EXPERIMENT-2

PURIFICATION OF SAMPLE OF A COMPOUND BY CRYSTALLISATION:

AIM:

Purification of sample of any one of the following Potash alum, Copper sulphate or Benzoic acid by crystallisation.

THEORY:

Crystallisation is one of the techniques for the purification of an impure compound particularly when the original crude material obtained after a reaction is in a very impure condition. First step of the process involves choosing a single solvent or a mixture of solvents, which dissolves the crude material readily when hot, but only to a small extent when cold. The crude substance is then dissolved in the minimum amount of boiling solvent to obtain a saturated solution. Insoluble impurities are removed by filtering the hot solution. It is then checked for crystallisation point and then cooled slowly when the solute crystallises out leaving the greater part of impurities in the solution. The crop of crystals is collected by filtration and the process is repeated until the crystals of pure substance are obtained. Sometimes during cooling minute quantity of the substance (solid which is being purified) is added to the solution to facilitate the initial crystallisation. This is called seeding. The added tiny crystal acts as a 'nucleus' for the growth of new crystals. Growth of crystals depends upon the conditions in which crystallisation is carried out. For obtaining good crystals, rapid cooling should be avoided because it results into small or disfigured crystals.

Purity of crystals is often judged from the colour of the crystals. For example, pure crystals of alum, copper sulphate and benzoic acid are white, blue and greenish white respectively. Impurities impart colour to the crystals; therefore, impure crystals have a colour different from pure crystals.

MATERIAL REQUIRED:

- Beaker (250 mL) : One
- Glass funnel : One
- Tripod stand : One
- Porcelain dish : One
- Glass rod : One
- Sand bath : One

- Potash alum, Copper sulphate and Benzoic acid : As per need

PROCEDURE:

(i) Take 30-50 mL distilled water in a beaker and prepare a saturated solution of potash alum/copper sulphate in it at room temperature by adding the impure solid sample in small amounts with stirring. Stop adding the solid when it does not dissolve further. To prepare saturated solution of benzoic acid use hot water.

(ii) Filter the saturated solution so prepared and transfer the filtrate into a porcelain dish. Heat it on a sand bath till nearly $\frac{3}{4}^{th}$ of the solvent is evaporated. Dip a glass rod into the solution, take it out and dry it by blowing air from the mouth. If a solid film deposits on the rod, stop heating.

(iii) Cover the porcelain dish with a watch glass and keep the content of the dish undisturbed for cooling.

(iv) When crystals are formed, remove the mother liquor (liquid left after crystallisation) by decantation.

(v) Wash the crystals of potash alum and copper sulphate, thus obtained first with very small quantity of alcohol containing small amount of cold water to remove the adhering mother liquor and then with alcohol to remove moisture. Wash the crystals of benzoic acid with cold water. Benzoic acid is soluble in alcohol. Do not use alcohol to wash its crystals.

(vi) Dry the crystals between the folds of a filter paper.

(vii) Store the dry crystals thus obtained at a safe and dry place.

(viii) Repeat steps (ii-vii) for obtaining maximum amount of pure substance.

PRECAUTIONS:

(a) Do not evaporate the entire solvent while concentrating the solution.

(b) Do not disturb the solution while it is being cooled.

(c) Use the washing liquid in 3-4 very small installments rather than in one installment.