

Aim:- To purify an organic compound by crystallisation (Kromwahukalewtd)

Theory:- Crystallisation is one of the most effective techniques for purification of organic solids. Generally the original crude material obtained after a rxn is impure. They may be contaminated with other compounds which are formed ~~by~~ as a byproduct, owing to a side rxn.

The organic compounds have to be purified before using them. Several methods of purification are:

- 1) Crystallization 2) Sublimation 3) Extraction, 4) Distillation
- 5) Chromatographic adsorption.

The method employed depends upon the nature of the material to be separated & purified.

The crystallization method takes advantage of differences in solubility between compound & its ~~impurity~~ impurities.

The process of crystallization can be divided into following steps:

- 1) Selection of an appropriate solvent (or mixture of solvent) for dissolution of compound.
- 2) Dissolution of the compound using minimum amount of hot solvent.
- 3) Decolorisation with activated carbon (charcoal) in case coloured impurities are present.
- 4) Hot filtration to remove suspended solids.
- 5) Cooling of the filtrate to allow crystallization.
- 6) Collection of crystals by filtration & subsequent washing with appropriate solvent.
- 7) Drying of crystals.

1) Selection of solvent:- A solvent (or mixture of solvents) which dissolves the crude substance readily at high temperatures is considered to be a suitable solvent. Few examples of solvent in increasing order of polarity are: hydrocarbons (pentane, hexane, petroleum ether, benzene etc.) diethylether, chloroform, acetone, ethyl acetate (methyl acetate), ethanol, methanol, water etc. The following points should be noted while choosing a solvent for crystallization.

- i) Substances tend to be more soluble in chemically similar solvents (like dissolves like). A nonpolar compound dissolves in nonpolar solvent & vice versa.
- ii) It should have different solubility characteristics for compound & impurities.

- iii) The solvent should not chemically reactive towards the ^{Compound} ~~substance~~ to be crystallized.
- iv) It should well-formed crystals of the pure compound.
- v) It must be capable of easy removal from the crystals of purified compound.

2) Dissolution of crude substance:- After selecting an appropriate solvent, the crude ~~sub~~ compound is dissolved in minimum amount of hot solvent to form a saturated solution.

3) Removal of coloured impurities:- In case of coloured impurities, activated charcoal is added to the solution & then it is heated. The impurities get adsorbed onto the surface of activated charcoal, which has an extremely large surface area per gram. ~~If too little charcoal is added, the solution will still be coloured after filtration. If too much is added,~~

4) Hot filtration:- If the solution after removal of coloured impurities still contains suspended particles or undissolved impurities, it should be heated on water bath or directly depending on the nature of solvent. A hot filtration should be carried out through a fluted filter paper. If the solution is clear then hot filtration may be omitted.

5) Cooling of the clear filtrate:- The clear solution so obtained is to be left for cooling in a china dish or boiling tube ^{covered with perforated} at ^{room} temperature to induce crystallization. The size & shape of crystals is controlled by the rate of cooling, a slow to moderate rate of cooling is usually best as it will favour medium sized crystals having good shape.

A few steps can be used to facilitate the crystallization:

i) Some organic compounds do not crystallize readily, even from supersaturated solutions. A small crystal of pure compound may be added to induce the crystallization process. This technique is known as "seeding" where a pure crystal act as a nucleus for growth of new crystals.

ii) The crystallization can also be induced by scratching the sides or bottom of boiling tube with glass rod. This process provides a surface for the formation of crystals during crystallization.

iii) The excess solvent ~~are~~ should be removed by boiling followed by slow ^{cooling}.

6) Separation of crystals from mother liquor:- The crystals are then separated from mother liquor (liquid left after crystallization) by suction or vacuum filtration. The crystals are then washed by cold & pure solvents to remove the sticking mother liquor.

7) Drying of crystals:- The crystals should be dried bez of presence of solvent or moisture which may affect their melting point. A solid that has been crystallized from a volatile solvent is usually dried by allowing it to air dry at room temperature. Filtration using suction removes excess of the solvent thereby facilitating the drying process. For more effective drying, desiccators with suitable desiccants like silica gel, phosphorus pentoxide or fused calcium chloride may be used. Oven drying, if necessary, can be carried out at temperatures below melting point of the compound.

Procedure:- 1) Preparation of crystals of benzoic acid:

Take 1g of benzoic acid in a boiling tube & add ~~minimum~~ small amount of distilled water to it. ~~Heat~~ Heat the content on flame/bunsen burner directly, till whole of the benzoic acid dissolves to give a clear solution. If required, hot water may be added in slow to avoid the loss of water due to evaporation. Allow the hot solution to cool at room temperature. Allow the solvent to evaporate overnight. Scrap the crystals with the help of spatula on a watch glass.

Weigh the crystals formed & report the yield of crystals in gram.

2) Preparation of crystals of m-dinitrobenzene (or naphthalene)

Dissolve 2.0g of crude m-dinitrobenzene (or naphthalene) in minimum amount of ethanol by heating in ~~the~~ boiling tube on water bath (do not directly heat on flame) till a clear solution is formed. Allow the solvent to evaporate. Dry the crystals & weigh it & report the yield of crystals in gram.

Result:- 1) The yield of crystals of benzoic acid = ... g

2) The yield of crystals of m-dinitrobenzene = ... g

3) Shape of crystals of benzoic acid is ~

4) Shape of crystals of naphthalene is ~