

Resprep™-C8-47 SPE Disks

(cat.# 24048)

Physical Description & Data

Disk appearance:	Circular white rigid disk
Disk diameter:	47mm
Disk thickness:	0.6mm
Disk composition:	Glass fiber embedded with surface-modified silica
Void volume:	500 µL
Particle shape and size:	Irregular, 30µm (average particle diameter)
Silica pore size:	70 Å average
Flow-through porosity:	5µm
Disk retention capacity:	Approximately 20mg

Operating Instructions

IMPORTANT: Apparatus. When using rigid, glass fiber disks, it is important to use disk holders that provide an adequate seal around the edge of the disks, for proper sampling and to prevent leaks. If using a standard aluminum funnel clamp or any system with questionable sealing, a very slight vacuum pressure should be maintained at all times during the pretreatment and elution steps. Using -1 to -5mmHg will provide enough negative pressure on the disk to prevent leaking from the edges of the disk, without allowing the disk to dry out. The disks should not be allowed to soak in contact with liquids, with the vacuum turned completely off, when using the systems described above.

General Description: Resprep™ disks consist of glass fiber embedded with bonded silica.

Bonded Phase: Resprep™-C8-47 disks contain C8 (octyl) bonded phase.

Capacity: The capacity of a Resprep™-C8-47 disk is approximately 20mg for a well-retained compound. The disk will have a lower capacity for poorly retained compounds (i.e., for which the bonded phase has poor selectivity). The total capacity of the disk includes all analytes and interferences that are retained.

Sample Volume: Sample volumes of one liter or more can be processed through a Resprep™-47 mm disk.

Flow Rate: Typical Resprep™ disk procedures can be run at flow rates from 50 to 150 mL per minute for clean water samples. Flow rate through a Resprep™-47 mm disk is dependent upon the differential vacuum level that is applied and the particulate content of the sample matrix. Control of flow rate depends primarily upon vacuum level control.

Vacuum Level: Flow rate varies directly with the differential vacuum strength. For example, clean water samples run on Resprep™ disks will flow at 75-100mL per minute at approximately 10 in. Hg (35 kPa) of vacuum. High vacuum levels (25 in. Hg, 85 kPa) are typically used for disk drying procedures or high-particulate samples. To stop flow through the disk, it is important to release vacuum throughout the entire system, rather than only shutting off the vacuum source.

Solvents: The Resprep™ disk is inert in the presence of virtually all organic solvents. Resprep™ disks can typically be run at a pH range of 2.0 to 7.5. For short residence times, the pH range may be extended from 0 to 10. Elution solvents should be chosen to provide: (1) strong selectivity for the analyte of interest, and (2) compatibility with subsequent procedures, such as evaporation or derivatization and the analytical detection method. For C8 bonded phase, the elution solvent is typically a moderately polar or nonpolar organic solvent. Examples of solvents include ethyl acetate, methanol, acetonitrile, dichloromethane, and hexane.

Particulate Matter: Resprep™ disks provide excellent depth filtration performance when processing samples that are high in particulate matter. Samples that are high in suspended solids should be run at maximum available vacuum (up to 25 in. Hg, 85 kPa).

Interferences: Care must be exercised when performing trace organic analysis that glassware, equipment, solvents, and reagents are free of contaminants. Resprep™ disks, if used according to procedure, will not contribute analytical interferences. Glassware and equipment must be kept clean at all times. Solvents and reagents must be trace organic grade or better. Other consumables, such as filter paper, disposable pipets, or other materials used in the laboratory should also be free of organic contaminants.

Procedures

1. Sample Pretreatment

Purpose: To modify the sample so that conditions are ideal for extraction of the analyte of interest. This can include weakening the sample matrix to reduce the solubility of the analyte of interest, modification of the analyte itself, or modifying the sample matrix to improve interactions with the bonded phase.

Examples: The following are a variety of procedures that, depending upon the specific analyte and matrix, may improve extraction efficiency when used singly or combined. (1) Add methanol to the sample to a final concentration of 0.5% to maintain conditioning of the C8 surface while running a one liter water sample. (2) Adjust sample to pH 2 as a preservative step prior to extraction and analysis. (3) Remove particulate matter by filtration or decanting. (4) Add internal standards if required. (5) Adjust pH and increase ionic strength to decrease solubility of polar analytes.

2. Apparatus Assembly

Purpose: To properly position the Resprep™ disk for maximum extraction efficiency and no leakage.

Example: Refer to the detailed instructions for the vacuum extraction equipment. Insert Resprep™ disk WRINKLED SIDE UP.

3. Disk Precleaning

Purpose: To remove potential interferences by washing the Resprep™ disk with elution solvent before use.

Example: Add 5mL of elution solvent to the disk. Allow the solvent to soak the disk for approximately 1 minute. Draw the solvent through the disk. Dry the disk under vacuum at 15 in. Hg (51 kPa) for 5 minutes to remove all solvent.

4. Disk Conditioning

Purpose: To prepare the Resprep™ disk to ensure optimal extraction of the analyte of interest. Conditioning typically involves a prewetting procedure with an organic solvent such as methanol, followed by solvent exchange to match the sample matrix. Once the disk has been wetted, it is critical that the disk is not allowed to go dry prior to the addition of the sample. Conditioning solvents should be chosen to sufficiently wet the hydrophobic bonded phase, yet be water miscible. Typical solvents include methanol, acetonitrile, and isopropyl alcohol. **Caution:** If the conditioning step is omitted or is performed improperly, effects such as slow flow rates, low recoveries, and erratic results can occur.

Example: Add 5mL of methanol to the disk and let the disk soak for approximately 1 minute. **Do not allow any air to pass through the disk or reach the top surface of the disk.** Add 5mL of water to the disk. **Note:** It is preferable to leave extra liquid above the disk rather than to allow any air to contact the surface of the disk.

5. Sample Addition

Purpose: To apply the sample to the Resprep™ extraction disk for maximum extraction efficiency.

Example: Apply the sample to the disk (**NOTE: See IMPORTANT: APPARATUS section on page 1**), adding it directly to the film of water left on the disk from the conditioning step. Adjust the vacuum to approximately 7.5 in. Hg (25 kPa) to achieve a flow rate of 75-100mL per minute for a clean water sample (the vacuum level may be increased as necessary for samples containing suspended solids). Once the sample addition has begun, the disk must not go dry until the entire sample has been drawn through the disk.

6. Disk Drying

Purpose: To remove as much water as possible from the Resprep™ disk prior to elution. This is especially important if the elution solvent is water immiscible. Incomplete drying can result in ineffective wetting of the disk with the elution solvent and excess water in the final sample eluate.

Example: Dry the disk under vacuum at approximately 15 in. Hg (51 kPa) for 5 minutes, or longer if necessary. It is essential to determine the maximum drying time before loss of analytes begins. Following elution, it may be necessary to pass the sample eluate through anhydrous sodium sulfate to remove all residual water.

7. Analyte Elution

Purpose: To selectively elute the analyte(s) of interest from the Resprep™ disk with maximum efficiency. The elution solvent should be strong with respect to the analyte (i.e., the analyte will preferentially dissolve in the elution solvent rather than remain on the bonded phase surface). The elution solvent should also be compatible with the subsequent steps in the procedure, such as derivatization, concentration, or analysis. Examples of typical elution solvents include methanol, acetonitrile, ethyl acetate, dichloromethane, and hexane. The volume of solvent used should be minimized to provide the greatest concentration factor, but should be large enough to completely saturate the disk and take into account the dead volume of the apparatus. For some procedures (particularly those involving hydrophobic analytes), the sample bottle and apparatus reservoir should be rinsed with the elution solvent, which is then added directly to the disk as part of the elution procedure.

Example: Place a suitable collection vessel below the disk without disturbing the placement of the disk in the apparatus. Add 5mL of ethyl acetate to the disk, and immediately draw the solvent through the disk into the collection vessel at low vacuum (1-2 in. Hg, 3-7 kPa). If necessary, additional 5mL aliquots of solvent can be added to the disk and drawn through under low vacuum to complete the elution process.

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



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