SERDOLIT®

SERDOLIT® Ion Exchange Resins: General Information

SERVA offers 2 types of ion exchangers which are based either on polystyrene (= SERDOLIT®) or cellulose (= SERVACEL®) as matrix. The polystyrene exchangers mainly are used for the separation of small molecules up to 3,000 daltons either in the batch mode or by column chromatography, whereas the cellulose exchangers are applied in most cases for the liquid column chromatography of biomolecules.

Polystyrene ion exchangers

These are insoluble spherical or irregular porous particles grafted with negatively (sulfonic or carboxylic) or positively (quarternary, tertiary, secondary or primary amino) charged groups resulting in cation resp. anion exchangers. The particles have excellent chemical, mechanical and in most cases thermal stability.

Pore size

During synthesis the polystyrene chain is crosslinked with a bi- or multifunctional reagent, in most cases divinylbenzene(DVB). Higher amount of cross linker means smaller pores (gel type resins, pore size 20 - 50 Å, 2 - 5 nm). This results in increased chemical and mechanical stability as well as lower water content. Therefore more ionized groups are available per volume unit, resulting in higher capacity.If the DVB concentration exceeds 20 % the polymer will become inhomogeneous and large pores will arise. During the production process, the larger pores are transformed into a labyrinthic structure which results ultimately in macropores. The so-called macroporous resins feature pores of 200 - 500 Å (20 - 50 nm).

Water content

Ion exchangers are very hygroscopic and lose all their water only after prolonged heating. If a dry resin is necessary (e.g.for catalysis) the anion exchangers must be transformed first into the chloride form, the cation exchangers into the sodium form. Then they can be heated up to 60 ℃ (140 ℉) resp. 120 ℃ (248 ℉) preferably in vacuo. If the ion exchangers are to be used water-free in the free base or hydrogen form, they are best washed with dry organic solvents (e.g.acetone, alcohol, DMF, DMSO etc.). With the dry resin the pore structure becomes nearly impermeable, thus for reactions in water-free medium macroporous resins are to be preferred.

Regeneration

Ion exchangers can take up only a defined amount of ions depending on their capacity. If this is attained they must be regenerated which means the fixed ions must be removed and replaced in most cases by hydroxyl (anion exchanger) or hydrogen (cation exchanger) ions. For this purpose, the particles are suspended in demineralized water, poured into a funnel and treated with diluted caustic resp. hydrochloric acid. Finally, water is percolated according to table 2. Because of the possible bed expansion during regeneration and cleaning cycles these should not be performed in thin-wall glass columns which might be destroyed. For the regeneration of mixed bed ion exchangers at first a physical separation must take place. This can be effected by making use of the different densities of the 2 resins. The mixture is removed from the column and placed into a beaker. A 50 % solution of sodium hydroxide is added until the (lighter) anion exchanger is floating in the caustic and is well separated from the cation exchanger remaining on the bottom (with indicator-dyed resins, the border line can be seen quite well). The anion exchanger is carefully decanted or (better) siphoned off into a funnel, washed with 2 N NaOH and finally water until pH neutral. The cationic component is treated in the same manner with HCl according to table 2. As the 2 resins tend to separate in water, they are mixed "dry" (they still contain about 50 % water) after regeneration and filled "dry" into a column (or e.g. pipette), hold in place by an adaptor, filter paper or wool plug.

Customer service

For special purposes other counter ions than described above may become necessary. For availability, please inquire.

Table 1: Relation between cross linkage, water content and exclusion limits.

Cross Linkage	Moisture Content	ExclusionLimit	Resin Type
2 %	80 %	3000 dalton	**Gel Type**
4 %	70 %	1500 dalton	
8 %	55 %	1000 dalton	
12 %	45 %	700 dalton	
16 %	40 %	600 dalton	
20 %	50-65 %	12000 dalton	**Macroporous**

Applications

Desalting of proteins

Diluted protein solutions should never come into direct contact with ion exchangers in the free base or hydrogen form.There will always be unspecific adsorption, precipitation or even denaturation. Dialysis against a mixed bed resin is the method of choice: the protein solution is filled into a dialysis tubing (e.g. Visking, cat.no. 44104) and suspended in a 10 % suspension of mixed bed ion exchanger with exhaustion indicator (e.g. SERDOLIT® MB, cat.no. 45500) in a beaker. The resin is slowly agitated. If exhaustion is indicated by colour change, the resin is removed and replaced by fresh material.

Removal of SDS

The unbound (surplus) detergent can be removed in the same manner as described above for desalting of proteins.

Removal of SERVALYT™ carrier ampholytes

Dialysis against mixed bed ion exchangers is an effective and also gentle method (VISKING® dialysis tubing cat. no. 44104 is recommended). As SERVALYT™ gives a positive ninhydrin reaction, a negative reaction indicates the end of the dialysis.

Purification of formamide

To 100 ml of formamide 5 g of SERDOLIT® MB (cat.no.45500) are added. The mixture is slowly agitated for 1 h at room temperature and finally filtered. After filtration the purified formamide is stored at -20 ℃ (-4 ℉). Be aware that the mixed bed resin contains 50 % of water which will dilute the formamide.

Tip: *By passing the formamide through a small ion exchange column which had been percolated with some milliliters of formamide to remove the water, better results will be obtained in shorter time.*

Cleaning of acrylamide and urea solutions

Some preparations of acrylamide or urea may contain free acrylic acid resp. ammonium isocyanate. These impurities can easily be removed by passing the solutions through a small column of mixed bed ion exchanger with exhaustion indicator. Again, it should be kept in mind that the resin contains about 50 % of water.

Other examples of application:

- Water treatment
- Sugar refining
- Preparation and purification of pharmaceuticals (antibiotics, vitamins, alkaloids, nucleotides, amino acids, peptides)
- Catalysis
- Galenic (tablet disintegrants, odour masking, sustained drug release)

Table 2: Conditioning of Ion Exchangers

The experimental procedures given in the table below should be applied for quantitatively transferring an ion exchange resin from one ionic status into another. An excess of reagents is applied at the laboratory scale, economical considerations are of minor importance as compared to completeness of the reactions.

Ion Exchanger	Reaction	Reagent	Reagent/ Resin Volumes	Flow Rate cm/min[1, 2]	Rinse Water/ Resin Volumes	Rinse until
Strongly acidic sulfonic acid cation exchanger	$Na^+ \rightarrow H^+ \rightarrow$ ("Regeneration")	2N HCl	5	2	6	pH >5 neutral
	$H^+ \rightarrow Na^+$	1N NaCl, 1 N NaOH	5	2	6	Cl^- negative
	$Ca^{++} \rightarrow Na^+$	2N NaCl	5	2	6	Cl^- negative
Weakly acidic carboxylic acid cation exchanger	$Ca^{++} \rightarrow H^+$ ("Regeneration")	0.5N HCl	4	1	15	pH > 5
	$Ca^{++} \rightarrow Na^+$ ("Regeneration")	1N NaCl	10	1	15	Cl^- negative
	$H^+ \rightarrow Na^+$	2N NaOH	8	1	15	pH< 8
Weakly basic tertiary amine anion exchanger	$Cl^- \rightarrow$ free amine	1N Na_2CO_3, 1N NH_4OH	8	1	10	pH < 8
	free amine $\rightarrow$ Cl	1N HCl	3	1	4	pH > 6
Strongly basic quart. amine type I	$Cl^- \rightarrow OH^-$ ("Regeneration")	2N NaOH	8	2	8	pH < 9
	$HCO_3^- \rightarrow OH^-$	2N NaOH	4	2	8	pH < 9
	$OH^- \rightarrow$ formate	2N formic acid	5	2	6	pH > 5, E_{280} <0.01[3]
	$Cl^- \rightarrow$ formate	same as OH-form				
Strongly basic quart. amine type II	$Cl^- \rightarrow OH^-$	2N NaOH	5	2	10	pH < 9
	$Cl^- \rightarrow NO_2^-$	1N $NaNO_2$	4	2	6	Na-flame negative
Chelating resin	$Cu^{++} \rightarrow H^+$	1N HCl	4	1	6	pH > 5
	$Na^+ \rightarrow NH_4^+$	2N NH_4OH	4	2	6	

1) Flow rate is given for fine mesh resins (below 0.3 mm). For standard particle size resins (0.3 - 1.0 mm) use 1/4 of this flow rate. Rinsing of the resin is effected with the same flow rate

2) Volume flow rate in ml/minute is obtained by multiplying the flow rate with the cross section of the column in cm^2.

3) If E_{260} = 0.01 is not attainable, wash with 50 - 100 volume acid at max. 0.1 cm/minute.