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

Recrystallization-Calculation of % Theoretical Recovery

Need to know the solubility of the sample at RT and the solubility at the BP of the solvent.

Use a 1.00 gm sample for simplicity

Temperature	Solubility
RT	1.00g/100. ml
BP	1.00g/10.0 ml

Assuming a minimum amt of solvent is used to dissolve the 1.00 g sample at the solvent BP

(1.00 g of sample)(10 ml solvent/1 g of sample) = 10.0 ml solvent needed to dissolve sample at BP

Assuming no loss of solvent by evaporation on cooling, we still have 10 ml of solution,
but the solubility of the sample is lower

(10.0 ml)(1.00g/100 ml) = 0.100 gm left in solution on cooling

% recovery = ((amt of sample at BP) – (amt of sample in solution at RT)) / original sample mass x100%

(1.00gm-0.100 gm)/1.00 gm x 100% = 90.0 % maximum theoretical recovery

If 20.0 ml of solvent were used instead of the 10.0 ml actually required, the recovery would be reduced

On cooling, we have (20 ml)(1.00 g/100ml) = 0.200 gm left in solution, so

% = (1.00 gm-0.200gm)/1.00 gm x 100% = 80% maximum recovery

In general, the greater the difference between the solubility at RT and the solvent BP,
the greater the % theoretical recovery, and any excess solvent will always result in a lower % recovery.
If we cool the sample in ice, solubility is reduced further and a larger yield may be expected.