How to Degas Solvents

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For many (possibly most) organic reactions, the presence of oxygen in a reaction is more detrimental than traces of H_2O. If a reaction fails unexpectedly, it is a good idea to ensure an oxygen free environment, part of which entails degassing the reaction solvent.

For certain reactions, degassing is essential:

- **Any reaction heated above 120°C and many reactions heated for a prolonged period over 80°C.** These reactions are typically performed in a sealed tube and are often run by heating over the boiling point of the solvent. Especially in the case of intramolecular reactions, the formation of a yellowish or brown color in the reaction is indicative of the presence of oxygen. High temperature reactions are extremely sensitive to oxygen and even the act of opening the reaction to take a sample for TLC analysis can introduce enough oxygen to destroy or damage the reaction
- **Organometallic reactions.** For many organometallic reactions, it is essential to provide an oxygen free (and sometimes N_2 free) environment. It is not uncommon to see strict procedures prescribing 10 cycles of freeze-pump-thaw (see below) degassing for the use of sensitive catalysts.
- **Radical and photochemical reactions**: unless oxidization is the desired product, radical reactions, for obvious reasons, must be degassed.
- **Substrates containing thiols, thioethers, phosphines, electron-rich aromatic, etc.**: Heating, solvents, or the presence of other reagents can often induce the oxidation of such substrates even when otherwise stable.

Methods of Degassing

- **Freeze-Pump-Thaw**
  This is the most effective methods for solvent degassing. A solvent in a sealed Schlenk or heavy wall sealed tube is frozen by immersion of the flask in liquid N_2. When the solvent is completely frozen, the flask is opened to the vacuum (high vacuum) and pumped 2-3 minutes, with the flask still immersed in liquid N_2. The flask is then closed and warmed until the solvent has completely melted. This process is repeated (usually three times) and after the last cycle the flask is backfilled with an inert gas. Degassed solvent in a sealed Schlenk flask can usually be kept for 1-2 days.

- **Atmosphere Exchange Under Sonication**
  Solvents can be roughly degassed by repeated sonication under light vacuum (i.e.

house vacuum) for 0.5-1 min and replenishing the atmosphere with an inert solvent. By using 5-10 cycles, degassed solvents for HPLC and some reactions can be obtained quickly.

- **Purging**
  Of the methods listed here, purging is the least effective way of degassing solvent, however it is acceptable for some applications, particularly when large amounts of solvent need to be roughly degassed. As it sounds, purging consists of bubbling an inert gas (usually N_2 or Ar) through the solvent for 30 min - 1 hour. Care should be taken to prevent solvent evaporation and especially the condensation of water in the solvent by using an appropriate setup.