Melting Point

M.P of an organic Compound defined as a temperature at which the Solid exists in equilibrium with its liquid under an external Pressure

The melting Point of a Solid is

On heating a Solid its molecule absorbs energy in the form of heat. As a result the force of attraction between the molecule decreases and the molecules become increasingly Separated. At a Particular temperature called the melting Point of the Solid, the Separation of the molecules increases by the large amount and the Solid melts and is Converted into liquid.

Our aim is to determine the melting Point of the given Compound urea.

Materials Required -

Liquid Paraffin in a 100 ml beaker
Powdered urea
Thin walled tube of approximately 8-10 cm length and 2 mm diameter. thread
Glass Plate, Thermometer

Stand with clamp
Stirrer, Spatula
Hot Plate.

Procedure → Take a Capillary tube and Seal one end by heating it in the flame of the Bunsen Burner. Now using the Spatula, make a heap of Powdered urea on the glass Plate. Push the open end of the Capillary tube into the heap. Some Substance will enter into the Capillary tube. Now tap the sealed end of the Capillary tube on the glass Plate gently and fill the Capillary tube up to 2.3 mm. Now attach the Capillary tube to the thermometer using the thread. Take the 100 ml beaker or Kjeldal Containing liquid Paraffin and Place it over the hot plate.

Clamp the thermometer carrying the test tube to the iron Stand and immerse them in the bath of liquid Paraffin. Start heating the liquid Paraffin bath

Slowly and Stir the bath gently using the Stirrer to ensure uniform heating. Note the temperature t1 when the urea starts melting, continue heating and note the temprature t2 when the urea in the Capillary tube is Completely melted. The average of the temperature t1 and t2 gives the M.P. of urea

Literature M.P of urea - 132°c

Precalitions

use dry and Powdered Sample for the determination of M.P.

Packing of the Powder Shold uniform without any big air gaps between the Solid Particles

The liquid paraffin bath must be heated very Slowly and the bath is stirred gently to ensure uniform heating

The Procedure will be Same for the determination of oxalic acid.

Literature value of oxalic acid M.P.

MP = 101°C

5.1.7 Determination of melting point and boiling point

Melting point

Determination of the melting point is extremely valuable for identification of an organic compound. Melting point of a solid is the temperature at which it begins to change into a liquid; the change from solid to the liquid state being quite sharp (within 0.5°).The melting point is considerably influenced by the presence of other substances (impurities) and is therefore an important criteria of purity.

The correct melting point can be determined by using a calibrated thermometer. The calibration is checked by determining the melting or boiling points of a few selected organic compounds such as *p*-dichlorobenzene (53°), benzoic acid (122°), salicylic acid (159°) and anisic acid (184°) as reference compounds. Acetone (b.p. 56°) and water (b.p. 100°) are also useful reference compounds.

PROCEDURE

Powder the organic compound (100 mg) with the help of a spatula on a clean, dry watch glass. Seal one end of a capillary tube (~3 " long) and fill it (3–5 mm) with the finely powdered sample to form a compact column. Attach the capillary to the thermometer and place it in the kjeldahl flask (100 ml capacity) containing the bath liquid (~50 ml) (concentrated sulphuric acid or paraffin wax) as shown in the diagram (Fig. 5.5(a)). Heat the kjeldahl flask gently at a uniform rate (1–2° rise in temperature per minute). Note the temperature at which the solid melts.

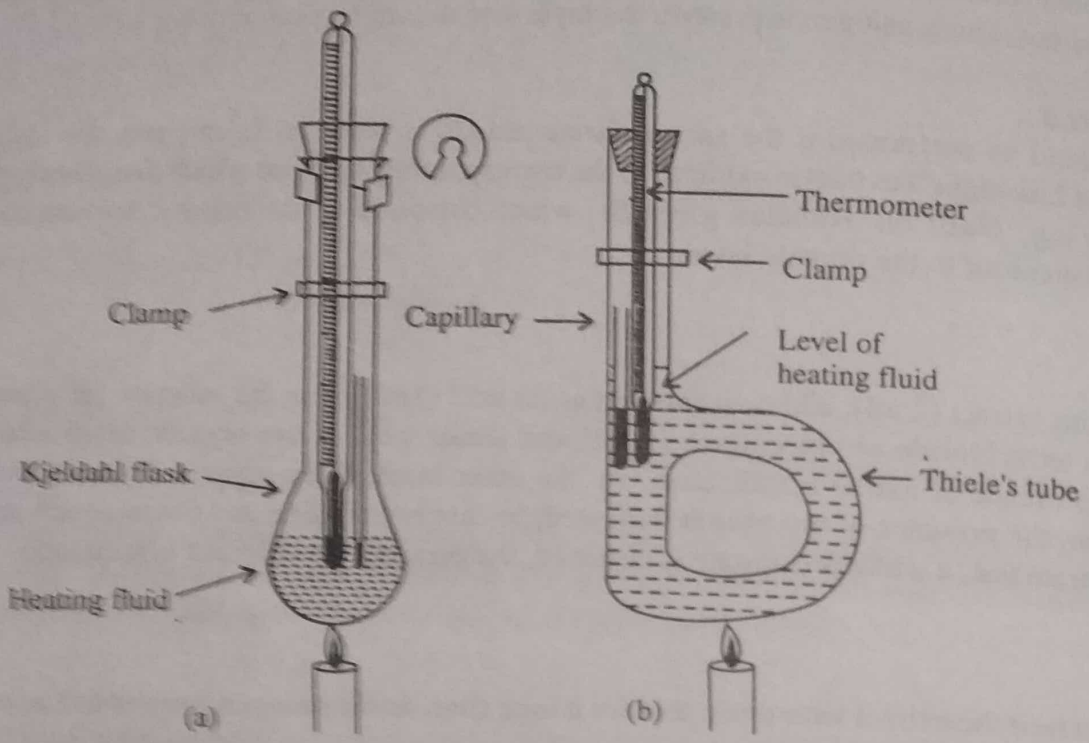

Fig. 5.5 *(a) Kjeldahl flask (b) Thiele's tube*

NOTE

1. It is advisable to determine the approximate melting point first. Repeat to determine the correct melting point. In the second determination, the rate of heating should be carefully controlled (specially near the melting point of the compound).
2. The organic compound should be finely powdered.
3. The compound should form a compact column at the sealed end of the capillary tube. This can be done either by dropping the capillary tube on the working table through a glass tube (18–20" long) or tapping the lower end of the capillary.
4. A Thiele's tube may be used in place of a Kjedahl flask (Fig. 5.5(b)).
5. Many organic compounds undergo a change in crystalline structure just before melting, usually because of the release of the solvent of crystalisation. This may lead to shrinkage of the sample in the capillary tube. This should not be taken as the beginning of the melting process.

Boiling point

Like the melting point of a solid, the boiling point is useful for identification of a liquid organic compound. The boiling point of a liquid (at a given pressure) is defined as the temperature at which its vapour

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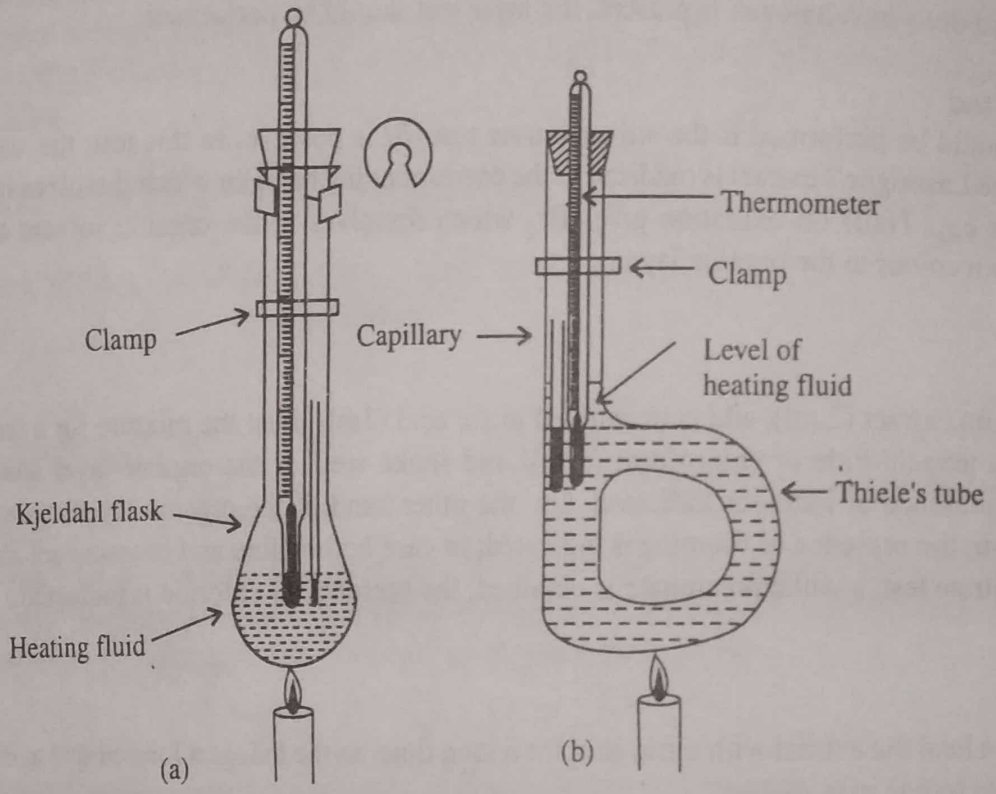

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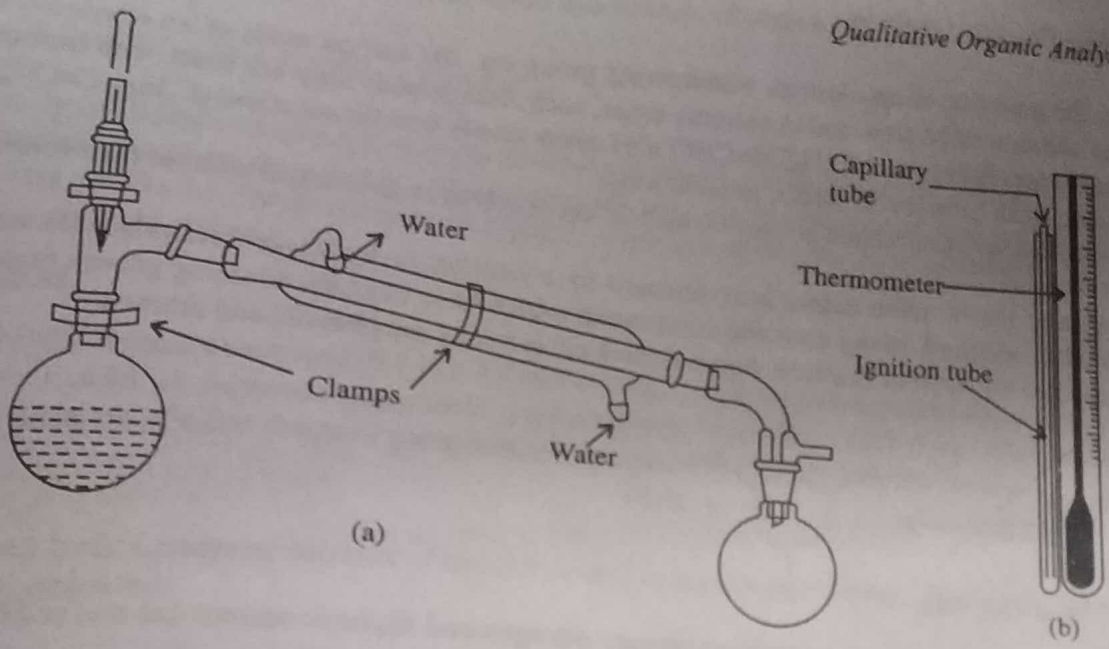

Fig. 5.6

pressure is equal to the atmospheric (surrounding) pressure. Though a pure liquid has a sharp boiling point – the converse is not always true. A sharp boiling point may sometimes be caused by a constant boiling mixture (azeotrope) of two or more liquids.

PROCEDURE

Take the given liquid compound (20–25 ml) in a dry round bottomed flask (100 ml). Add a few pieces of pumice stone and set up the distillation apparatus as shown in Fig.5.6(a).

Heat the liquid gently at a uniform rate. Discard the first fraction of the distillate (2–3 ml). Note the temperature at which the liquid boils (constant temperature) and collect the distillate in a clean dry flask.

In case only a small amount of the liquid is available, determine the boiling point by Siwoloboff's method. Take the organic liquid (0.5–1 ml) in an ignition tube (4–5 mm in diameter and 80–100 mm long). Introduce a capillary tube sealed at the upper end into the liquid (as shown in Fig. 5.6(b)). Attach the ignition tube with the help of a rubber band to the thermometer and fit this into a melting point bath. Heat the bath gently. The temperature at which a rapid and continuous stream of bubbles first emerges from the capillary tube is the boiling point of the liquid. At this temperature, the bubbling ceases and the liquid begins to rise in the capillary.

5.1.8 Detection of unsaturation

Unsaturation in the given compound can be established by the following tests.

(a) *Test with bromine in carbon tetrachloride solution*

Compounds containing a carbon–carbon multiple bond react with bromine.