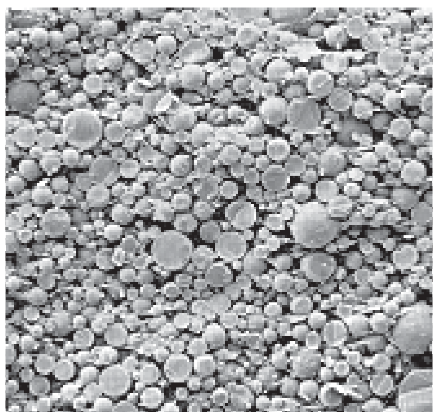
Empore™
Solid Phase Extraction Disks

Chelating

General Information

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 inert 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.



High Density (HD) Empore™ Membrane (10-12 µm particle size)

Product Information

Empore™ Chelating Resin Disks provide a mechanism to selectively remove multivalent metal cations from solutions by simply passing the solution through the porous membrane. The selectivity can be used to concentrate either the metals from "large" volumes of matrix or to selectively isolate them from a complex organic or inorganic matrix. If the intent is to quantify the ionic metals, the chelating resin is eluted and the eluate analyzed. In the reverse application, the chelator could be used to purify a solution when the metals are the source of problems in an application. In this case, the purified filtrate is now ready for use, and the metal ions are bonded to a solid support which can be disposed in an environmentally acceptable manner.

The disk consists of a polymer support (cross-linked poly[styrenedivinylbenzene]) functionalized by bonding at the nitrogen atoms to iminodiacetic acid groups. The functional group exhibits differing ionic charges depending on the pH of the solution. For instance, at a pH of 2, the carboxylate groups will be neutral; however, the nitrogen will have a net positive charge and the molecule a weak anion exchange capability. Increasing the pH ionizes the carboxylate groups and at pH 5 both are negatively charged. As the pH approaches neutral, the molecule functions as a cation exchanger or metal cation chelator as described herein.

In general, a chelating agent, or ligand, contains two or more electron donor atoms that can form coordinate bonds to a single metal ion; and with successive donor atoms, creates a ring containing the metal ion. The ring structure is referred to as a chelate, the name deriving from the Greek word *chela* for the great claw of the lobster. Stability of the chelate complex is strongly dependent on the relationship between the size of the metal ion and that of the opening within the crown or crypt. Mercury (Hg^2), for example is extracted efficiently from solution; however, it is extremely difficult to elute from the sorbent. This iminodiacetate chelator has an affinity for multivalent (primarily +2 and +3) transition elements; and it will bind better with alkaline earth than alkali metal ions. Relative selectivity roughly follows the EDTA formation constants: Pb>Cu>Cd>Co>Fe>Ca>Sr. Capacity is relatively high and is dependent on the matrix pH (about pH 5 or higher) and which ions are present in solution. Elution is accomplished with an acid solution such as 3 molar(M) nitric or hydrochloric acid.

Suggested Product Applications

Sorbent	Suggested Applications	Product Number 47 mm	Product Number 90 mm
Chelating	Divalent metals and other divalent cations	2271	NA

CDS Analytical

Extraction Method with Chelating Disk

Step A: Sample Preparation

- Filter Aid 400 and/or prefiltration may be helpful if the sample contains excessive suspended solids.

Step B: Extraction Disk Conditioning

The Empore™ Chelating Disk should be wet directly with water. **Prewetting with an organic solvent such as methanol is not recommended. Such prewetting will not improve flow rate, but will cause the disk to wrinkle.**

For many applications, the disk can be used directly; however, for very low level (ppb) metal analysis and maximum activity, the disks should be preconditioned. The following procedure is recommended for conditioning the 47 mm disk:

1. Place the disk in a glass filter holder (see diagram). A Kel-F™ filter screen is recommended.
2. Apply vacuum to the dry disk (20 inHg/0.68 bar is typical), then wet the disk with about 20 mL of water.
3. Wash the disk with 20 mL of 3 M nitric acid or 3 M hydrochloric acid followed by two 50 mL water washes. Let the disk go dry between each wash.
4. To put the disk in the ammonium form (its most active form), wash with 50 mL of 0.1 M ammonium acetate buffer at pH 5.3 followed by several water washes. It is essential that this reagent be free from analyte metals of interest.

Step C: Sample Extraction

In the ammonium form, the Chelating disk will bind most multivalent metal ions such as Cu^{+2}, Pb^{+2}, Cd^{+2}, Fe^{+3}, etc. The selectivity of the membrane roughly follows the EDTA complex formation constants for various metal ions (Pb>Cu>Cd>Co>Fe>Ca>Sr). Capacity is generally higher at pH 5 and above. Tighter binding ions such as Cu^{+2} and Pb^{+2} will displace other ions when capacity is reached.

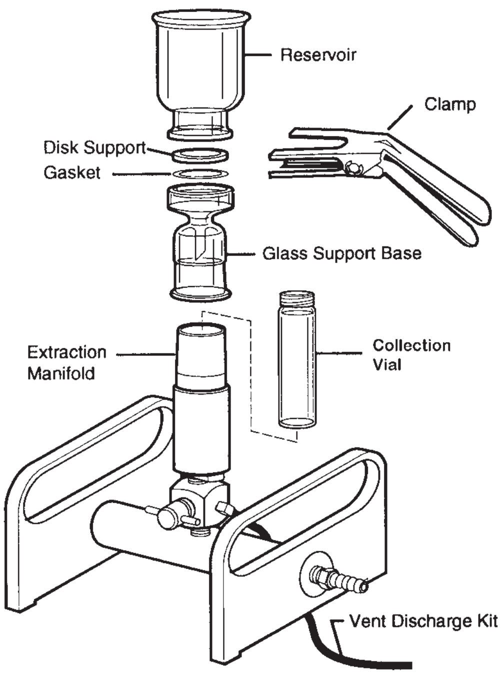
- Pour the sample into the reservoir and apply vacuum. In general, recovery of tightly binding metal ions is not affected by flow rate; however, in some cases, recovery will be improved at slower flow rates. Flow rate is dependent on vacuum source and particulate content of the sample. Flow rates through individual extraction disks may vary.
- After sample extraction is complete, remove as much residual water as possible from the disk by applying vacuum to the disk for several minutes until dry.

Step D: Sample Elution

Elution of metals from the Chelating disk is achieved with strong acid such as 3 M nitric acid or 3 M hydrochloric acid. For the 47 mm disk in a standard glass holder, 95% recovery of most metals can be achieved by eluting twice with 10 mL of 3 M nitric acid or 3 M hydrochloric acid and combining the eluates. For a one-liter sample, this will yield a fifty-fold increase in the concentration. A few metals such as chromium may be difficult to elute, particularly if left on the disk for several hours.

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

Disk Manifold System Setup



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 chelating is listed in the table below.

Each assay will need some further optimization in terms of selecting the best elution solvent (commonly methylene chloride, methanol or acetonitrile).

EPA Methods will require specific reagents; please refer to those methods when using the Empore Disks for agency reporting.

Volume Guidelines: Chelating (See previous page for steps.)

Step	Solvent	47 mm disk	90 mm disk
Condition	Water	20 mL	50 mL
Optional:	3 molar acid then water	20 mL	50 mL
Aqueous	Sample solution	100-1000 mL	500-2000 mL
Elute	Strong acid	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.

Product Characteristics

Composition	90% or greater sorbent particle 10% or less PTFE
Thickness	0.50 mm ± 0.05 mm
SPE Flow Rate	< 10 min/L DI H_2O @ 25°C @ 20 inHg (47 mm disk)
Particle Size	12 µm (nominal)
Solvents	Compatible with all organic solvents
pH Range	Stable between 1 and 14 under normal use conditions

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Note: Empore Solid Phase Extraction 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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