EXPERIMENT-3

DETERMINATION OF MELTING POINT OF AN ORGANIC COMPOUND:

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 ther mometer 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.

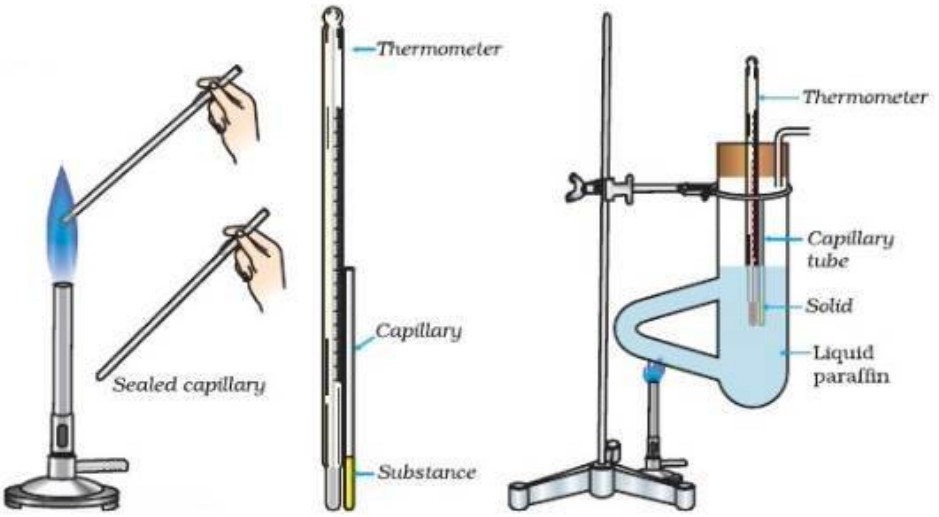

Fig. 3.1 : (a) Determination of melting point using Thiele's tube

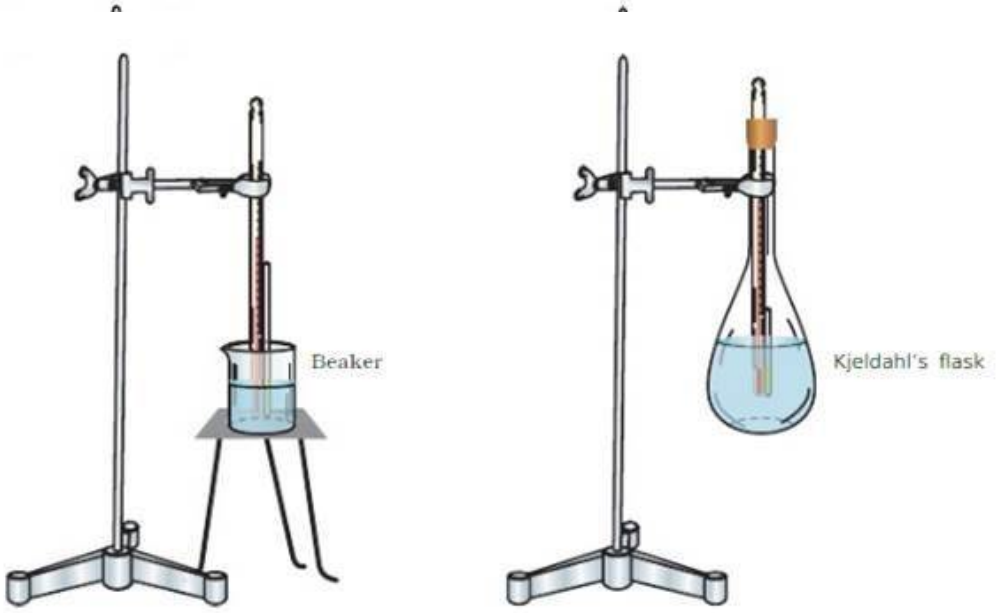

Fig. 3.1 : (b) Different apparatus used for determining melting point

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.