



ZEOCHEM®

PACKING INSTRUCTIONS FOR ZEOSPHERE® IN DAC-COLUMNS

- Always use clean frits, preferably new (outlet and inlet 2-5µm porosity). If the frits are clogged or contaminated, clean them with sodium hydroxide (0.5-2M) in an ultrasonic bath overnight, and then rinse thoroughly with water, acetic acid, water and an alcohol the following day.
- Select the right slurry solvent. Here is a list of solvents that have been successfully used to generate efficient column beds.

ZEOSphere® C18

EtOH
IPA
THF/IPA (50/50)
MeOH/IPA (50/50)

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Acetone/IPA (50/50)
EtOH
IPA/toluene (50/50)

- Prepare a slurry at a concentration of ≤1kg / 2 litre solvent. The silica gel amount needed can be calculated using the packing density of ZEOSphere®. This is equal to 0.48 g/mL for ZEOSphere® bare silica. The packing density of ZEOSphere® 70Å C18 is 0.71 g/mL and of the one of ZEOSphere® 100Å C18 0.65 g/mL.
- Use maximum piston pressure during packing (70-100bar)
- Use high capacity air supply and large diameter air tubing to the packing pump during the packing process.

Column	Air tubing
50 mm ID	≥ 8 mm ID
150mm ID	15 mm ID
≥ 150 mm ID	≥ 20 mm ID

- - Raise the piston and introduce the slurry.
- Expel the air above the slurry by moving the piston slowly downwards.
- Plug the solvent inlet (piston side) and continue the packing process at high speed.
- Decrease the piston pressure during chromatography to 10-20 bar above the maximum bed pressure drop obtained during the purification process.
- For column performance evaluation, it is preferable to introduce the test compound close to the column or to inject via a loop. It is of considerable importance to ensure that the delivery system is well optimized and a sufficient sampling rate of the detector is utilized. As test substances for RP, acetophenone or toluene can be used; for NP use nitrobenzene, dimethylphthalate or aromatic esters.
Solvent linear velocity for column performance evaluation should be in the range of 0.45 - 1.1 mm/sec for 10-20 µm silica.



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UNPACKING AND CARE OF USED ZEOSPHERE®

ZEOsphere® silica is superior in terms of mechanical and chemical stability, and can therefore, under most circumstances, be repacked many times.

ZEOsphere® can be either stored as a dry powder, or in a solvent. For storing the silica in a solvent it is recommended that the used silica is unpacked and stored in the solvent, which will be used later in the packing process as a slurry solvent. For recommended slurry solvent see our **packing instructions**.

ZEOsphere® bare silica, and packing used in normal phase mode:

If toluene/isopropanol is intended as slurry solvent during your next packing, you should switch to pure isopropanol prior to unpacking. You should allow at least 2 column volumes of this solvent to flush through the silica. Apart from cleaning the silica, this is also a good solvent for storing.

ZEOsphere® reversed phase silica:

When unpacking reversed phase silica, it is important to first rinse the column bed from any buffer solution. To remove the salts from the packed bed it is recommended to use mixture of isopropanol/water, ethanol/water, methanol/water or acetonitrile/water. After any of these mixtures, all water should be flushed out using 100% of a water miscible solvent like isopropanol, ethanol, methanol or acetonitrile. Before unpacking you should then switch to a solvent, which is included in the packing solvent.

To store ZEOsphere® dry, apply the methods above, and then use either acetone or tetrahydrofuran as the final solvent. Unpack the bed, and dry the packing in a vacuum oven at 100°C for reversed phase silica and in a air circulation oven at 100°-120°C for bare silica.



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STORAGE AND LIFETIME OF ZEOSPHERE®

Unused ZEOSphere® should be stored in dark closed containers at or below room temperature and in low humidity, it can be stored for at least five years. More than five-year-old ZEOSphere® should be tested in analytical scale columns before used in large scale columns.

When ZEOSphere® is used in campaigns in a multipurpose DAC-column, one batch of ZEOSphere® can easily be saved and stored for later use in new campaigns. It is very important to avoid buffers and water in the reversed phase ZEOSphere® during long-time storage. Before unpacking the column, it should be rinsed in a certain sequence as is described in the former in chapter.

The moist ZEOSphere®, containing some of the coming packing solvents, is unpacked from the column and should be stored into stainless steel or plastic containers. If the

preparation as described has been made and if ZEOSphere® is stored in closed containers at or below ambient temperature, it can be stored for several years.

In the case of dry non-derivatized ZEOSphere®, a slow uptake of water unavoidable, since ZEOSphere® is hygroscopic. This water content must be removed from the silica before using it. At easiest this is done after the packing process by rinsing the column bed with a eluent containing an alcohol.

The optimum time to replace the used ZEOSphere® with a new one must be decided for each application separately and has to be determined by its chromatographic performance for this particular application.

Sometimes the ZEOSphere® is contaminated and even discoloured at the inlet of the bed by irreversibly adsorbed impurities. In this case careful unpacking of the column bed is recommended so that contaminated ZEOSphere® can be cut away and replaced with new ZEOSphere® for the next packing process.

Although the ZEOSphere® has a very high mechanical strength, numerous slurry preparations using pumps and stirrers cause very high grinding forces with subsequent formation crushed particles and fines. These particles can be easily removed from the bulk ZEOSphere® by sedimentation and by discarding the supernatant.

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