

SAVE THIS DATA SHEET!
It Contains Important Information About This Product.

Procedures for Preparing and Using Columns of Amberlite XAD, Diaion, Dowex, MCI GEL, Sepabeads, and Supelite DAX Adsorbent Resins

Precautions: DO NOT use nitric acid or other strong oxidizing agents with these materials. Elution with organic solvents, such as methanol, ethanol, or isopropanol, can cause these resins to swell. Use enough resin to half-fill the column only. This will allow room for expansion when resin-swelling solvents are used.

Resin Wetting Procedure

Most of these materials are supplied in wet form. However, prolonged exposure to air during shipment and/or storage may cause the material to dry. The material must be wetted before use unless the application is air sampling. Follow the procedure outlined here.

1. Transfer the dry resin to a 500mL beaker. Add sufficient methanol to cover the resin bed by 1-2 inches (2.5-5cm).
2. Stir the resin gently for a minute to ensure complete mixing. Allow the material to stand for 15 minutes.
3. Carefully decant most of the methanol and replace it with distilled water. Stir the mixture, then allow it to stand for 5-10 minutes. Follow the column preparation instructions below.

Preparation of Columns

Always use fully hydrated resin when preparing a column. If necessary, wet the material by following the procedure described above, and do not allow the resin bed to dry during preparation or subsequent use. A constant head of about 1" (2.5cm) of water or solvent above the resin bed will prevent resin dehydration and will reduce channeling in the bed.

1. Add approximately 1" (2.5cm) of deionized water to the empty column before you add the resin slurry.
2. Slowly pour the resin slurry into the column. As the column fills, drain excess water through the bottom of the column, but do not allow the liquid level to fall below the top of the resin bed. Add enough resin to half-fill the column only. This will allow room for expansion when resin-swelling solvents are used.

Backwashing

This procedure removes air bubbles, classifies the resin particles (allows them to stratify according to size, smallest particles at the top of the column), and rids the new bed of resin fines (small particles). Approximately 30 minutes of backwashing usually is sufficient to prepare the bed in a new column.

For columns in use, backwashing removes debris that has collected in the bed. Typically, the bed is backwashed after the adsorbate has been adsorbed to the resin, prior to elution of the adsorbate.

1. Attach a water line to the bottom of the column.
2. Introduce a slow upward flow of deionized water. Carefully increase the flow until the entire bed of resin is suspended.
3. Maintain the water flow until all air bubbles are dislodged. Resin fines will pass out of the top of the column.
4. Stop the flow of water and allow the resin to settle.
5. Adjust the water level to 1" (2.5cm) or more above the top of the resin bed.

Determining the Bed Volume

After the column has been filled, backwashed and allowed to settle, and the liquid level has been adjusted, determine the volume of resin in the column:

$$\text{volume of a cylinder} = \pi \times (1/2 \text{ inside diameter})^2 \times \text{length of the bed}$$

This value, the "bed volume," is helpful for properly operating the column.

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Loading the Resin

The process of passing a sample or feed solution through the column, to allow compounds of interest to adsorb to the resin, is termed "loading." Adsorption is an equilibrium phenomenon, and the adsorbate is never totally adsorbed. Normally, the most effective flow rate for loading the resin is within the range of 2-16 bed volumes per hour. Flow rates at the lower end of the range allow higher adsorption rates; flow rates exceeding 8 bed volumes per hour produce greater leakage of the adsorbate through the adsorbent bed.

Generally, loading is allowed to continue until adsorbate leakage in the column effluent increases to a predetermined level. At this point, the adsorbed compound(s) is removed by elution.

The solution passed through a bed of resin should be free of suspended particles. Accumulation of particles in the resin bed can cause an uneven distribution of sample flow and thereby reduce adsorption efficiency. To ensure a particle free resin bed, filter your samples whenever possible and backwash the column after each adsorption/elution cycle.

Selecting an Elution Solution

For most resins, a suitable organic solvent will effectively elute adsorbed materials. The solvent should be one which will solubilize the adsorbed material and will interact with the resin surface (e.g., a hydrophobic solvent with a hydrophobic resin). Methanol, isopropanol, and acetone generally are good elution solvents (see precautions on the first page of this form). The typical flow rate for adsorbate elution is 1-2 bed volumes per hour. Normally the adsorbed material can be recovered in 2-3 bed volumes, or less, of this solvent.

Ionic or potentially ionic materials generally will be much more strongly adsorbed in the nonionic form than in the ionic form. Thus, for example, acidic compounds are best adsorbed when they are not ionized and can be eluted well with a base, which ionizes the acidic groups.

Other special techniques may be applicable. Compounds adsorbed from high-molarity salt solutions often can be eluted with plain water. Steam can also be effective for removing adsorbed volatiles.

Because the elution process reactivates the adsorbent surface, this process also has been termed "regeneration."

Equilibration

Following elution, void space in the resin bed is filled with the elution solution. Before the column can be used again, this solution must be removed from the column. Typically 2 bed volumes of an equilibration solution (rinse water or an appropriate solvent) are used. Apply the first bed volume of equilibration solution at the same flow rate as used for adsorbate elution, since equilibration represents the completion of elution. After equilibration, the column is ready for re-use.

Flow Rates and Solution Volumes

Backwashing Rates and volumes as required to classify the resin particles by size and remove fines and accumulated solids.

Loading

2-16 bed volumes per hour.
Volume determined by leakage of compounds of interest in eluate.

Elution

1-2 bed volumes per hour.
Use 2-3 bed volumes in most systems. Smaller volumes can sometimes be used, especially if some of the spent elution solution can be recycled through the column.

Equilibration

1-2 bed volumes per hour (the same as for elution).
Use 2 bed volumes. Apply the first bed volume of rinse water/solvent at the same flow rate as used for elution, since equilibration represents the completion of the elution process.

Trademarks

Amberrite — Rohm and Haas Co.
DAX — Sigma-Aldrich Co.
Diaion — Mitsubishi Chemical
Dowex — Dow Chemical Co.
MCI GEL — Mitsubishi Chemical
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