Evaporating an Organic Solution

One of the advantages of organic solvents is that they have boiling points that are much lower than water, and are relatively easy to evaporate. This means that if you have a desired product in a solution, all you need to do to get the product out is to evaporate the solvent. (This would be like boiling away the water in a sample of sea water to obtain the salts that are dissolved in it.)

Once the solution is dry (free from water), the solvent can be evaporated by using a device called a rotary evaporator, or rotovap for short. The idea behind the rotovap is to immerse the solution in a hot water bath and turn it. The vapor travels up the column until it encounters the coils which are cooled by ice water circulated by a small pump. The vapor condenses and runs down into a collection flask. Once all the solvent has evaporated, the product is left behind in the flask as a solid or an oily liquid.

Low boiling solvents like diethyl ether and dichloromethane are easily evaporated at atmospheric pressure. Solvents with higher boiling points, such as hexanes, ethanol, or methanol, are more difficult to evaporate, and require reduced pressure to raise their boiling points. Recall that boiling point is dependent on both temperature and pressure – at lower pressures the boiling point becomes lower as well. The vacuum pump may be connected to the rotovap, and the pressure controlled by the valve at the top of the cooling coils. A second dry ice-cooled trap should be used to prevent any vapors that escape the coils from being sucked into the pump.

<u>Procedure:</u>

- To rotovap a solution, first obtain a clean, dry round-bottom flask with a 24/40 joint (your 50 ml and 100 ml round bottom flasks have joints this size). If the solution you are rotovapping will be your final product, weigh the flask first so that you can obtain the mass of the product when you are finished.

- Transfer your solution to the flask. If you have just dried the solution with sodium sulfate, avoid transferring any of the crystals along with it, and remember to rinse the crystals and add this solvent to your solution.

- Attach the flask to the rotovap using a green clip (larger ring towards the flask). Be careful not to let any of the water get into the flask. Start the flask turning by pushing the large gray button. Make sure the pump is pumping ice water through the coils (if you touch the top of the pump you can feel it vibrating). Watch the solvent collect in the collection flask.

- When all of the solvent has evaporated, stop the flask from turning by pushing the gray button again. Carefully remove the flask (use a twisting motion rather than trying to pull it straight off). Because of the small amounts we are using, it is common for a small amount of solvent to be trapped in the column and run back down when you stop the flask from turning. The best way to get rid of this is simply to hold the flask in the hot water for 30 to 60 seconds to allow this last bit to evaporate into the air. You can often see it condense on the neck of the flask, then disappear. When your solid appears dry or a liquid as oily as it's going to get, you're done.

- If you were rotovapping a final product, wipe any water droplets off of the outside of the flask and allow it to cool. Then weigh the flask again and subtract the tare mass (the mass of the empty flask) from it to obtain the mass of product you obtained. Make sure to use the same balance as you did for the tare weight.