C Killeen 2025 McIndoe Lab

Using molecular sieves for solvent drying:

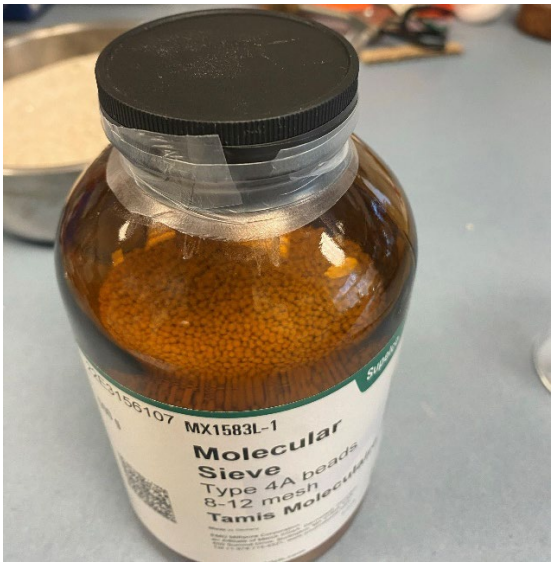
Molecular sieves are a modern alternative to other common drying agents such as calcium hydride, sodium/benzophenone, and phosphorus pentoxide, and benefit users in a few ways.

1. Safety:
    a. Molecular sieves are not flammable or corrosive
    b. They don't need to be quenched or otherwise treated after use before disposal
    c. They don't require special training or care in handling, as other drying agents do (e.g. Na, CaH_2)
2. Ease of use:
    a. Though there may be reason to anyway, solvents dried with sieves do not always need to be distilled afterwards
    b. Solvents dried with sieves can simply be stored without removal of the sieves.
3. Efficiency:
    a. Though they can take longer to completely dry solvents than other drying agents, molecular sieves get solvents **more dry** than other drying agents do. See: https://pubs.acs.org/doi/10.1021/jo101589h

Thus, in almost all cases in the lab, drying solvents with molecular sieves is preferable to other methods. Notably, molecular sieves cannot be used to dry acetone or other ketones, as they promote self-aldol condensation. For drying methanol or acetonitrile, 3 Å sieves must be used, as 4 Å sieves have large enough pore sizes that methanol and acetonitrile compete with water for site binding.

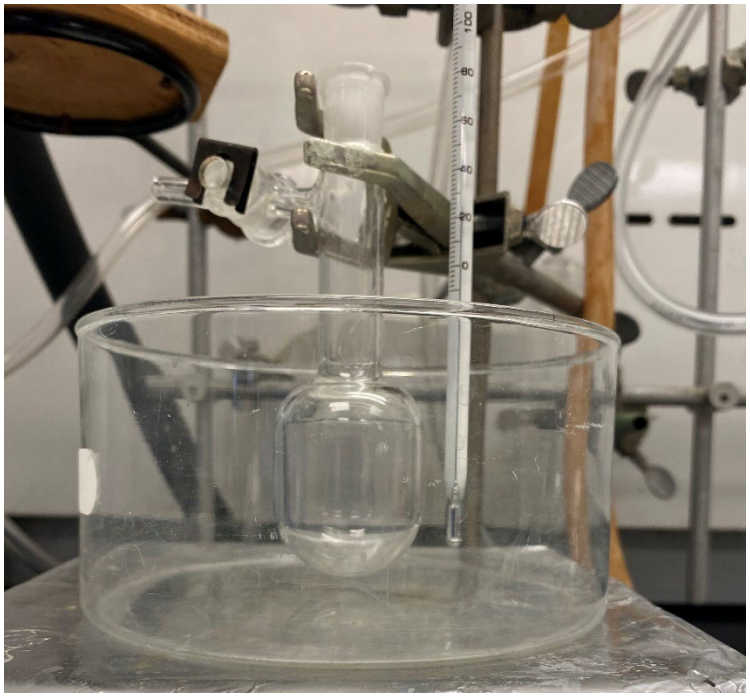
Activating molecular sieves:

Get sieves. The common ones stores stock are 4 Å, which are fine for most purposes (see above).

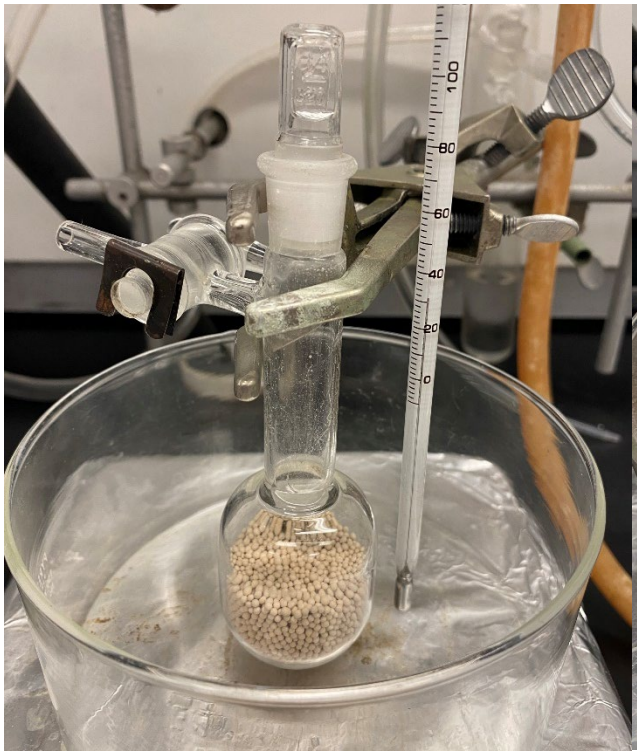


Note that this bottle must be kept tightly sealed with electrical tape to minimize water absorption. Next, get a Schlenk flask to put the sieves in. The top joint can be sealed with Teflon tape or grease, but if grease is used it must be thoroughly cleaned out of the joint before pouring out the activated sieves.

Next, the Schlenk flask should be clamped in a sand bath with a thermometer close beside it, with the bulb at the approximate level of the middle of the sieves. I prefer setting this up first, and adding the sand after.




The requisite amount of sieves should then be obtained, and if necessary (especially with older bottles) the dust should be sifted away. Don’t breathe this dust! This dust must be avoided for any mass spectrometric applications of the solvent, as it will plug up PEEK tubing and the ESI source capillaries. The sieves are then transferred to the Schlenk flask, and sand bath set up.




While the sand bath is heating up, the vacuum pump should be turned on and a liquid nitrogen trap should be installed on the Schlenk line. Then, the Schlenk flask of sieves can be connected to the vacuum manifold.

The sand bath should be heated to approximately 180-200 °C, and the sieves left under vacuum for 8-12 h, after which they should be cooled under a stream of dry nitrogen or air. Your molecular sieves are now active and ready for solvent drying!