Drying solutions

The name might seem odd but it refers only to the drying of organic solution. What it refers to is the removal of trace amounts of water from organic solution.

Many organic solvents are immiscible with aqueous solutions, but they are able to dissolve significant amounts of water because of their polarity i.e., diethyl ether dissolves 7 % of its mass in water!

Unfortunately, water is a compound that is very difficult to remove from many compounds, because they are either holding on to it well (i.e., alcohols) or the compound itself is steam volatile. Generally, the more polar the solvent is, the more hygroscopic it will usually be because it dissolves the water better. Thus, removing water and other impurities from a solution can be difficult but is necessary if

* the reagents are also sensitive towards water i.e., Grignard reagents
* where water has a detrimental effect on the yield or rate of the reaction.

The way of doing this is to add a small amount of an anydrous inorganic solid. This will absorb the water to form a hydrated salt and the now dry liquid can simply be decanted off.

The problem with drying agents is there is a tendency to add to much. So how much is enough?

Start off small.

Add a spatula tip and then swirl.

After the drying agent has settled, look at the solution. If it is transparent you are probably close to enough so only add a tiny amount more.

If the solution is cloudy, add another spatula tip and look again.

Once the solution begins to look clear, observe the drying agent itself. Does it look all clumpy or is it freely flowing? In other words, does it look the same as when you first added it? Drying agents clump and stick to the bottom of the vessel as they pick up water, so if the agent looks really clumpy and sticky, you need to add more. Continue to add drying agent until the newly added material appears unchanged and free flowing. Remember that the originally clumped material will never unclump.

There are various solids that can be used. The best ones have a high capacity and will dry completely.

*Capacity refers to how much water per gram the drying agent holds and complete means that drying equilibrium favors the hydrate*

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| Magnesium Sulphate | Magnesium sulphate has a high capacity, is complete in its drying and is rapid.. Disadvantagesit is normally available in a powder form and must be filtered out. magnesium is a very strong Lewis acid and as such, is not inert to all functional groups. For example, epoxides are sensitive to magnesium. |
| Sodium Sulphate:  | Sodium Sulfate is the most widely used drying agent. It is very similar to magnesium sulfate in its capacity, but it is less complete (will leave more water in solution) and it is slower in terms of its rate. Sodium sulfate has the advantage in that it is less reactive and in granular form, is very easy to remove from liquids. The liquid can often be decanted off the drying agent without filtration. |
| Calcium Sulphate | Calcium sulfate has a low capacity, but it is very complete and rapid. This means that you will have to use more of it to dry a solution but it will do a good job. Calcium sulfate can be purchased in a chunky form, which is very convenient to work with.  |
| Calcium Chloride | Calcium chloride is very much like calcium sulphate However, it is not compatible with hydroxy (alcohol, phenol), amino (amine, amide) and carbonyl (acid, ketone, ester) functions due to basic impurities. |
| Potassium Carbonate | Potassium carbonate is basic and as such, is generally used in basic media. It is of average capacity, completeness and rate.It cannot, of course, be used to dry acidic solutions as it will react. |
| Molecular sieves | These need to be heated in an oven beforehand to make sure they are properly dehydrated but are quite efficient and quick. The advantage is that they can be simple re-dried after each use. |