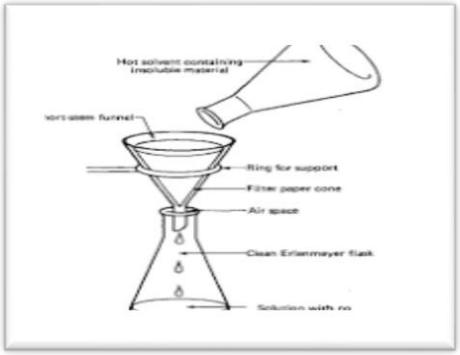
Recrystallization



Recrystallization

Solid organic compounds when isolated from organic reactions are usually impure , they are usually contaminated with small amounts of other compounds that are produced along with the desired compound . the purification of impure crystalline compounds is usually done by recrystallization from suitable solvent or a mixture of solvents.

The purification of solids by crystallization is based upon differences in their solubility in a given solvent or a mixture of solvents.

Choosing a solvent for recrystallization

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

The ideal solvent should :

١-be chemically inert toward the solute.
٢-dissolve the solute readily at its boiling point but sparingly at low temperatures (٠ – ٢٥ C).
٣-dissolve impurities either very easily or not at all .
٤- not be flammable , of low cost , and of low toxicity .

Practically , to choose a good solvent take about ٠.١ gm of the compound to be purified (a pure sample) and try to dissolve it in ١ ml 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 not be good for recrystallization . solvents extensively used for recrystallization include water , ethanol, chloroform , ether , acetone , and benzene .

Decolorizing Charcoal

Frequently , samples to be purified may contain soluble colored impurities that may cause the solution and the crystals to be colored when they should be colorless ; they dissolve in the boiling solvent and are adsorbed on the crystals produced upon cooling .

These impurities can be removed by treating the colored sample with decolorizing (activated)charcoal that is composed of fine carbon particles with a large active surface on which the colored impurities will be absorbed .

Charcoal is added to the hot solution before boiling and the solution is kept hot at or near the boiling point for about ٣-٥ minutes with shaking to wet the charcoal . the solution is then filtered through a fluted filter paper. No more charcoal than actually needed should be used because any excess amount will cause the desired compound to be adsorbed on the charcoal .

Charcoal is not added at the boiling point of the solvent because its particles function as thousands of boiling chips causing to boil over and foam .

Charcoal is not used in the recrystallization of phenolic compounds (Ar – OH) because it contains ferric ions that , on heating the solution for some time , can react with the phenolic hydroxyls forming red colored complexes , thus impairing the purification process .

Recrystallization using mixed solvents

This is applied when our compound is readily soluble in a solvent at room temperature . and at the same time is not soluble in another solvent . the two solvents must be completely miscible with each other such as alcohol and water, ether and pentane , and glacial acetic acid and water .

So the compound is dissolved in the solvent that it is soluble in , charcoal is used if required, and the solution is filtered to get rid of the insoluble impurities.

Then the other solvent (in which the compound is insoluble) is added to the filtrate gradually until turbidity appears . the mixture is then left aside to facilitate crystallization .

Procedure

Take an unknown weight of impure acetanilide and start dissolving it in a small volume of water with heating until the entire sample dissolves . remove the solution from the Bunsen burner and leave it inside for a minute to cool . then add a small quantity of charcoal and resume heating again and stirring for ٣-٥ minute . filter the mixture while being hot . leave the filtrate to cool at room temperature to induce crystallization . Observe the produced crystals .