 mL for 90 mm) until the SPE filter is completely submerged in the solvent. Wait 5 seconds and briefly apply vacuum to pull solvent through the SPE filter into the waste collection tube. Repeat. After 2nd rinse, apply vacuum for 15 seconds (30 seconds for 90 mm) to remove any residual solvent and dry the SPE filter. Note: Optional prefilters and/or prefilter fibers may be inserted into the Xenosep® coupler before funnel attachment to reduce filtration times of extremely high particulate containing samples.

- **Condition SPE Filter**

Step one: Pre-wet the SPE filter with 10 mL of isopropanol (30 mL for 90 mm) until the SPE filter is completely submerged in the solvent. Note: Do not allow the SPE filter to dry out. Wait 5 seconds and briefly apply vacuum to pull the isopropanol through the SPE filter into the waste collection tube. Remove the waste collection tube and discard waste solvent appropriately. Step two: Cover the SPE filter with 20 mL DI water (60 mL for 90 mm) and briefly apply vacuum to pull the DI water through the SPE filter into the 1 L Flask. Note: If the SPE filter becomes dry at any point during conditioning, repeat the conditioning process beginning at Step one.

- **Extract Sample**

Slowly pour sample into funnel and turn on vacuum. Minimize sample contact with the funnel as much as possible by maintaining the level of sample in the funnel below the Xenosep® logo or at approximately 150 mL. For samples containing a high amount of sediment, decant and pour separated portion of the sample into the funnel first. Just before dryness, add the portion of sample containing the heaviest amount of sediment. Scoop out any sediment left in the bottle onto the SPE filter. Use DI water to rinse any sediment remaining in bottle and/or on the funnel walls to the surface of the SPE filter. If used, leave spatula resting in the funnel.

- **Remove Water from SPE Filter and Trapped Sediment Layer**

After the sample is completely filtered, continue drying the SPE filter with vacuum for at least 4 minutes but not longer than 8 minutes (8 and 16 minutes respectively for 90 mm) as loss of recovery may occur from the more volatile analytes. If there is a trapped sediment layer on the surface of the SPE filter, add small amounts of sodium sulfate to the surface of the SPE filter and carefully mix with a spatula until the sediment/sodium sulfate layer appears dry, granular and free-flowing. After the SPE filter drying step is complete, turn off vacuum and use a glass funnel to transfer the solids into the Xenosep® inline sodium sulfate column below. Rinse the spatula and/or funnel used for mixing/scooping/transfer with n-hexane and add it to the final extract.

- **Analyte Recovery and Water Removal from Hexane Extract**

Insert a Xenosep® inline sodium sulfate column into the Xenosep® SPE elution apparatus and attach a pre-tared, Xenosep® flat sided erlenmyer flask to the outlet. Add 10 mL of n-hexane (30 mL for 90 mm) to the sample bottle and vigorously rinse side walls and cap in a horizontal, circular motion for 10 seconds. Use the 15 mL Xenosep® pipette to transfer the n-hexane extract from the neck of the sample bottle to the apparatus and thoroughly rinse the funnel side walls at least 3 times with each transfer. Leave pipette resting in funnel and repeat two more times. Briefly apply vacuum to carefully transfer the n-hexane extract from SPE filter, through the inline sodium sulfate column into the pre-tared boiling flask. Rinse funnel with 10 mL of n-hexane (30 mL for 90 mm) for the final wash of the apparatus and then briefly apply vacuum for 5 seconds to complete the transfer.

- **Determine Hexane Extractable Material (HEM)**

Distill n-hexane from the pre-tared, flat sided boiling flask using the Xenosep® distillation apparatus. Move the pre-tared, Xenosep® flat sided erlenmyer flask to the short end of the condenser and attach the round flat bottom flask to the long end. Submerge the flat sided erlenmyer flask into a 1 L beaker containing minimum 85°C water so the Xenosep® logo is covered. As soon as part of the flask bottom appears dry, remove flask from the water bath and wipe the outside surface to remove any moisture and fingerprints. Place the flask on its flat side in a hood to ensure the gentle removal of any residual solvent. When dry, remove the flask and place it in a desiccator for a minimum of 30 minutes until a stable weight can be obtained. Record weight of the boiling flask and compare value to the tare weight. The difference is the amount of HEM present in the sample and is expressed in mg/L. Note: Optional aluminum pans could be used in this step as well – contact Xenosep Technologies for more information.

- **Determine Silica Gel Treated – n-Hexane Extractable Material (SGT-HEM)**

Re-dissolve “dried” HEM from flat sided flask above with 85 - 90 mL n-hexane. Quantitatively transfer to a 100 mL volumetric flask and dilute to mark with n-hexane. Aliquot an appropriate amount of extract, dilute and return to flask as described in EPA Method 1664A. Add 3 gm of anhydrous silica gel for every 100 mg HEM or fraction thereof. Stir for 5 minutes and filter though a 2nd inline sodium sulfate column into a 2nd pre-tared flat sided flask. Rinse inline silica gel/inline sodium sulfate column with 10 mL n-hexane to complete the transfer. Distill n-hexane and obtain stable weight per HEM section above. Record weight of the boiling flask and compare value to tare weight. The difference is the amount of SGT-HEM present in the sample and is expressed in mg/L.

- **Clean-In-Place and Inline Sodium Sulfate Column**

After analysis, discard the inline sodium sulfate column. Rinse the tip of the extraction apparatus with very small amounts of acetone and hexane (< 1 mL each). Collect and discard all waste solvents according to appropriate local, state and/or federal regulations.