

Bulk HPLC Sorbent Storage and Cleaning for Reuse

Often times, because of cost, users want to save and store the media for future use. There are steps one can take to store bulk resins for short term and long term use. Please keep in mind, that the media will not last forever and eventually needs to be replaced or performance will be compromised.

Sorbent reuse and storage can be put into two categories: short term vs. long term. If the HPLC media is going to be reused and repacked into the DAC column within 1-5 days, the following protocol is recommended:

Displace the mobile phase with slurry solvent (this composition should be supplied by the sorbent manufacturer). If the slurry solvent is not miscible with the mobile phase, flush the column with an intermediate solvent that is miscible with the slurry solvent and mobile phase components (good choices are IPA, Acetone and THF). Flush the column with 10 column volumes of slurry solvent. Push the sorbent out of the column in the vessel used for slurry preparation. Remove the top portion of sorbent (5-10mm layer against the piston) and replace with new media as needed. Re-suspend the sorbent in a slurry solvent by mixing until free of clumps. Pass the slurry solvent through a 200 mesh sieve (the sieve dimension should fit your column diameter). It is important to pass the well dispersed slurry through this sieve in order to break apart any particle agglomerates formed during the previous bed compaction process. Reload slurry into column and repack.

Please note, if buffers/salts are used, they may be insoluble in some organics and will crash inside the pores of the sorbent. In this case, flush the column with the mobile phase minus the salt. Then flush with the slurry solvent or solvent compatible with water, ACN and the solvent used for slurry.

Lastly, do not add the volume of slurry solvent recommended in the packing procedure to the wet sorbent unpacked from the column. Wet sorbent holds solvent. By adding the volume of solvent recommended in the recipe, you could exceed the volume of the column barrel. Add only as much solvent as it takes to raise the slurry volume to the final volume recommended in the packing recipe.

Some newer technology sorbents such as Luna, Jupiter, Synergi and Gemini are able to withstand numerous repacking operations at the recommended packing pressure for the specific phase. However, if there is a suspicion of broken/crushed particles (called "fines") present in the stored/re-cycled sorbent, these fines then need to be removed. To remove these fines or crushed particles, follow the below protocol:

Suspend the recycled sorbent in slurry solvent by stirring until homogenous. Decant the supernatant after the sorbent has visibly settled. Repeat this operation as needed until free of fines (supernatant is clear). Add the slurry solvent up to the total volume indicated in the packing recipe, re-suspend, and transfer into the DAC column barrel for repacking.

For long term storage of the HPLC sorbent for future reuse perform the following protocol:

Flush the column with 3 column volumes of acetone and unpack the DAC. Remove the top portion of the sorbent (5-10mm layer against the piston). Re-suspend the sorbent in acetone and pass through a 200 mesh sieve. It is important to pass the re-suspended sorbent through the sieve in order to break apart particle agglomerates formed during the bed compaction. Such agglomerates may fuse together and be difficult to disperse after the sorbent has been dried. Filter the sorbent on a Buchner funnel and lay it out in a tray. Leave the sorbent under an IR lamp to dry for 8 hours. Place tray in a vacuum oven for 2 hours at 80 °C. Store the dry sorbent in a sealed plastic container (confirm if the media manufacturer specifies a shelf life or temperature storage parameters for a specified sorbent). If the sample leaves severe contamination on the sorbent, flush the column with the best possible solvent capable of removing the contaminants (i.e. the solvent used to load the sample onto the column is a good choice). If this is impractical, use a mixture of the sample solvent with a slurry solvent or mobile phase component (i.e. DMSO in ACN).

It is important to clean out these crushed particles as they will lead to high pressures by building up on the outlet frit or giving low permeability in an already packed column bed (the probability of this is low with Phenomenex name brand sorbents as long as the protocols described above are followed).

Ultimately over time, sorbent will expire and frits will need to be cleaned and replaced frequently (clogged/dirty frits are also a know problem with process chromatography and sorbent reuse). Sorbent lifetime is often dependant on the sample. It should be noted that the same sorbent may last longer for one project compared to another project. Further, as stated above, company or laboratory policies will often dictate protocols for storage and usage of HPLC sorbents for large scale chromatography.