EPA Method 1664 Using Resprep® Oil & Grease Disks and Flow Filters

cat.# 26022 and 26024

Introduction:
The following instructions are general guidelines for the extraction of oil and grease from water. Sample volume, solvent type, pH adjustment and conditioning may be changed to adapt to specific methods as needed.

List of materials: Disk: 26022; Filter: 26024; 25 mm Filter: 26145. If using glass filter beads, see end note for procedural modifications.

Procedure

Sample Preparation:
1. Adjust sample to pH 2.0 with 6N HCl.
2. If sample is high in suspended solids, allow solids to settle. When adding the sample, tilt the sample container to allow particulates to settle on one side. This is suggested to aid in the decanting of the liquid portion of the sample.

Extraction Disk Conditioning:
Proper disk conditioning is critical for a successful extraction. Conditioning provides a good interface between the sorbent and the sample matrix. Failure to condition the disk properly may result in erratic and low recoveries.

1. Place disk on manifold, ripple side up. Connect reservoir securely to disk base.
2. Place the Resprep® Flow Filter in the bottom of the reservoir, flush with the edge of the glass.
3. Wash the disk with 25 mL of *n*-hexane. Note: Always run solvents down the sides of the glassware when washing or eluting. Apply a light vacuum (~5 in. Hg) and pull approximately 1mL through the disk. Vent the vacuum and allow the disk to soak for two minutes.
4. Apply vacuum to pull the remaining solvent through the disk. Allow the disk to dry.
5. Repeat steps 3 and 4.
6. Add 25 mL of methanol to the reservoir. Apply a light vacuum (~5 in. Hg) and pull approximately 1 mL through the disk. Vent the vacuum and allow the disk to soak for one minute.
7. Apply vacuum and pull methanol through the disk until the methanol surface is 1-2 mm above the surface of the disk or prefilter.
8. Add 10 mL of reagent-grade water to the reservoir. Apply a light vacuum and pull the water through the disk until the surface is covered with about 1-2 mm of water.
9. Repeat steps 7 and 8.

Note: It is important that the disk is not allowed to dry out before introducing the sample. Drying of the disk at this point could lead to decreased yields.

Sample Extraction:
1. Pour or decant the sample into the reservoir and apply vacuum. Decant and extract as much liquid as possible before adding sediment to the reservoir. Do not let the disk go dry before adding sediment. Note: Use an appropriate vacuum to keep the extraction time to not less than 10 minutes, as faster extraction times will reduce recoveries.
2. After sample extraction is complete, remove as much residual water as possible from the disk by applying vacuum to dry the disk for 10 minutes.

Sample Drying:
1. Use acetone (5-6 mL) to remove any remaining water from the disk.
2. Start with 5 mL. Leaving an open vacuum, add acetone around the lower portion of the reservoir and to the top of the disk. Apply maximum vacuum to dry the disk for 5 minutes. Do not allow acetone to sit in the reservoir.
3. If 5 mL does not remove all remaining water, try 6 mL, but do not exceed this amount.

Sample Elution:
1. Put receiving vessel in place. Elute twice with 25 mL of *n*-hexane. Smaller volumes of solvent may be used if the elution techniques have been validated. When adding elution solvent, rinse down the sides, washing the walls of the reservoir in the process.
2. Carefully apply vacuum to pull a few drops of *n*-hexane through the disk, and then release the vacuum. Allow the *n*-hexane to soak the disk for two minutes before applying full vacuum to remove residual *n*-hexane and dry the disk.
3. Repeat steps 1 and 2 with a second aliquot of eluting solvent.

   *Optional Step: It may be desirable to further dry and filter the combined hexane eluates before transferring to a preweighed vaporation dish. Pass the hexane through a sodium sulfate containing tube (cat.# 26207), and a 0.45 µm PTFE syringe filter (cat.# 26145), connected to the outlet of the tube.
4. Transfer eluted hexane sample to the preweighed evaporating dish. Rinse collection vessel with two more 5 mL aliquots of *n*-hexane, add rinse to evaporating dish.
5. Evaporate the *n*-hexane, at a temperature of 30-40 °C, from the preweighed dish until a constant weight is reached.

End Note: when using glass filter beads instead of the prefilter, modifications to the procedure are required. Due to the difference in physical properties, it is necessary to reduce solvent use. All quantities of the solvents used should be reduced to 10 mL instead of 25 mL during the disk conditioning and sample elution. In the sample drying, start with 4-5 mL of acetone. If there is remaining water in the sample, try using up to 6 mL.

Call Technical Service at 800-356-1688 or 814-353-1300, ext. 4 (or your Restek representative) if you have any questions about this product or any other Restek product.

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