

Instructions for Use

Product Description

Empore[™] Solid Phase Extraction (SPE) Disks provide an efficient alternative to liquid/liquid extraction for sample preparation. A proprietary process is used to entrap adsorbent particles into a matrix of PTFE to create a mechanically stable sorbent disk. The disks can be used for purification and concentration of analytes from aqueous samples.

Empore SPE disks provide a sample prep solution for large volume aqueous samples. The disk format provides a large surface area for sorbent/sample contact. Fast flow rates and high throughput may be realized with use of an Empore solid phase extraction disk.

Empore extraction disks are available in a variety of sorbent chemistries to complement most analytical applications. Each sorbent exhibits unique properties of retention and selectivity for a particular analyte. The choice of which sorbent is best for a particular method will be influenced by the difference in chemical nature of the analyte from the sample matrix and the cleanliness of the resulting chromatography.

Product Characteristics

The Empore SPE disks are available in 47 and 90 mm diameters. Empore 47 mm disks are efficient at processing relatively clean samples (samples with low suspended solids). Empore 90 mm disks are efficient at processing larger volume samples or samples with greater amounts of suspended solids.

Empore[™] Filter Aid 400 and/or prefiltration may be helpful if the sample contains excessive particulates. If the sample contains a high concentration of suspended solids, allow the particles to settle (overnight if necessary). Tilting the sample container to allow particulates to settle on one side is suggested to aid in decanting only the liquid portion of the sample. Particulates can be added to the reservoir after most of the sample has been processed. The 90 mm disk is recommended for samples with excessive particulates or to reduce sample processing times.

Empore SPE disks are high density (HD) membranes composed of chromatographic particles averaging 10-12 μ m in size. The high density membranes are designed for maximum extraction efficiency with minimal elution volumes for samples that have less matrix interference.

Sorbent	Suggested Applications 47			Prod 47 mm	Product Number 47 mm 90 mm	
C8 Bonded Silica	EPA Method 549.1 Diquat and Paraqu	at		2214	2314	
C18 Bonded Silica	EPA Methods • 506 Phthalate & Adipate Esters • 508.1 Chlorinated Pesticides, Herbicides and Organohalides	 525.2 Semi-Volatile Organic Compounds 550.1 Polynuclear Aromatic Hydrocarbons 	 608 ATP 3M0222 Organochlorine Pesticides and PCBs 1613B Dioxins and Furans Other EPA Methods 	2215	2315	
SDB-XC Polystyrenedivinylbenzene	• EPA Method 515.2 Chlorinated Acids		2240	2340		
SDB-RPS Polystyrenedivinylbenzene Reverse Phase Sulfonated	• Explosives Residues (HDX, RDX)			2241	2341	
Cation Exchange - SR	Metals, Amines			2251	Not Available	
Anion Exchange - SR	• EPA Method 548.1 Rev. 1 Endothall	• EPA Method 552.1 Rev. 1 Haloacetic Acids and Dalapon	 Other analytes containing carboxylic acid groups 	2252	2352	
Oil & Grease	• EPA Method 1664 Rev. A n-Hexane Extractable Materials		2270	2370		
Chelating	Divalent metals and other divalent cations		2271	Not Available		
Activated Carbon	N-nitrosodimethylamine (NDMA)	• Water-soluble or volatile analyte	s such as oxamyl and methamidophos	2272	2372	

Filter Aid Filtration Media

Accessory

Product Number FA400



Empore[™] Disk Technology

Empore[™] Solid Phase Extraction Disks are produced by trapping sorbent particles within an inert matrix of polytetrafluoroethylene (PTFE). The resulting particle-loaded membrane yields a denser, more uniform extraction bed than can be achieved with traditional loosely packed SPE particles. The result is improved mass transfer kinetics with consistent performance in solid phase extraction methods.

The dense particle packing and uniform distribution within Empore disks offer outstanding sample preparation efficiency and reproducibility of results. Since the diffusion distance between particles is minimized, adsorption is more efficient, and extraction can be accomplished using low sorbent mass. The following performance gains can be realized:

- Reduced solvent volumes
- Small elution volumes
- Reduced time for eluate evaporation
- High throughput
- Channeling effects eliminated
- Excellent reproducibility/low CVs



High Density

Cross section of a high density (HD) $Empore^{\mbox{\tiny M}}$ Disk (10-12 μm particle size)

Product Selection

Empore extraction disks are available in a variety of sorbent chemistries to complement most analytical applications. The following list of available sorbents is organized by the type of extraction.

Reversed Phase Extractions

C8 or C18 SDB-XC Oil & Grease Disk Activated Carbon

Mixed Phase Extractions

SDB-RPS

Ion-Exchange Extractions

Anion Exchange-SR Cation Exchange-SR Chelating Resin

Volume Guidelines

The small bed mass of sorbent in the Empore membrane allows for the use of smaller solvent volumes compared with traditional SPE products. A general guide to solvent volumes for a disk SPE method using reversed phase sorbents (C8, C18, SDB-XC) is listed in the table below.

Volume Guidelines: Reversed Phase (C18 and C8)					
Step	Solvent	47 mm disk	90 mm disk		
Condition	Methanol Reagent water	10-15 mL 20-50 mL	20-30 mL 30-100 mL		
Sample Load	Aqueous	100-1000 mL	500-2000 mL		
Elute	Organic	10-15 mL	20-30 mL		

Note: Suggested solvent volumes will vary according to the disk diameter, the amount of filter aid material, the analyte, the analyte's affinity for the chosen sorbent, and the strength of the eluting solvent. A general guide for solvent volumes is to completely cover the disk and bed of filter aid, such that 2-3 mm of solvent is above the top surface.

Performing an Extraction Method

Standard Reversed Phase Extraction Steps for C8, C18, SDB-XC, and SDB-RPS

Note: When using solvents or other chemicals, be sure to read and follow the manufacturer's precautions and directions for use.

Four Basic Steps Reversed Phase Extraction

- 1. Disk Washing
- 2. Disk Conditioning
- 3. Sample Extraction
- 4. Sample Eluation

1. Disk Washing

For C8 and C18: Assemble the filtration apparatus with the Empore disk. Wash the disk and apparatus with the final elution solvent to assure the system is free of contaminants that may be soluble in the eluate.

For SDB-RPS and SDB-XC: To swell the resin and pre-wash the disk, place 10 mL acetone onto the disk surface (see Note). Allow disk to soak for about three minutes. Pull remaining acetone through the disk with vacuum to dry the disk. Repeat using 10 mL isopropyl alcohol.

Note: The disk will swell upon addition of acetone. This may cause cosmetic wrinkles; however this will not affect the performance of the disk. An alternative method of pre-swelling the disk is to apply a small amount of acetone directly to the surface of the disk prior to clamping the glass reservoir into place.

2. Disk Conditioning

Proper disk conditioning is critical for a successful extraction. Conditioning prepares the sorbent to interact efficiently with the sample matrix. **Failure to condition the extraction disks properly will result in erratic and low recoveries.**

Condition the extraction disk with methanol by pulling most of the methanol through the disk under vacuum (20 inHg, 0.68 bar). Leave a small amount of the solvent above the surface of the disk. Allow the disk to soak in the methanol for 30 seconds. Add reagent grade water to the disk and draw the water through the disk until the water just covers the surface of the disk. At this point, the sample may be added directly to the system for extraction. If at any time prior to sample extraction, the disk should become dry, repeat the conditioning steps above.

3. Sample Extraction

Add the sample to the reservoir and apply the vacuum. Flow rate is dependent on the vacuum source, disk diameter and the particulate content of the sample. Recoveries are generally not effected by the flow rate. After extraction is complete, remove as much residual water as possible from the disk, by applying vacuum to dry the disk for 5 to 20 minutes. Shorter periods of air drying may be necessary with some semi-volatile analytes.

4. Sample Elution

Eluting at least twice with equal volumes of solvent is recommended. Add the first extraction solvent aliquot to the sample bottle rinsing down the sides of the container. Transfer the solvent to the system by pipette, rinsing the glassware and all parts that came in contact with the sample. Apply the vacuum to draw the elution solvent into the disk. Shut off the vacuum and allow the disk to soak in the elution solvent before applying vacuum to collect the eluate. Repeat this process with a second aliquot of eluting solvent. A third aliquot of elution solvent may be used as a final rinse of the system and disk to enhance recoveries.

Other Types of Extractions

Anion Exchange and Cation Exchange: Due to the nature of the ion exchange reaction, slower flow rates may be desirable to allow the reaction kinetics adequate time to occur. Evaluating recoveries at varying flow rates of sample extraction and elution allows the determination of optimal sample processing time for a given analyte and sample matrix.

Carbon: Activated carbon extraction disks are designed for the solid phase extraction of highly polar compounds, such as N-nitrosodimethylamine, which are very water soluble; and volatile compounds, such as trihalomethanes, that are not easily captured by other solid phase adsorbents.

Activated carbon provides one of the most aggressive adsorbents available for solid phase extraction. The carbon surface is a complex combination of characteristics, which includes positive and negative charges. When coupled with high surface area, activated carbon provides adsorption and retention of soluble and volatile analytes.

Chelating: Used to selectively remove multivalent metal cations from solutions. This selectivity can be used to concentrate metals from a sample for analysis or to isolate and remove metal interferences from a complex organic or inorganic sample matrix. In the latter case, the purified filtrate is ready for use.

The chelating disk has an affinity for multivalent (primarily +2 and +3) transition elements. The functional group exhibits differing ionic charges depending on the pH of the solution.

At pH 2, the carboxylate groups are neutral and the nitrogen has a net positive charge. This allows the sorbent to function as a weak anion exchanger. Increasing the pH ionizes the carboxylate groups and at pH =5, both are negatively charged. As the pH approaches neutral, the sorbent functions as a cation exchanger or a chelating resin.

Step A: Sample Preparation	 Microbiological growth can be retarded by lowering sample pH to 2. Filter Aid 400 and/or prefiltration may be helpful if the sample contains excessive suspended solids.
Step B: Extraction Disk Conditioning	 Disk conditioning is critical for a successful extraction. Conditioning provides a good interface between the sorbent and the sample matrix. Failure to condition the extraction disks properly will result in erratic and low recoveries. Center the extraction disk on the base of the filtration apparatus and clamp the reservoir on top of the disk.* Wash the disk with 10 mL of elution solvent. Apply vacuum to dry the disk. Add 10 mL methanol to the disk. Apply vacuum and pull approximately 1 mL through the disk. Vent the vacuum and allow the disk to soak for 30 seconds. Apply vacuum and draw methanol through the disk leaving a small amount of methanol on surface. Add 20 mL of reagent grade water to the reservoir, apply vacuum and draw the water through the disk until the water just covers the disk surface. If disk should become dry while conditioning with methanol or water, repeat steps 4 through 6. * Place a vial in the vacuum apparatus to collect and dispose of wash and conditioning solvents. Remove vial prior to sample extraction.
Step C: Sample Extraction	 Pour the sample into the reservoir and apply vacuum to draw through the disk. Flow rate is dependent on vacuum setting and solids content of the sample. However, recoveries are not affected by flow rate. After sample extraction is complete, remove residual water from the disk by applying vacuum to dry the disk for approximately 5-20 minutes.
Step D: Sample Elution	 At least 2 elutions with 10 mL solvent are recommended. 1. Place tip of filter base into the collection vessel (see diagram). 2. Add 10 mL elution solvent to sample container, carefully rinsing the sides. Transfer solvent from sample container to reservoir with a pipet, washing the walls of the reservoir in the process. 3. Apply vacuum and draw approximately 1 mL elution solvent through the disk. Vent the vacuum and allow the disk to soak for 30 seconds before reapplying vacuum to draw the eluting solvent through the disk. 4. Repeat this process with a second aliquot of eluting solvent.

Extraction Method with C8 or C18 Disk

Extraction Disk Processing Options

Empore extraction disks work with vacuum or positive pressure and can be used at either full or reduced pressure.

Vacuum Manifold Systems

One-, three- or six-station manifolds offer efficient extraction approaches. These manifolds allow multiple sample processing and are more stable and compact than a series of single glass filtration setups. Using the manifold, up to six extractions can completed simultaneously, and multiple manifolds can be managed by a single operator.

The manifolds are constructed from corrosion resistant stainless steel. Each is individually controlled by a valve that allows extraction or venting to the atmosphere. The valve stem is PTFE in a chrome-plated brass housing with stop-pins for positioning.

These manifolds are intended for use with standard filtration glassware. The drip tip is positioned to accommodate a variety of elution receptacles such as K-D tubes, straight-wall vials, and 10 mL volumetric flasks.





Product Specifications

Compositions	C8, C18, Anion, Cation, SDB-RPS, SDB-SC & Chelator	90% or greater sorbent particle 10% or less PTFE	
	Carbon	80% or greater sorbent particle 20% or less PTFE	
Thickness	0.50 mm ± 0.05 mm		
SPE Flow Rate	Typically < 10 min/L DI H ₂ 0 @ 25°C @ 20 inHg (47 mm disk)*		
Particle Size	12 μm (nominal)		
Solvents	Compatible with all organic solvents		
pH Range	Silica based sorbents	Stable between 2 and 12 under normal use conditions	
	Resin based sorbents	Stable between 1 and 14 under normal use conditions	

*The actual sample flow rate is dependent upon the sample viscosity and the amount of suspended particulates in the sample.

Note: Empore Sample Preparation Products are intended for solid phase extraction during scientific research only. These products are not intended for use in medical devices or in assessment and treatment of clinical patients.

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