



Empore™

Extraction Disks

APRIL 1995

General Guidelines for Applications

Empore™ Cation-SR Ion Exchange Disk

Summary

Cation exchange is a procedure for extracting ionized species with a positive charge from liquid matrices. The strong acid cation exchanger (SCX) resin is a Poly(styrenedivinylbenzene) (SDB) with a sulfonic acid functionality. These are resin-based as opposed to silica-based exchangers, which means they are stable at pH extremes (pH 0 - 14). As with other Empore™ disk products, the high extraction efficiency and fast flow rates are intrinsic because of the small particle size, lack of channeling and uniformity of flow.

These ion exchange resins are shipped in a hydrogen form. Estimated capacity is 1.1 milliequivalents/gram or 0.35 meq/47mm disk. Because ion exchange in general has a finite capacity, it is important to be aware of the total ionic strength of the matrix to be extracted. Any competing ions in solution will compete for the binding sites and those of higher selectivity than the isolate of concern can potentially replace analyte already bound to the sorbent. The total ionic strength of a solution to be extracted should not exceed 0.1 M. If the ionic strength cannot be determined in advance, a method of serial dilutions of the sample can be used to determine whether competing ions are impacting the recovery of the analytes of concern.

Candidate isolates for the Cation-SR are metal cations and compounds with amine functional groups. Literature references are common for primary, secondary, and some tertiary amines. Strong cation exchangers are not recommended for quaternary ammonium analytes, as the bond is so strong they do not elute.

To obtain retention on ion exchange sorbents, both the sorbent and analyte moieties must be in an ionized form. The sulfonic acid has a negative charge at any reasonable pH. To select a pH for the sample matrix containing the analytes, one must be aware of the pKa value(s) for the compound(s). Generally, the pH is adjusted to at least 2 units below the pKa of the analyte cation. If metals are the intended analytes, do not exceed the pH at which the metals will precipitate from solution.

Elution is achieved by neutralizing the sorbent with a relatively concentrated acid solution (e.g. 20% HNO₃), or neutralizing the analyte using a basic pH eluant. A third option is to displace the analyte cation using a higher selectivity counter-ion than the charged moiety of the analyte. A chart of relative selectivities of the common counter-ions is included in this document for guidance. Additional information on selecting the counter-ion and matrix are provided in the specific sections of this document.

General Method for Using Cation Exchange Disks

This section is intended to provide background information on methodology for cation exchange resins.

Pre-Wash *Organic solvent, eluting reagent*

Depending on the analytes, pre-washing may be necessary to remove any trace impurities on the disk or glassware. If using the eluting reagent, keep in mind the relative selectivity of any counter ions. Use of a strong counter ion at this stage is inadvisable.

Because these are polymer based, the disk material will swell upon the addition of a solvent. The swelling is cosmetic only and will not affect the performance of the sorbent. Wetting with a small amount of acetone prior to placement of the reservoir and clamp will pre-swell the material and avoid the cosmetic wrinkling.

When extracting organic compounds at low levels of detection, it is often advantageous to pre-wash the disk material with an aliquot of acetone followed by one of isopropanol. Though these washes are optional, analysis of a blank disk extract would indicate whether the acetone/isopropanol washes would be beneficial.

Conditioning *Methanol*

Our experience has shown that conditioning with methanol may not be necessary with this hydrophilic material. If desired, we recommend methanol as the conditioning agent. Add the methanol to the reservoir, allow to soak for a minute, then draw most through, leaving a layer (2-3mm) above the disk.

Wash *Reagent grade water*

If a methanol conditioning step is used, excess methanol can be removed by the addition of a water wash step. This is done by adding the water to the methanol remaining atop the disk and drawing most through, again leaving a layer of liquid atop the disk. The sample to be extracted is added directly to this remaining liquid.

Sample Extraction

Adjust the sample pH to the range appropriate for ionization of both the target analytes and the sorbent. See notes in the previous section on selecting the pH based on the pKa value(s) of the analyte(s). If the total ionic concentration of the sample is known to be >0.1 M, the sample should be diluted until the concentration is below that value. Add the sample to the extraction glassware reservoir and apply vacuum to draw the entire sample through the disk.

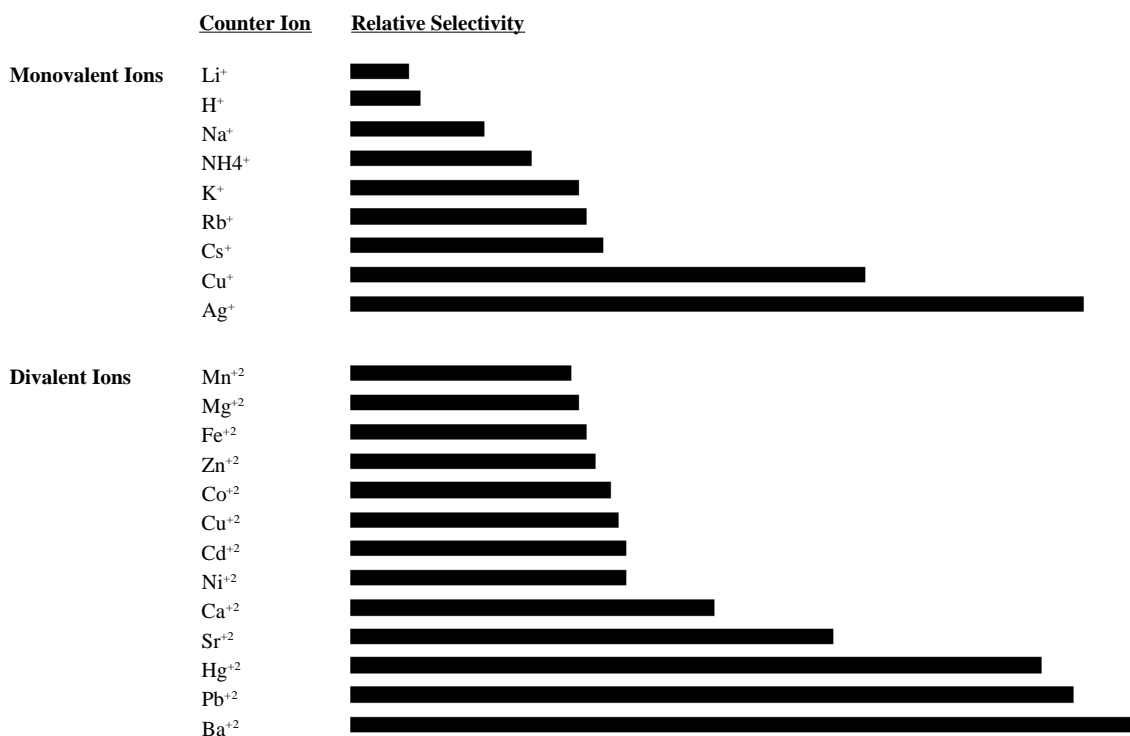
Wash *Reagent grade water, organic solvent*

A wash step is optional at this stage. The disk material has an estimated dead volume of 0.5 mL for a 47mm disk. This dead volume can be assumed to hold about 0.5mL of the sample just extracted. A reagent water wash would rinse this dead volume of any water or residuals, should their presence cause problems with the elution reagents or instrumental analysis. If the matrix extracted is a solvent, this wash should also be a solvent which does not contain any of the analytes of concern.

Elution

There are three approaches to elution using strong cation exchange resins. Elution can be accomplished by neutralizing the exchange moiety on the sorbent using a relatively concentrated acid solution (e.g. 20% [v/v] nitric acid). To neutralize the analyte, a basic solution such as 0.1N NaOH can be used. Another effective mechanism for elution is to displace the cations of interest with a cation of stronger relative selectivity. The cation should be in an aqueous solution generally mixed 50:50 with methanol for organic cations or a water-based solution for metal cations and should have a pH at least 2 units above the pKa(s) of the analyte(s). The chart below compares relative selectivity of several commonly encountered cations.

Counter Ion Selectivity
Strong Cation Exchange Sorbent



Extraction Method for Metal Cations

The following outline is intended to provide guidance when developing methods for the isolation and quantification of cationic metals in solutions:

Pre-clean all glassware using non-metal containing cleaner and pre-rinse with concentrated HNO₃ (trace metal grade). For ultra trace levels, PTFE, polypropylene or silanized glassware should be considered.

Assemble extraction glassware with a Cation-SR disk in place. A solvent pre-wash using acetone is optional. Prepare the disks using the conditions appropriate for the matrix you are using. The volumes in these instructions assume a 47mm disk for the extraction:

Pre-wash:

Aqueous Matrix

10 mL acetone (Optional)
soak one minute then, evacuate
air dry

10 mL 20% HNO₃, soak one minute
2 x 10 mL reagent water

Non-Aqueous Matrix (Solvent-based)

10 mL 20% HNO₃, soak one minute
2 x 10 mL acetone

Extract Sample: Adjust the sample pH to that at which the sorbent and all cations of concern will be ionized. Note total ionic strength should not exceed 0.1M. Add the sample to the reservoir and apply vacuum. Draw the entire sample through the disk, then allow the disk to air dry.

Elute: With collection vial in place, add 5 mL 20% HNO₃ to the reservoir, rinsing down the sides. Allow to soak for one minute, then apply vacuum and collect eluate. Repeat the elution step with a second 5 mL aliquot of acid. Adjust the final volume of eluate to that which is compatible with the detection limits you wish to achieve. Analyze by AA, ICP, etc.

Extraction Method for Organic Cations

The following outline is intended to provide guidance when developing methods for the isolation and quantification of organic cations in solutions:

Place a Cation-SR disk on the base of the extraction glassware. Apply 1-2 mL acetone to the surface of the disk to pre-swell the disk prior to the placement of the reservoir and clamp. This will prevent cosmetic wrinkling when the reservoir is clamped in place. Place the reservoir atop the base and clamp in place.

Pre-wash: Add 10 mL acetone to the reservoir, washing down the sides. Allow to soak for a minute, then draw through under vacuum. Air dry the disk for about 30 seconds. Wash with the following amounts indicated:

10 mL 20% HNO₃
2 x 20 mL reagent water

Sample Extraction: Adjust the sample pH to that at which the sorbent and all cations of concern will be ionized. Note total ionic strength should not exceed 0.1M. Add the sample to the reservoir and apply vacuum. Draw the entire sample through the disk, then allow the disk to air dry. A wash step using reagent water or solvent could be inserted at this point.

Elution: With a receiving vial in place, add 5 mL of 0.1N NaOH to the reservoir, rinsing down the sides of the reservoir in the process. Allow to soak for one minute, then apply vacuum and draw through. For non-polar analytes, elute with 5 mL of 50% 0.1N NaOH:Methanol. (Methanol, by itself, may work for eluting certain analytes.) Concentrate or dilute as necessary for instrumental analyses.

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