As a general rule, consult a copy of Perrin (Purification of Laboratory Chemicals, 4th Ed; [http://books.google.com/books?id=SYzm1tx2z3QC](http://books.google.com/books?id=SYzm1tx2z3QC)) for further details on how to purify your solvents. These days many laboratories will use a commercially available solvent purification system, others will distil solvents using more traditional techniques.

Tetrahydrofuran, dichloromethane, dimethylformamide, chloroform, acetonitrile, methanol, diethyl ether and toluene are all commonly used solvents, and in many cases they are required in anhydrous form. In some cases there are multiple ways to dry a given solvent. Here are some suggestions:

### Specific solvents

- **THF** (Tetrahydrofuran): distilled from sodium benzophenone ketyl. Add sodium wire and benzophenone to a volume of THF (pre-dried over calcium hydride or 4A molecular sieves), heat at reflux/under nitrogen for several hours until the solvent turns deep blue in colour. This indicates the solvent is dry, and you can distill off the volume you require. With time (weeks-maybe months if used correctly) the still will turn orange, this indicates that it is time to make a new one!

- **DCM** (Dichloromethane): pre-dry over calcium hydride, then distil over calcium hydride... unfortunately no colorimetric indicator to tell you when the solvent is dry.

- **MeCN** (Acetonitrile): pre-dry by shaking with type 4A molecular sieves, the distil over calcium hydride. Dried acetonitrile can be stored over 4A molecular sieves.

- **Diethyl ether**: distilled from sodium benzophenone ketyl (see THF), will turn a deep purple/blue colour when dry.

- **Methanol**: For most purposes, drying over 3A molecular sieves overnight followed by distillation is sufficient. Alternatively, the methanol can be dried from magnesium methoxide. Magnesium turnings (5 g) and iodine (0.5 g) are refluxed in a small (50-100 mL) quantity of dry methanol (from a previous batch) until all of the magnesium has reacted. The mixture is diluted (up to 1 L) with reagent grade methanol, refluxed for 2-3 hours then distilled under nitrogen.

- **DMF** (N,N-dimethylformamide): Decomposes slowly at room temperature and more rapidly at reflux, releasing dimethylamine and carbon monoxide. This decomposition is catalysed by acidic and basic impurities, and standing DMF for several hours at room temperature with basic drying agents such as calcium hydride or sodium hydroxide leads to noticeable decomposition. Dry DMF can be prepared by drying overnight over barium oxide or 4A molecular sieves, followed by decantation of the drying agent and vacuum distillation (~20 mmHg is a sufficient vacuum to lower the boiling point over DMF to a reasonable value). Dry DMF can be stored over 4A molecular sieves.

### General Precautions

As a general precaution ethers can produce explosive peroxides, making distillation of these solvents hazardous. If one is available, use a blast guard (really thick piece of pyrex? attached to a stand), and do not use stills once they have passed their use-by date. The process of distillation also removes the stabilisers that are added to the ethers, consequently distilled ethers should not be stored for long periods of time. For directions on handling sodium etc. consult 'reagent specific hazards'.

### How to quench a solvent still

Stills that use metals (THF) should be quenched by pouring any excess solvent into a large container filled with
isopropanol or tert-butanol. The reaction may become exothermic, so addition of the solvent to the alcohol must be done slowly. The remaining metal in the still can be quenched like normal.

You can reuse 4A molecular sieves from most applications, though you should discard those used in the preparation of DMF. Sieves can be regenerated by heating at 350° C for 24 hours or under vacuum. The sieves should cool in a desiccatior or they will become saturated with moisture again.