CHEM 321: USEFUL PROCEDURAL GOODIES

Recrystallization Technique: Work in a beaker. Choose size so that it is ~1/2 to 3/4 full after all solvent has been added. ***Keep it covered*** with a watchglass unless you are actively working inside it. Put your crude solid into the beaker and dissolve in a suitable solvent as you heat the beaker. Often ethanol is a good choice, but consult the protocol you are using. Use the smallest volume of solvent that, when at its boiling point, is able to completely dissolve the solid (trial and error). The key to success is having a hot **clear** solution that you cool slowly **(keeping it covered)** so that crystals form slowly and, in so doing, exclude impurities.

Sometimes there is insoluble matter (does not disappear if you add a little more solvent). Insoluble material needs to be removed by hot filtration – see below.

After you have a clear, hot solution, you can do a little "quick and dirty" work in a small test tube to speed things along. Pour about 1 mL of the hot solution into the tube. Swirl the tube vigorously to cool its contents fast (if no crystals form, cool in ice).

- **If crystals do form in the tube**, this solvent will work well. Swirl the tube contents, return them to the beaker, and cover it. Reheat if necessary to get a clear solution, then cool slowly, eventually on ice.
- **If no crystals form in the tube,** either you have used too much solvent, or this solvent won't work.
  - If water is miscible in the solvent, add water slowly with mixing to see if the mixture becomes cloudy. If it becomes cloudy, stir and scratch gently to see if crystals will form. If they do, note the approximate percent incease in volume due to added water. Return the tube contents to the beaker, add the appropriate amount of water, cover, reheat as needed to get a clear solution, and cool as above.
  - If adding water doesn't help, or if water is not compatible with the major solvent, you'll need to boil off at least half of the solvent. Return the contents of the tube to the beaker, boil it down, and then start again at the top of this paragraph.
- Instructors may be able to help in hard cases.

Filter crystals on a **Hirsch funnel** and let air-dry for at least a day before taking a melting point.

HOT FILTRATION **(to remove insoluble material from a recrystallization in progress)**

Assemble the apparatus shown using a 400 ml beaker and stemless funnel.

Fold the largest piece of filter paper you can find into a cone and put into funnel. Cover with watchglass.

Bring beaker solvent to a slow boil and allow vapors to heat up all glassware.

Heat suspension you wish to filter, then pour as much as possible into funnel.

Cover and continue to boil *very gently* (don't let it go dry!).

Desired material is in filtrate.