

## *Refluxing a Reaction*

Organic reactions run faster at higher temperatures and slower at lower temperatures. Many reactions commonly run in an organic laboratory need to be heated in order to proceed at a satisfactory rate. The most convenient way to heat a reaction is by boiling it in a solvent in a conical vial or round bottom flask. It is easy to observe whether or not a liquid is boiling, and because of the laws of thermodynamics, once boiling begins the temperature cannot rise any higher. To control the temperature of the reaction, simply choose a solvent with a boiling point at this temperature.

To prevent the solvent from boiling away, a reflux condenser is used. This is a glass column with a second column surrounding it through which cool water flows. As vapor from the boiling solvent rises into inside column of the reflux condenser, it is cooled by the jacket of water on the outside and condenses. It then falls back into the solution. In this way, you can maintain a reaction at the boiling point of the solvent indefinitely, as long as the water in the reflux condenser is cool enough to condense all of the vapor.

Any time you heat a liquid, you should always stir it to avoid bumping. Bumping occurs when only the liquid at the very bottom is hot enough to boil, and it builds up pressure and bursts suddenly, sometimes spattering out of the container.

Also, you should never heat a closed system – pressure can build up and burst the glassware. The reflux condenser is open at the top to prevent pressure build-up. If you need to protect a reaction from moisture, a drying tube filled with calcium carbonate is used so that water cannot get in but air can get in and out to prevent pressure build-up.

### Procedure:

- To reflux a reaction, add all of the reagents to a conical vial or round bottom flask as directed. Add a spin vane (conical vial) or a spin bar (round bottom flask) and top it with the reflux condenser, securing it with a clamp (attach the clamp to the condenser - if you attach it to the reaction vessel, the plastic sleeve will melt).
- Attach tubing to the condenser so that water flows from the tap into the bottom of the condenser, and out of the top and into the drain. (If you attach the tubing backwards, the water will just run down part of the inside of the condenser instead of filling it up, and won't do a good job of cooling.)
- Turn on the stirrer and the water (not too fast – all you need is for the water to be moving through the condenser fast enough to cool the vapor – no need to waste it), and then gradually heat the reaction until you can see the solvent refluxing. You should observe liquid forming in the condenser and flowing back into the reaction. It may appear as droplets or as a continuous stream. You can often see a reflux line where solvent is condensing and then running back down.
- When you are finished refluxing, allow the reaction to cool, turn off the water, remove the spin vane with forceps, and proceed with the instructions. Be patient while waiting for it to cool - do NOT lift the vial or flask off of the stirrer/hot plate by raising the clamp attached to the reflux condenser – nothing is holding the vial to the condenser, and it can fall and break, spilling your reaction.