

# Purification of Solutions

## Recrystallisation

- 1) Method of recrystallisation involves finding a solvent which is chosen so that the crude substance is very soluble in the hot solvent, but soluble only to a small extent in the cold solvent.
- 2) The crude substance is then dissolved in a minimum amount of the boiling solvent.
- 3) Impurities which are insoluble in the solvent used can then be removed by filtering the hot solution through a hot funnel (to prevent the solution from cooling and crystallising prematurely).
- 4) The resulting solution is then cooled. At this stage the solute will crystallise out, leaving the greater part of the impurities (which were soluble in the solvent used) in solution.
- 5) The crop of crystals can then be obtained by filtering off the solution.

## Technique of Recrystallisation

To the substance in a boiling tube (or beaker or conical flask) add just enough solvent to dissolve the substance. Gently heat to boiling and add more solvent until all the substance has dissolved. Filter hot if necessary. Allow to cool and crystallise (this process can be sped up by cooling in water). Filter. If an organic solvent is used it is advisable to do the heating under reflux.

## The Use of Melting Point to Check Purity

The purity of the final product can be checked by finding the m.pt.. A pure compound has a sharp m.pt. (i.e. the substance melts over a range of  $0.1^{\circ}\text{C}$ ), and the m.pt. is the same when the product is recrystallised. An impure substance has an indefinite m.pt. And the substance will therefore melt slowly and over a range of several degrees. The presence of impurities lowers the m.pt. And also makes it less definite. When the substance is purified by recrystallisation the m.pt. is higher and sharper. Generally, experimental determination of m.pt. involves placing the finely divided compound in a capillary tube, attaching it to a thermometer and placing them in a bath of suitable liquid (the temperature is noted when the compound melts).

E.g. If a compound is suspected of being  $\text{C}_6\text{H}_5\text{COOH}$ , then a small amount of the substance is mixed with a known sample of  $\text{C}_6\text{H}_5\text{COOH}$  and the m.pt. of the mixture is determined. If the mixture has the normal sharp m.pt. of  $\text{C}_6\text{H}_5\text{COOH}$ , then the suspected sample is in fact  $\text{C}_6\text{H}_5\text{COOH}$ . If the m.pt. is indefinite then the substance is not  $\text{C}_6\text{H}_5\text{COOH}$  and the m.pt. of the impure mixture will be lower than the actual m.pt. of  $\text{C}_6\text{H}_5\text{COOH}$ .