Chem 224 Recrystallization WS2008

Steps in recrystallization

1. **Solvent Selection**

   grind sample to powder, examine at RT and BP of solvent, "like dissolves like"
   want low solubility of compound at RT, high solubility of compound at BP of solvent,
   also want BP of solvent < MP of compound, to avoid oiling out

2. **Preparation of hot, saturated solution**

   decolorize with activated carbon if solution has color,
   add ~5% excess hot solvent, then gravity filter hot through fluted paper
   skip this step if solution is colorless and no insolubles are present

3. **Slow cooling, crystal formation**

   slowly cool filtrate to RT, then cool in ice to minimize solubility of compound

4. **Separation of crystals, washing**

   vacuum filter, disconnect vac. to rinse crystals, reconnect to remove rinse solvent

5. **Drying of crystals**

   air dry in vac funnel. press crystals with shell vial or weighing paper
   high BP solvents may require overnight drying in desk
   determine MP, % recovery

Excess losses of product due to

1. **Too much solvent added**

   compound can be recovered by evaporating excess solvent and refiltering

2. **Too much charcoal added**

   compound also adsorbed on carbon, may be hard to recover

3. **Crystallization in funnel during filtration**

   heat funnel, rinse with hot solvent, evaporate excess solvent afterwards

4. **Filtration before crystallization is complete**

   some compounds form crystals very slowly, eg. cane sugar
   stirring and scratching of container with glass rod may induce crystallization
   allow covered solution to stand in desk to see if crystals eventually form