Name of experiment: Recrystallization.

Purpose of experiment:

a) To purify samples of organic compounds that are solids at room temperature
b) To dissociate the impure sample in the minimum amount of an appropriate hot solvent

Recrystallization is the primary method for purifying solid organic compounds. Compounds obtained from natural sources or from reaction mixtures almost always contain impurities. The impurities may include some combination of insoluble, soluble, and colored impurities. To obtain a pure compound these impurities must be removed.

The six steps used here to recrystallize a compound are.

1- carry out solubility tests to determine a suitable solvent.
2- dissolve the solute in a minimum of near boiling solvent.
3- allow the solution to cool slowly and undisturbed to room temperature (RT) then possibly to ice temperature.
4- collect the crystals by filtration.
5- rinse the crystals with a minimum amount of ice-cold solvent.
6- allow the crystals to dry.

Chooses a solvent for Recrystallization

The proper choice of a solvent is an important part of the art of crystallization.

The ideal solvent should.

1- Be chemically inert toward the solute.
2- Dissolve the solute readily at its boiling point but sparingly at low temperature ($0 – 25\ ^0C$).
3- Dissolve impurities either very easily or not at all.
4- Not be flammable of low cost and of low toxicity.

Practically to choose a good solvent take about **0.1gm** of the compound to be purified (a pure sample) and try to dissolve it in **1ml** of the solvent if it dissolves in the cold solvent the solvent will not be good for recrystallization if it dissolves in the solvent with heating, the solvent will be good for recrystallization. If it does not dissolve in the solvent even with heating, the solvent will no; be good for recrystallization. Solvent extensively used for recrystallization include water, ethanol, chloroform, ether, acetone, and benzene.

Procedure (crystallization of benzoic acid):

1- Heat some solvent (water) to boiling .Place the solid (benzoic acid) to be recrystallized in an Conical flask.

2- Pour a small amount of the hot solvent (water) into the flask containing the solid (benzoic acid).

3- Swirl the flask to dissolve the benzoic acid.

4- Place the flask on the steam bath to keep the solution warm.

5-If the benzoic acid is still not dissolved, add a tiny amount more water and swirl again.

6-After a while, crystals should appear in the flask.

7-You can now place the flask in an ice bath to finish the crystallization process.

8-You are now ready to filter the solution to isolate the crystals. Remove the filter paper from the Buchner funnel when done.

9- After your crystals are filtered from the solution, put them on a watch glass.

10- Let the crystal finish drying on the watch glass.

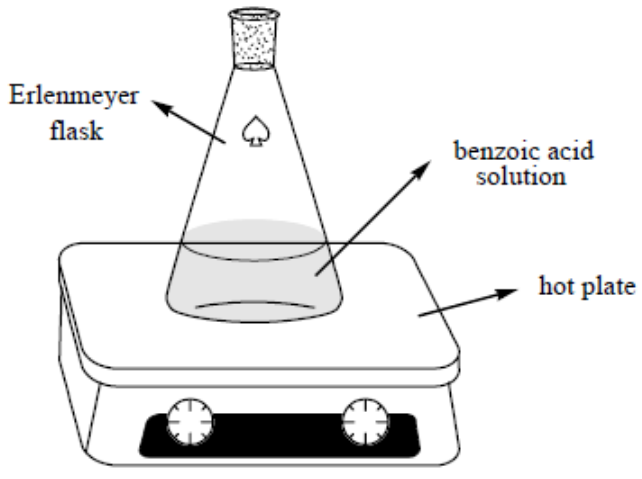

Fig 1- Dissolving benzoic acid