Preparation of Inorganic Compounds

A double salt is a substance obtained by the combination of two different salts which crystallise together as a single substance but ionize as two distinct salts when dissolved in water. The constituent salts are always taken in some definite molecular proportions. Alums are common examples of double salts.

Alums *are double sulphates having general formula,*

$$X_2SO_4.M_2(SO_4)_3.24H_2O$$

where, X = monovalent cation such as Na^+, K^+, NH_4^+ etc.

M = trivalent cation such as Al^{3+}, Cr^{3+}, Fe^{3+} etc.

Some important alums and their names are given below :

Potash Alum	:	$K_2SO_4.Al_2(SO_4)_3.24H_2O$
Chrome Alum	:	$K_2SO_4.Cr_2(SO_4)_3.24H_2O$
Soda Alum	:	$Na_2SO_4.Al_2(SO_4)_3.24H_2O$
Ferric Alum	:	$(NH_4)_2SO_4.Fe_2(SO_4)_3.24H_2O$

Alums are isomorphous crystalline solids which are soluble in water. Due to hydrolysis, their aqueous solutions have acidic character.

Another example of double salts is Mohr's salt. Its formula is $FeS04.(NH_4)2S0_4.6H_20$. It is used as primary standard in volumetric analysis. Its crystals do not lose water of crystalisation by efflorescence nor is it oxidised in air. It is stable salt unlike green vitriol ($FeS0_4.7H_20$) which gets oxidised by air.

Before we discuss preparation of some of these double salts, let us first review the process of crystallisation.

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 a glass rod giving circular motion as shown in Fig.
3. The solution in the beaker is heated (60°-70°C) on a wire gauze (Fig).

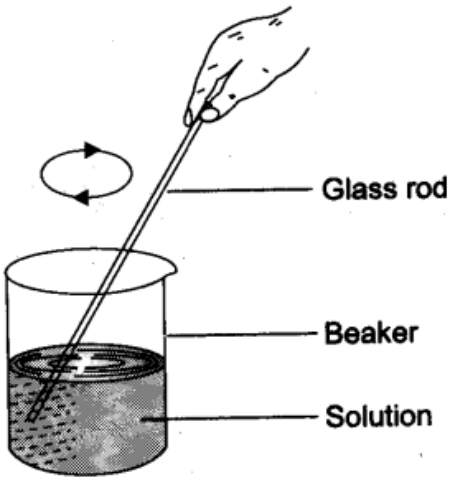

Fig. Stirring with a glass rod.

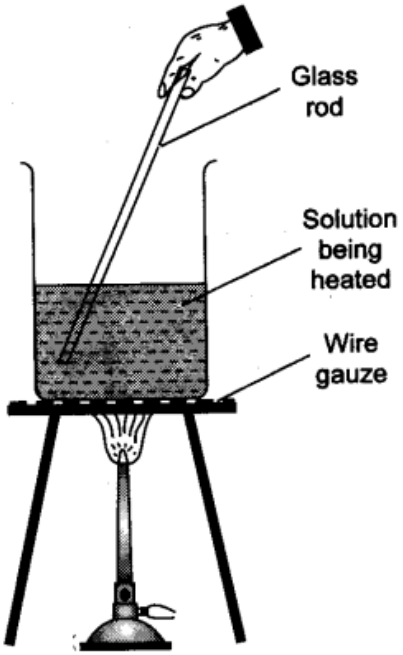

Fig. Heating of solution.

4. Stir the solution continuously and add more of impure substance till no more of it dissolves.

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. Open the filter paper, three folds on one side and one fold on the other side to get a cone (Fig).

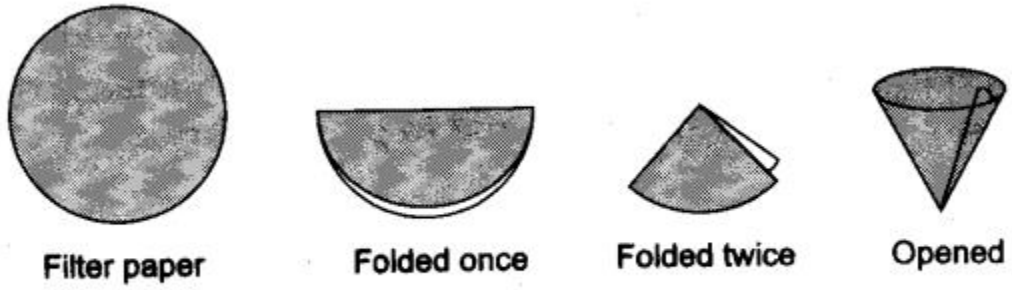

Fig. Making a cone.

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.
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).

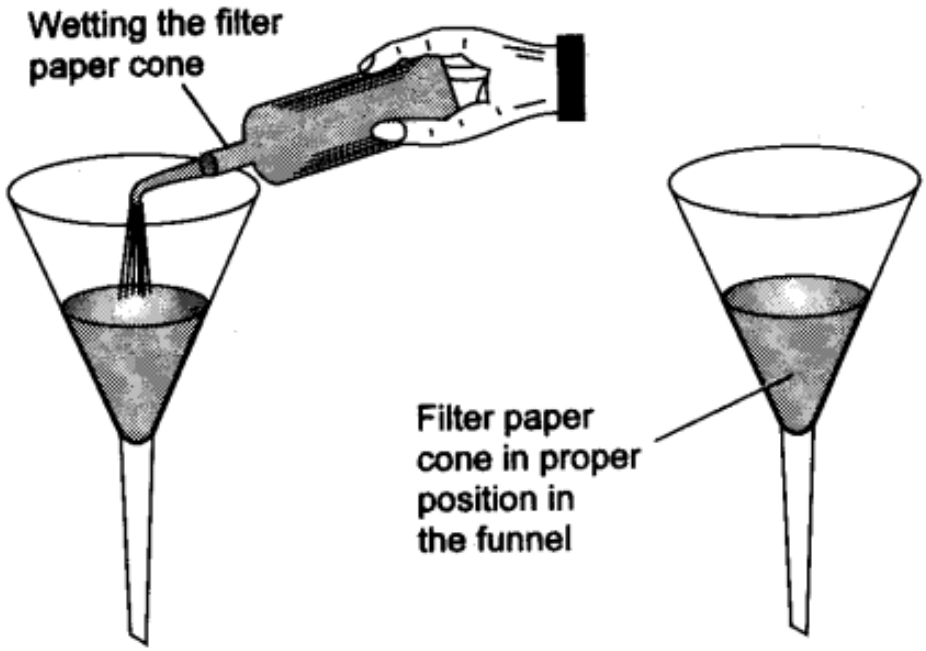

Fig. Fitting and wetting of filter paper cone.

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. The filtrate passes through the filter paper and is collected into the china dish placed below. The insoluble impurities are left behind on the filter paper.

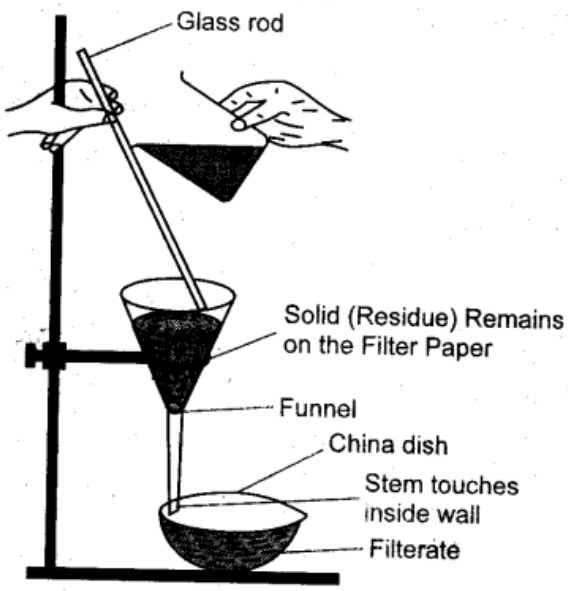

Fig. Removing insoluble impurities by filtration.

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). This is done to ensure uniform evaporation and to prevent formation of solid crust.

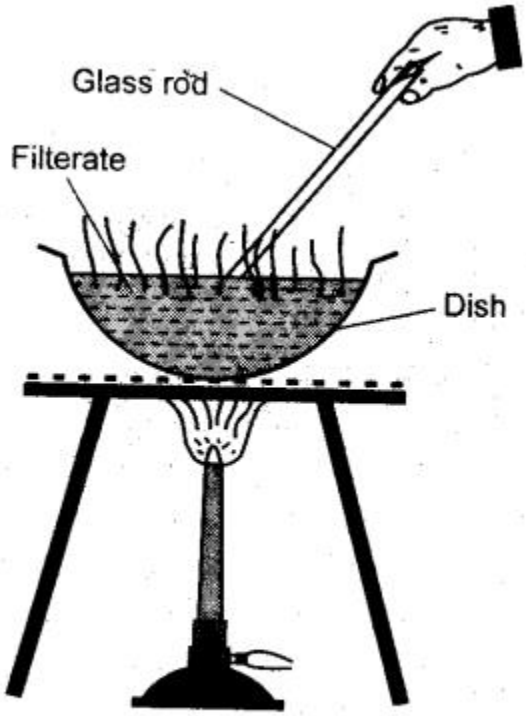

Fig. Evaporation of solution.

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). Formation of thin crust indicates that the crystallisation point has reached.

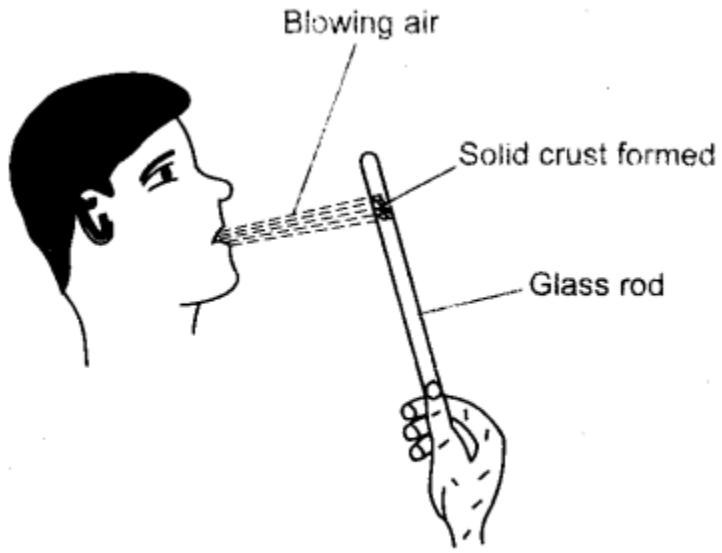

Fig. Checking the crystallisation point.

3. Stop heating by removing the burner.

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 (Fig).

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 beaker filled to the brim with cold water. Cooling may also be done by keeping the china dish in open air depending upon the weather conditions.

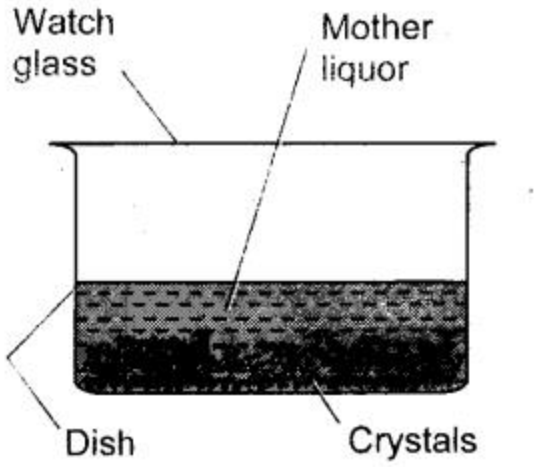

Fig. Cooling in crystallising dish.

5. Separation and Drying of Crystals

1. Decant off the mother liquor and wash the crystals with a thin stream of cold water with the help of a wash bottle.

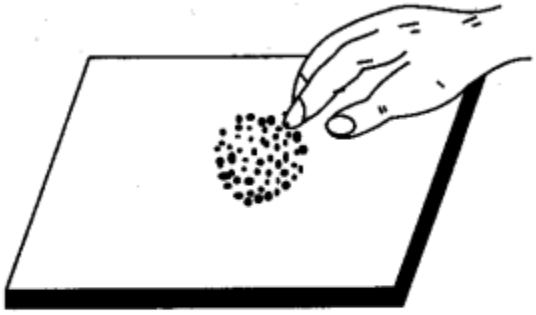

Fig. Pressing the crystals.

2. Dry the crystals by pressing them gently between the sheets of filter paper Fig. The crystals can be dried by spreading them on a porous plate for some time or by placing the crystals in vacuum desiccator.

Crystals have definite geometry and therefore a definite shape. Figure 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.

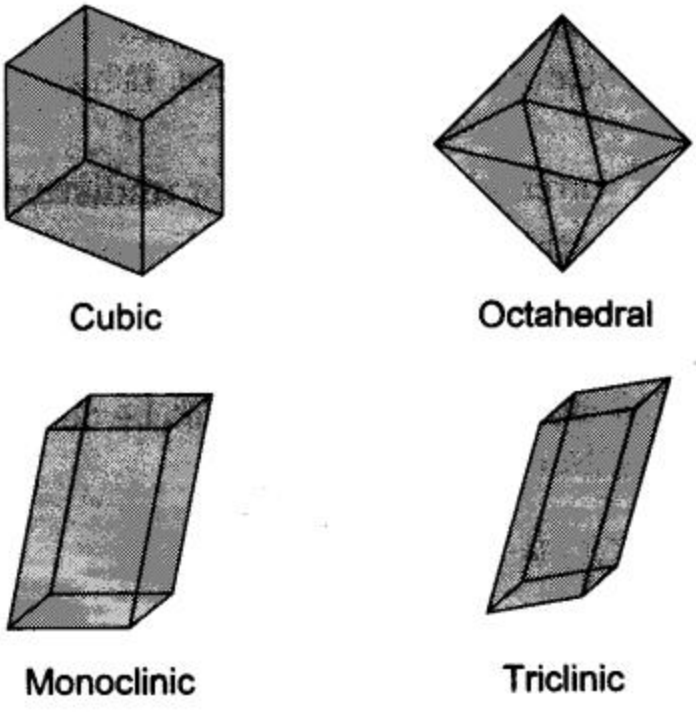

Fig. Shapes of crystals.

Shapes of crystals of some common substances are given in Table

Table Shapes of Crystals of Some Common Substances

Substance	*Geometry or shape of crystal*
1. Blue vitriol, $CuSO_4.5H_2O$	Triclinic
2. Green vitriol, $FeSO_4.7H_2O$	Monoclinic
3. Potassium nitrate, KNO_3	Rhombic (Needle-like)
4. Potash alum, K_2SO_4, $Al_2(SO_4)_3$,$24H_2O$	Octahedral
5. Sodium chloride	Cubic
6. Washing soda, $Na_2CO_3.10H_2O$	Monoclinic