

Water Sensitive Reactions

Some starting materials, reagents, and intermediate products used in organic reactions react easily with water. These include borane-THF, aluminum chloride, Grignard reagents, and anhydrides. In order to run a reaction with these compounds, the reaction needs to be protected from water. Several different methods are available, depending on the circumstances of the reaction and how sensitive the reaction is.

Procedures:

- With highly water sensitive compounds, it is important to dry the glassware before starting the reaction. The silicon-oxygen bonds in the glass attract a layer of water molecules along the surface which can interfere with more sensitive reagents. The two ways to do this are placing the glassware in a drying oven for 24 hours, or by flame drying. Oven drying is safer and easier, but takes time – it is usually used with conical vials. Flame drying is quicker but can only be done with round bottom flasks, because the thick base of a conical vial would heat unevenly and crack.
- After a closed environment is established, reagents must be added without introducing more water. For liquid reagents, the compound or solution to be added is drawn up into a syringe, the needle is poked through a septum, and the liquid is pushed out into the reaction. For solid reagents, the only way to totally avoid water is to weigh and add them in a glove box; we will simply weigh them out quickly in the air, then add them either by opening the top quickly or by making a solution and adding it with a syringe.
- While the reaction is running, it is protected from water vapor in the air by using a drying tube. This is a tube that fits into the top of a reflux condenser or Claisen-head adapter, and is filled with a layer of cotton, a layer of anhydrous calcium carbonate pellets, and another layer of cotton – the cotton holds the calcium chloride in place. The drying tube should always be secured with a yellow clip to prevent it from falling out and breaking. The calcium chloride absorbs water from the air, but still allows air to pass in and out of the reaction vessel, thus preventing pressure from building up in the reaction vessel due to heat or vapors being created.
- When the reaction is over and you are ready to begin isolating the product, it is no longer necessary to protect the reaction from water. The drying tube can be removed, and anhydrous reagents are no longer necessary. In fact, water is often added at this stage to quench the reactive reagent before continuing.