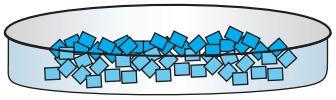
UNIT-3

PURIFICATION AND CRITERIA OF PURITY



FOR the identification of a compound, qualitative analysis of pure substance is required. Therefore, first we have to purify the substance and then check its purity. There are many techniques namely, crystallisation, distillation sublimation, chromatography etc. available for purification of a compound. In this unit you will learn about crystallisation as a technique for purification of a compound. The purity of a compound may be checked by determining its melting or boiling point. The technique for determination of melting and boiling points will also be described in this unit. Pure solid and liquid compounds possess sharp melting and boiling points. Therefore, melting and boiling points of a compound can be used as a criteria of purity.

EXPERIMENT 3.1

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

Copper sulphate 

(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 ¾ 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.

Discussion Questions

(i) Which one of the following formula is correct representation of potash alum(phitkari)? Explain.

(a) $K^{+}(H_2O)_6Al^{3+}(H_2O)_6(SO_4^{2-})_2$

(b) $K_2SO_4.Al_2(SO_4)_3 . 24H_2O$

(ii) What are isomorphous compounds?

(iii) What is meant by the term, 'water of crystallisation'?

(iv) Describe the effect of strong heating on each type of crystal prepared by you.

(v) What do you understand by the term mother liquor?

(vi) Which thermodynamic function favours the process of crystallisation?

(vii) Explain the term-saturated solution?

(viii) Why is the preparation of a saturated solution essential for making crystals?

(ix) Name the processes involved in crystallisation?

(x) What is Kipp's waste? How can we obtain crystals of ferrous sulphate from Kipp's waste?

EXPERIMENT 3.2

Aim

Determination of melting point of a solid organic compound.

Theory

The kinetic energy of molecules of a substance increases on heating. When it becomes high enough to overcome the attractive forces operating between the molecules, the lattice structure of the solid breaks, the solid melts and comes into the liquid state. Melting point of a substance is the temperature at which solid state of a substance begins to change into the liquid state, when the pressure is one atmosphere.

Material Required

- Thiele's tube /Kjeldhal's flask/beaker : One
- Thermometer : One
- Capillary tubes : As per need
- Iron stand with clamps : One

- Liquid paraffin /Conc. H_2SO_4 : As per need
- Organic Compound (Naphthalene/ *p*-Dichlorobenzene/ *p*-Toluidine) : As per need

Procedure

(i) Take a capillary tube of approximately 8 cm in length. Seal its one open end by heating it in a Bunsen flame. Rotate the capillary while sealing to ensure complete closure of the opening.

(ii) Crush the desired substance (about 100 mg) into fine particles and fill the substance in the capillary tube up to nearly 1cm length. For filling the capillary, dip its open end in to the powder. Hold the sealed end between the index finger and the thumb and tap the upper end gently with the other hand so that solid particles are tightly packed and capillary is prevented from breaking.

(iii) Moisten the capillary tube with liquid paraffin and stick it to the thermometer. It will stick to the thermometer by cohesive forces. See that the lower ends of the capillary tube and the thermometer bulb are at the same level. The thermometer is fitted into a rubber cork, which has a groove on its side for the escape of air and vapours.

(iv) Take a Thiele's tube (Fig. 3.1 a) and fill it with 50 to 60 mL liquid paraffin so that it crosses the bent portion of the Thiele's tube. Alternatively, Kjeldahl flask's may be used in place of Thiele's tube.

(v) Dip the thermometer along with the capillary tube in liquid paraffin and adjust the rubber cork in such a way that the thermometer bulb and the filled portion of the capillary is completely dipped in the liquid paraffin and the open end of the capillary remains in the air as shown in Fig. 3.1 a. The thermometer and the capillary tube should not touch the sides of the Thiele's tube.

(vi) Now start heating the side arm of the Thiele's tube with a low flame from the side opposite to that of the capillary tube and note the temperature when the solid starts melting.

p-Dichlorobenzene 

p-Toluidine 

Naphthalene 

Hazard Warning

- *Avoid contact with skin and eyes and don't inhale vapours of these chemicals.*

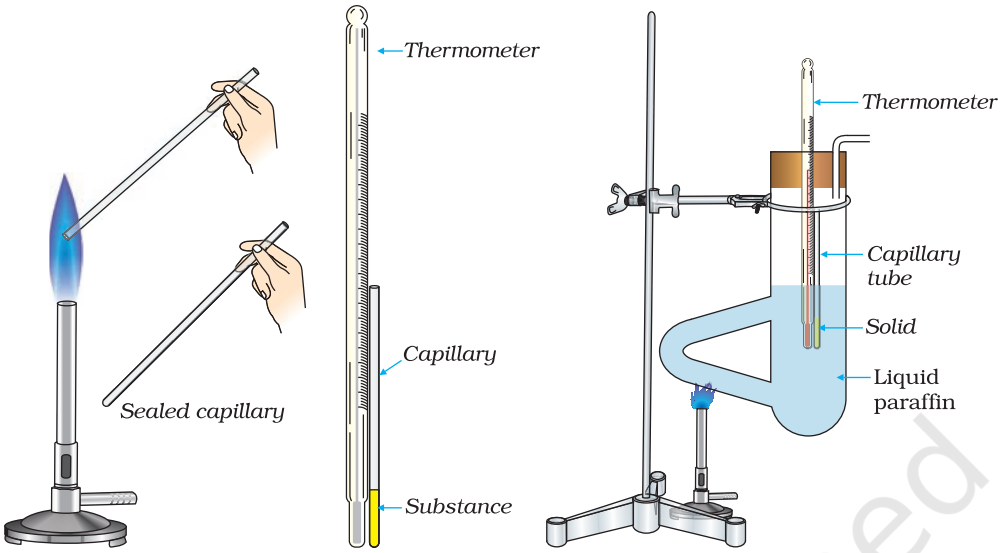


Fig. 3.1 : *(a) Determination of melting point using Thiele's tube*

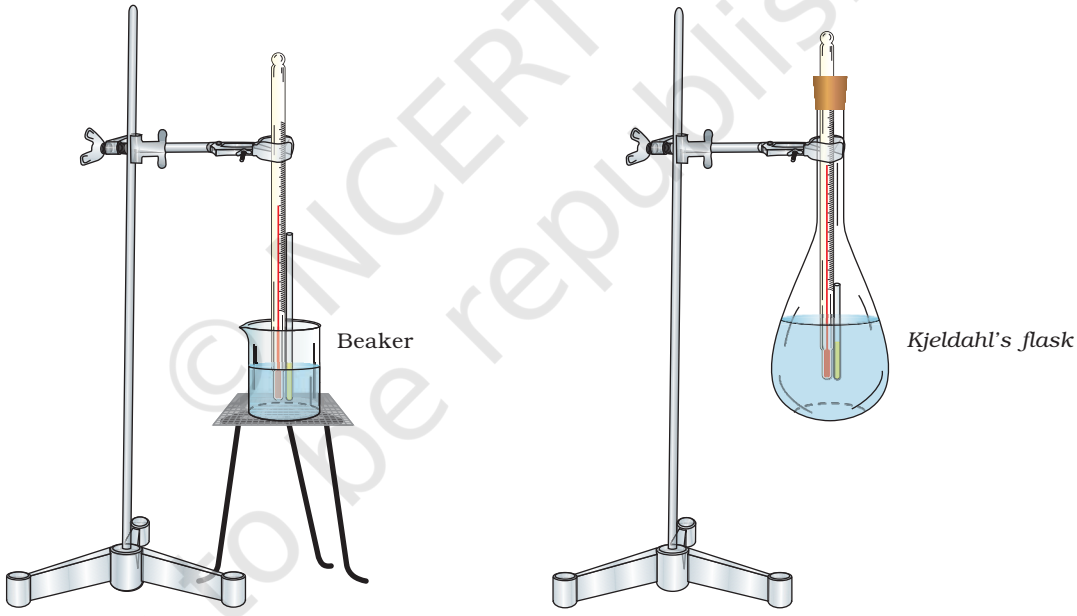


Fig. 3.1 : *(b) Different apparatus used for determining melting point*

This temperature is the melting point of the solid. If you have taken Kjeldahl flask, heat it by revolving the flame around the bottom of the flask to ensure uniform heating. For this, hold the burner in your hand and also keep a sand bath below the flask while heating. It will prevent spilling of acid in case of accident. Repeat the experiment with other solids.

Precautions

(a) Keep the lower end of the capillary tube and the thermometer at the same level.
(b) Capillary tube should not be very thick.
(c) Packing of the powder should be uniform without any big air gaps in between the solid particles.
(d) Thiele's tube should be heated at the side arm by using a low flame.
(e) The cork of the Thiele's tube or Kjeldhal flask holding the thermometer should have a side groove so that vapours can escape through it during the process of heating to prevent bursting of the tube or flask.
(f) Never fill the bulb of Kjeldahl flask's more than half.

Note : *Paraffin can be safely heated upto 220°C. Therefore for determination of melting point of a substance possessing melting point higher than this, conc. H_2SO_4 may be used which can be heated upto 280°C. Sulphuric acid has been suggested for use but is not recomended. Silicone oils are most satisfactory liquids and can be used in place of sulphuric acid.*

Discussion Questions

(i) Why do pure solids possess sharp melting point?
(ii) What is the effect of impurities on the melting point of a solid?
(iii) Why is the melting point of benzamide more than acetamide?
(iv) Can any other liquid be used in place of liquid paraffin to determine the melting point?
(v) Can we heat the capillary directly for the determination of melting point?
(vi) Why is liquid paraffin filled in the Thiele's tube/Kjeldahl's flask?
(vii) Why is Thiele's tube heated at the side arm?

Experiment 3.3

Aim

Determination of boiling point of a liquid organic compound.

Theory

The boiling point of a liquid is the temperature at which vapour pressure of the liquid becomes equal to the atmospheric pressure, which the surface of the liquid experiences. At 1.013 bar atmospheric pressure the boiling point of the liquid is termed as normal boiling point. Different liquids have different boiling point. The difference in the boiling points of liquids is essentially due to the difference in the intermolecular forces operating between the molecules of the liquid.

Material Required

- Thiele's tube/Kjeldahl's flask : One
- Thermometer 110°C or 360°C : One
- Iron stand with clamp : One
- Ignition tube : One
- Capillary tube : One

- Organic liquid : 1 mL
- Liquid paraffin/ Conc. H_2SO_4 : As per need

Concentrated H_2SO_4 

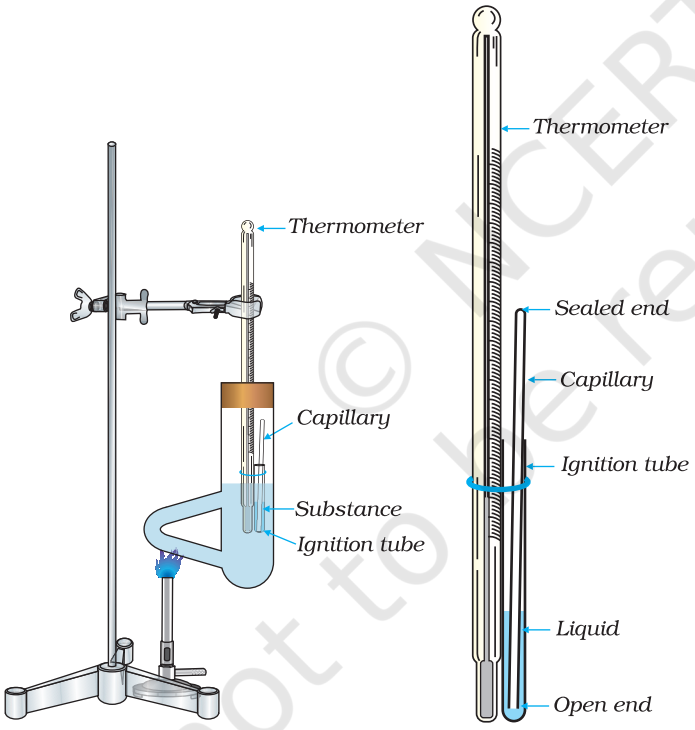


Fig. 3.2: *Determination of boiling point*

Procedure

(i) Fill Thiele's tube with the liquid paraffin so that it crosses the bent portion of the Thiele's tube.

(ii) Take 1-2 drops of the given liquid in an ignition tube and tie the ignition tube with the thermometer with a rubber band as shown in Fig. 3.2. Note that the lower end of the ignition tube and the thermometer bulb are at the same level.

(iii) Seal one end of the capillary tube of approximately 8 cm length by heating in the flame.

(iv) Place the capillary tube with its open end dipped in the liquid present in the ignition tube.

(v) Heat the side arm of Thiele's tube with a low flame.

(vi) Observe the escape of bubbles at the lower end of the capillary dipped in the liquid organic compound. Note the temperature at which bubbles start coming briskly and continuously. This temperature is the boiling point of the liquid.

Note : *For determination of boiling point of high boiling liquids, paraffin cannot be used as heating medium.*

Precautions

(a) Record the temperature as the boiling point at which brisk and continuous evolution of the bubbles starts from the lower end of the capillary dipped in the liquid organic compound.

(b) Keep the lower end of the ignition tube and the thermometer bulb at the same level.

(c) Heat the side arm of the Thiele's tube gently.

(d) Boiling point of the liquid filled in Thiele's tube should be 50-60°C higher than that of the liquid, of which boiling point is to be determined.

Discussion Questions

(i) Suggest a suitable liquid, which can be filled in the Thiele's tube for the determination of the boiling point of carbon tetrachloride?

(ii) In place of liquid paraffin, can any other liquid be used for the purpose of determination of boiling point?

(iii) Suppose boiling point of a liquid in Delhi is 100°C. At hill station, will it be the same or different? Give reasons.

(iv) Why is food cooked more quickly in a pressure cooker?

(v) How would the boiling point of water vary with the addition of equimolar quantities of urea, potassium chloride and potassium sulphate?

(vi) Why do different isomers of alcohol represented by the formula $C_4H_{10}O$ differ in their boiling points?

Do you know?

Learning technique of crystallisation is not only important from the point of view of purification of compounds but also from the point of developing large single crystals; because studies on single crystals have shown them to have many optical and electrical properties of great use. For example, slices from large crystals of silicon containing traces of certain impurities are used in making solar batteries used for the operation of instruments in satellites. Frequency control in radar, television and radio is done by making use of slices of some crystals. Also, use of crystals of some compounds is made in microphones and earphones. You can now realize how important it is for chemist to learn these techniques.