Chapter 15: Drying Organic Solutions

The process of synthesizing and isolating an organic compound often results in an organic compound or solution contaminated with traces of water. For instance, in aqueous extractions some water will be transferred into the organic phase because of the partial solubility of the organic phase in the water.

15.1 Saturated Aqueous Sodium Chloride

The bulk of the water can often be removed by shaking or washing the organic layer with saturated aqueous sodium chloride, also known as brine. The salt water works to pull the water from the organic layer to the water layer. This is because the concentrated salt solution wants to become more dilute and because salts have a stronger attraction to water than to organic solvents.

15.2 Solid Drying Agents

Final traces of water are removed by treating the organic solution with a drying agent. A drying agent is an inorganic salt that readily takes up water to become hydrated. The most commonly-used of these salts are listed in Table 15-1, along with their properties.

Table 15-1: Commonly-used drying agents.

Name	Capacity	Speed	Applications
Sodium sulfate, Na_2SO_4	High	Slow	Generally useful
Magnesium sulfate, $MgSO_4$	High	Fast	Not used for very acid-sensitive compounds
Calcium chloride, $CaCl_2$	High	Medium	Used for hydrocarbons

In order to be effective as a drying agent, an inorganic salt should quickly absorb large quantities of water. The above inorganic salts absorb water at different rates and to different degrees. The other requirements for a drying agent are that it be insoluble in the organic solvent and that it not react with either the compound or the solvent. Calcium chloride reacts with oxygen- and nitrogen-containing compounds and therefore is useful only for hydrocarbons or halides. Magnesium sulfate is a Lewis acid and might react with strong organic bases. It also reacts with water exothermically so care must be taken when used with a low-boiling organic solvent. In the organic labs, you will use sodium sulfate almost exclusively. This is due to its high efficiency as a drying agent and its ease of removal due to the large granular particles.

Drying agents do not require any special handling, nor do you need to look up the physical data for them for your lab report. While you must not ingest them, they are relatively safe, nontoxic, nonflammable, and nonreactive. Do keep them away from skin and eyes because their desiccant action might cause irritation.

15.3 Procedure for Using Solid Drying Agents

When the Lab Manual states: "Dry over…" it refers to the process below. Usually, you will perform a wash with saturated sodium chloride solution to remove the bulk of the water before treating with an inorganic salt.

1. Treat the solution with the drying agent.

Add a small amount of the solid drying agent directly to the organic solution. Swirl the solution and observe the drying agent; if it is all clumped together, add more. Continue swirling and observing the solution for 5–15 minutes, adding more drying agent only until a fresh addition no longer forms clumps.

Of the drying agents in the above table, magnesium sulfate is usually a fine powder and the rest are of a larger particle size. Each will have a slightly different appearance when "clumped" and practice will make you better at judging whether or not the inorganic salt is wet or dry.

There is no set "rule" as to how much drying agent needs to be added. The amount required depends on the amount of water in the solvent solution that you are drying, and this amount varies from solvent to solvent and experiment to experiment. Use as much as it takes to dry the solution. In most cases, drying is as complete as it will get in about 5–10 minutes.

2. Remove the drying agent from the organic solution.

You can either filter the solution by gravity filtration or decant the solvent from the desiccant (below). If the powder is quite fine (as when using magnesium sulfate) or if the volume is large, gravity filtration is the method of choice.

In small scale preparations, a large portion of the solution will be lost due to soaking into the filter paper. In these situations, decanting is the method of choice because there is less solvent loss. To decant, carefully pour the solution into another flask, leaving the unwanted drying agent behind. A glass rod can aid in the transfer both to keep the desiccant behind and to draw the liquid into the new flask.

In very small scale reactions, it may be easier to remove the liquid from the drying agent simply by drawing it off with a Pasteur pipet. Squeeze the bulb of the pipet, carefully place it flush with the bottom of the flask, and slowly draw liquid into the pipet, leaving the desiccant behind. Squirt the liquid into a fresh, clean flask.