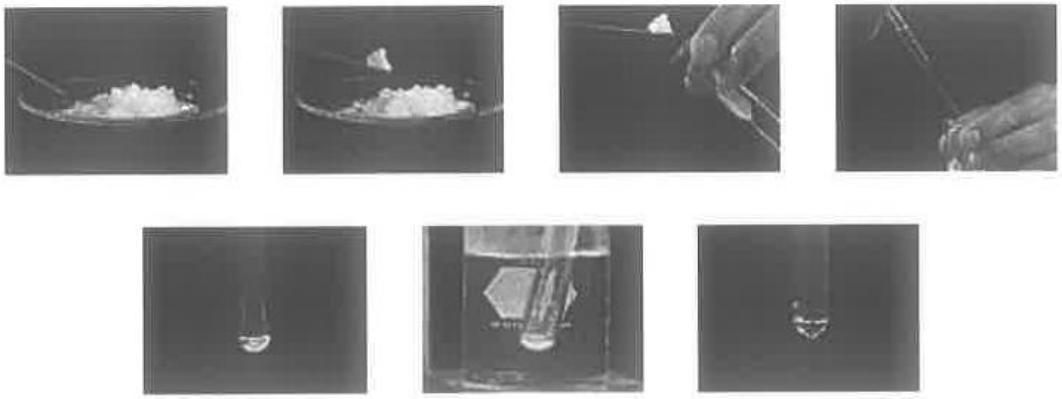
EXP. NO. (3)

RECRYSTALLIZATION



The purpose of exp. :

Separation & Purification of solid organic compound.

Theory:

Recrystallization is a laboratory technique used to purify solids, based on their different solubility, and can increase the solubility with heat.

"like dissolves like", which means that a nonpolar compound will dissolve well in a nonpolar solvent, and a polar compound will dissolve well in a polar solvent.

***The steps in the recrystallization of a compound are**:

a. Find a suitable solvent for the recrystallization.

b. Dissolve the impure solid in a minimum volume of hot solvent.

c. Remove any insoluble impurities by filtration.

d. Slowly cool the hot solution to crystallize the desired compound from the solution.

e. Filter the solution to isolate the purified solid compound.

*** the properties of suitable solvent used in recrystallization:**

1. It should not dissolve the compound in low temperature (at room temp.) but readily dissolve the compound in high temperature (at its boiling temp.).

2. It should not dissolve the impurities in high temperature.

3. It should not react with the compound to be purified.

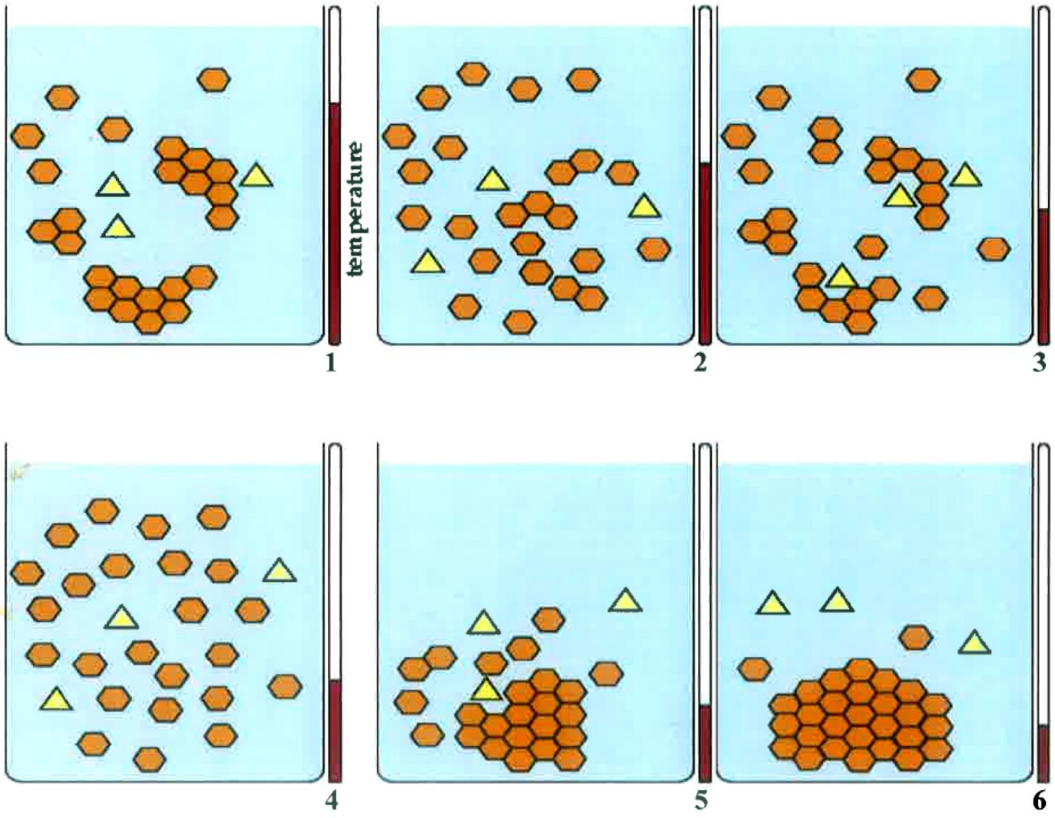
4. It should have a boiling point lower than the melting point of the solid purified

5. The solvent should be volatile enough to be easily removed from the Crystals after the compound has crystallized

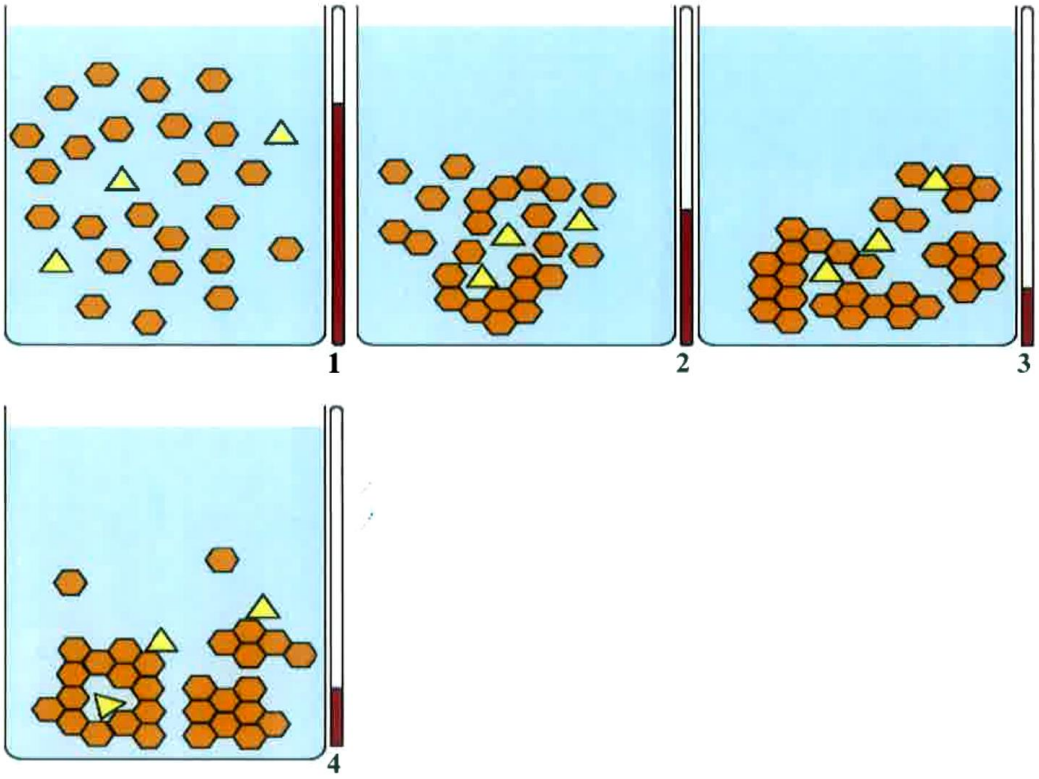
6. not toxic, available, cheap, inflammable

Crystallization: Slow cooling vs rapid cooling:

This first series of diagrams shows what happens if you let a crystallization proceed slowly: first by setting the flask at room temperature undisturbed until crystals form, and then carefully on ice. The yellow triangles are an impurity in the hot solution of orange hexagons. If the solution is allowed to cool slowly, the impurities may sit down briefly in the growing crystal lattice, but they soon leave as a compound with a more suitable geometry comes in to take their place. Suitable hexagons stay more readily in the growing lattice, and eventually pure crystals of orange hexagons are formed.



This second series of diagrams show what happens if you cool the solution too quickly. The yellow triangle impurities are trapped inside the crystals being formed by the orange hexagons, thus, the crystals isolated are impure.



Note:

that slow crystallization gives larger crystals than this series of fast crystallization. Small crystals have a large surface area to volume ratio and impurities are located on the surface of the crystals as well as trapped inside the matrix.

Procedure:

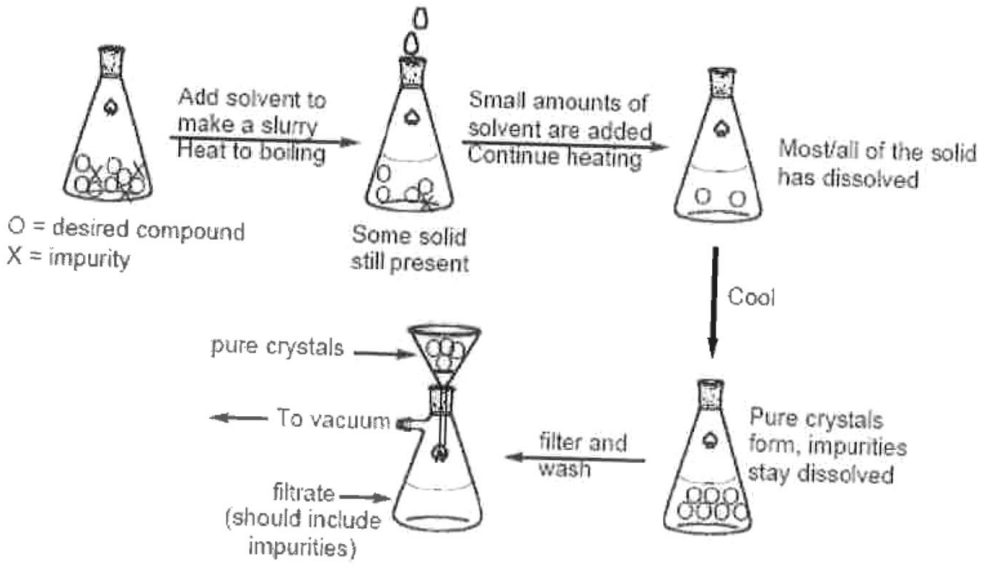
Add (1gm) of impure compound (benzoic acid) to a conical flask. Add (10ml) of hot suitable solvent(water) and heat in hot plat. Remove the flask from the hot plat When all of the benzoic acid has dissolved, the impurity did not dissolve, a hot filtration is removing the impurity. Left the solution to cool to room temperature, and then place it in an ice bath for few minutes. Isolate the resulting precipitate by filtration and wash with cold water. Dry the precipitate.

Notes:

*Always use a minimum amount of hot solvent to dissolve the crystals.

*measurement of the melting point range immediately gives an indication of whether the sample is pure or not.

* finely powdered activated charcoal, called (decolorizing carbon) is added to the mixture before the hot filtration, in order to remove colored organic impurities.



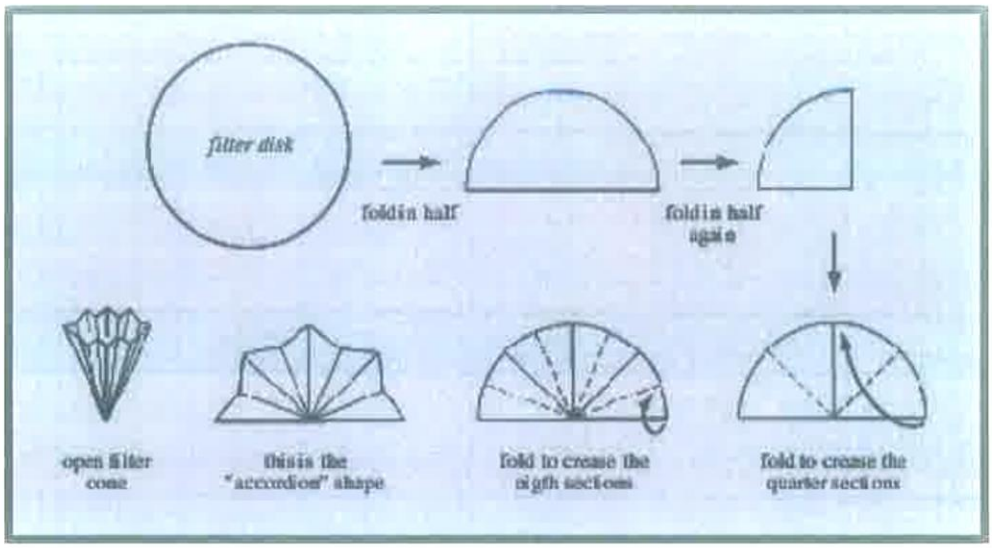


diagram of how to fold fluted filter paper

Procedure for Crystallization:

<u>The photos below illustrate the process of crystallization.</u>