Purification of Chemical Substances by Crystallisation

For chemical purposes the substances should be pure, completely free from any type of impurity. Impurities may be soluble or insoluble in the solvent in which the substance under consideration dissolves. So, method of purification of the substance depends on the nature of the impurity present and there are large number of methods available for the purification of the substance such as filtration, sedimentation, decantation and crystallisation. The simple laboratory technique applied for the purification of the substances by crystallisation is described below.

Process Of Crystallisation

The process of crystallisation involves following steps :

1. Preparation of Solution of the Impure Sample

1. Take a clean beaker (250 ml) and add powdered impure sample under consideration in it (~ 6.0 gm).
2. Add distilled water (25-30 ml) and stir contents gently with the help of glass rod giving circular motion as shown in Fig. 5.1.

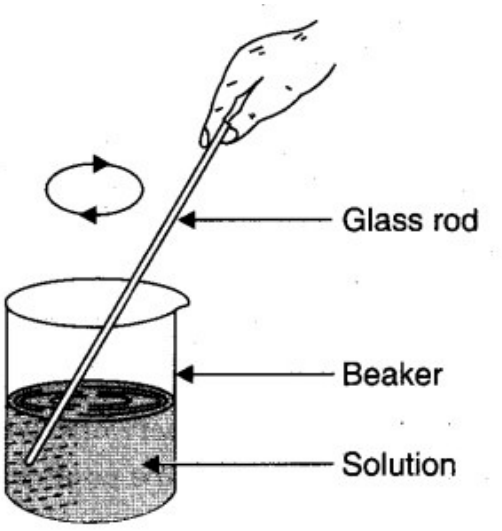

Fig. 5.1. Stirring with a glass rod.

3. The solution in the beaker is heated (60°-70°C) on a wire gauze (Fig. 5.2).
4. Stir the solution continuously and add more of impure substance till no more of it dissolves.

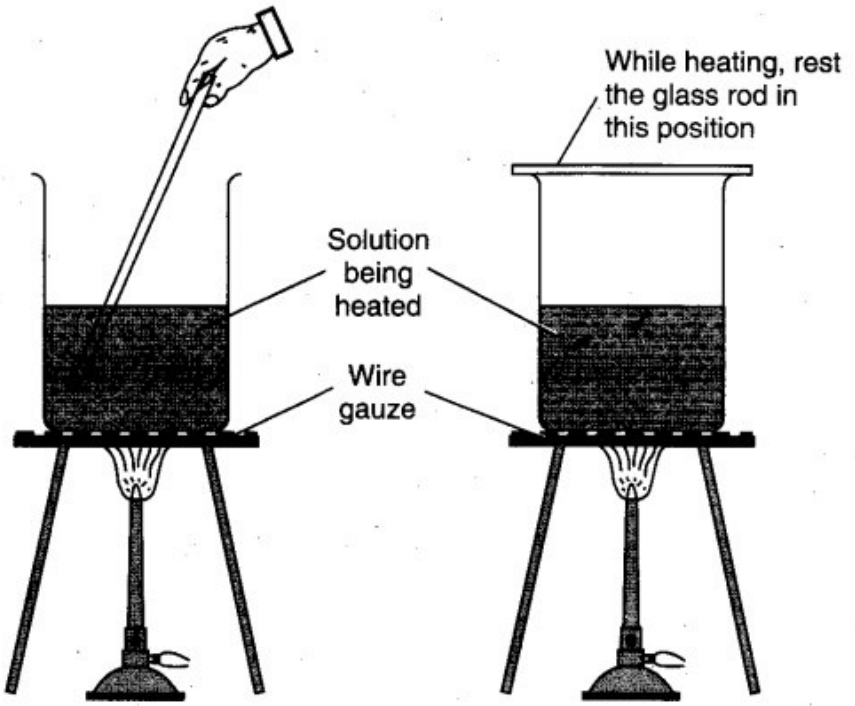

Fig. 5.2. Heating of solution.

2. Filtration of Hot Solution

1. Take a circular filter paper. First fold it one-half, then fold it one-fourth as shown in Fig. 5.3. Open the filter paper, three folds on one side and one fold on the other side to get a cone (Fig. 5.3).

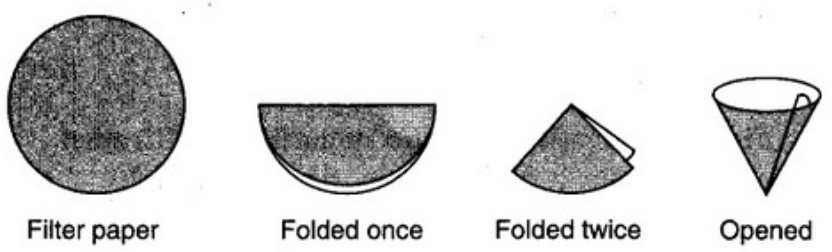

Fig. 5.3. Making a cone.

2. Take a funnel and fit the filter paper cone into the funnel so that the upper half of the cone fits well into the funnel but lower part remains slightly away from the funnel.

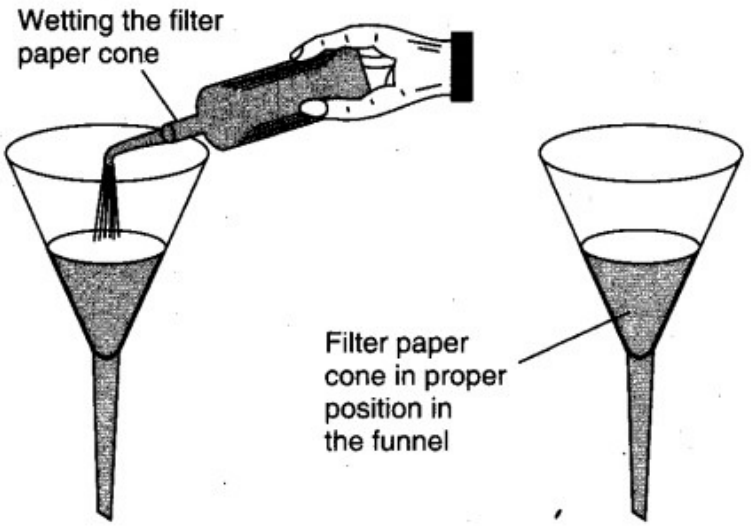

Fig. 5.4. Fitting and wetting of filter paper cone.

3. Wet the filter paper cone with a spray of water from a wash bottle pressing the upper part of the filter paper cone gently against the wall of the funnel with the thumb (Fig. 5.4).
4. Place the funnel on a funnel stand and place a clean china dish below the funnel for the collection of the filtrate. To avoid splashing of the filtrate, adjust the funnel so that its stem touches the wall of the dish.
5. Hold a glass rod in slanting position in your hand or with a precaution that the lower end of the rod should reach into the filter paper cone but it does not touch it. Pour the solution along the glass rod as shown in Fig. 5.5. The filtrate passes through the filter paper and is collected into the china dish placed below. The insoluble impuri¬ties are left behind on the filter paper.

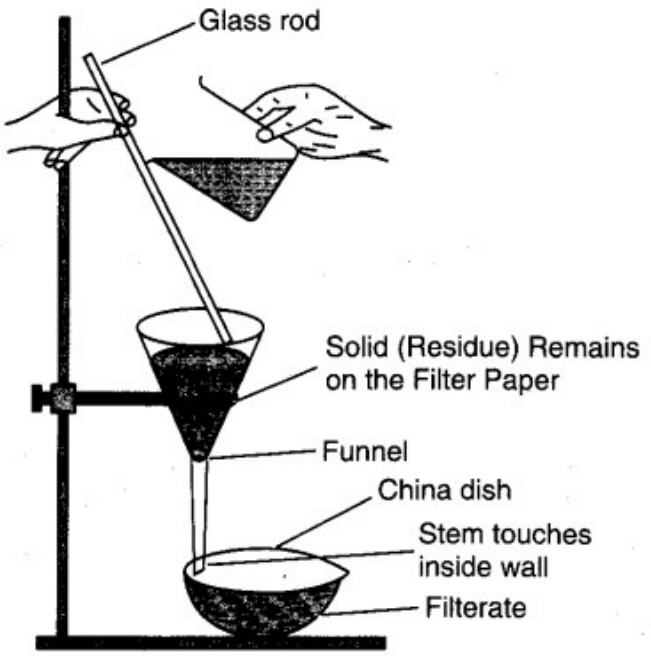

Fig. 5.5. Removing insoluble impurities by filtration.

3. Concentration of Filtrate

1. Place the dish containing the clear filtrate over wire gauze, kept over a tripod stand and heat it gently (Do not boil). Stir the solution with a glass rod (Fig. 5.6). This is done to ensure uniform evaporation and to prevent formation of solid crust.

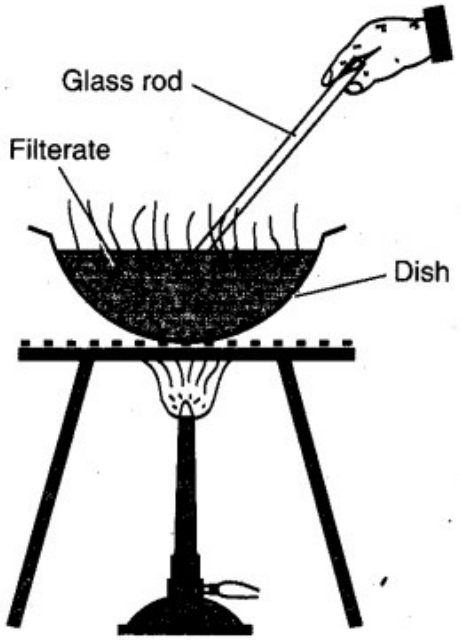

Fig. 5.6. Evaporation of solution.

2. When the volume of the solution is reduced to one-half, take out a drop of the concentrated solution on one end of glass rod and cool it by blowing air (Fig. 5.7). Formation of thin crust indicates that **crystallisation point** has reached.
3. Stop heating by removing the burner.

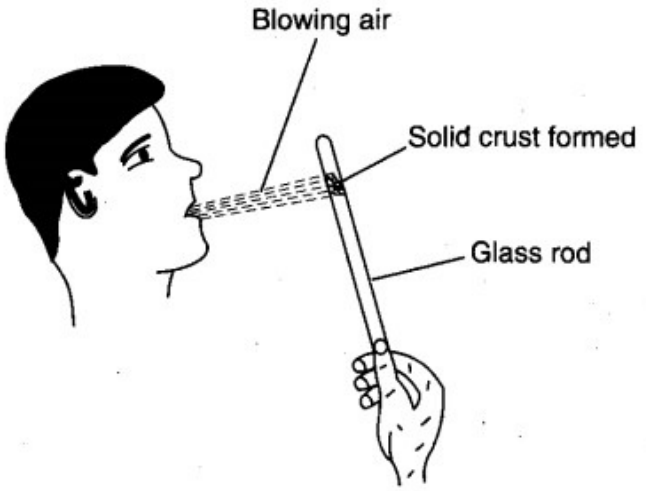

Fig. 5.7. Checking the crystallisation point.

4. Cooling the Concentrated Solution

1. Pour the concentrated solution into a crystallising dish. (It is a thin walled shallow glass dish with a flat bottom and vertical sides. It has a spout to pour off the mother liquor).
2. Cover the dish with a watch glass and keep it undisturbed.
3. As the solution cools, crystals separate out. The concentrated solution is cooled slowly for better yield of the crystals.

Sometimes the china dish containing the concentrated solution is cooled by placing on a

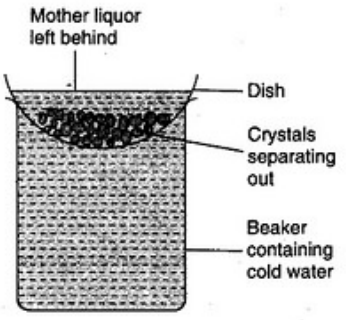

Fig. 5.8. Cooling the solution.

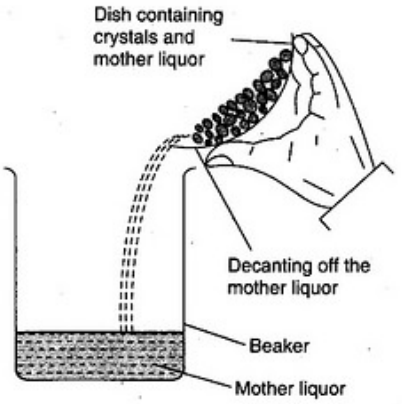

Fig. 5.9. Decanting off the mother liquor.

5. Separation and Drying of Crystals

1. Decant off the mother liquor Fig. 5.9, and wash the crystals with cold water or alcohol.
2. Dry the crystals by pressing them gently between the sheets of filter paper Fig. 5.10. The crystals can be dried by spreading them on a porous plate for sometime or by placing the crystals in vacuum desiccator.

Crystals have definite geometry and a definite shape. Fig. 5.11 shows some of these shapes. Copper sulphate crystals are formed in triclinic shape, potash alum comes out in octahedral geometry. Potassium nitrate crystals are needle like and ferrous sulphate have monoclinic shape.

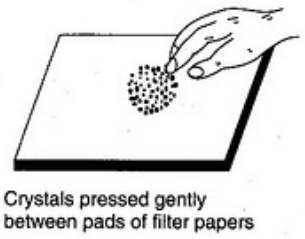

Fig. 5.10. Pressing the crystals.

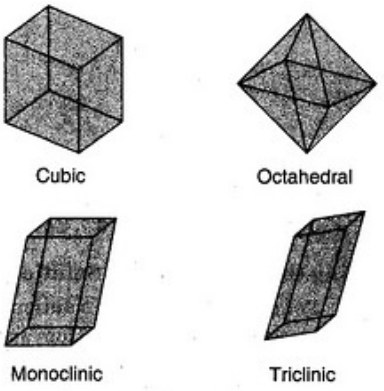

Fig. 5.11. Shapes of crystals.