

Experiment 1:

Melting Point

Purpose:

The purpose of this laboratory exercise is to identify and assess the purity of an unknown solid organic compound by determining its melting point and comparing this number to a list of possible compounds.

Introduction:

The melting point of a solid is the temperature at which transition from solid to liquid occurs at atmospheric pressure; or the temperature at which solid and liquid phases are in equilibrium at a pressure of one atmosphere.

Solid state \longleftrightarrow Liquid state

The melting point is practically unaffected by changes in external pressure, making it a convenient physical constant for the identification of solids. Many organic compounds are solids at room temperature as a result of strong intermolecular forces which hold the individual molecules together in a crystal lattice.

The nature and strength of these intermolecular forces are responsible for the observed differences in melting point. In general, if the forces are strong, the melting point will be high, and if they are relatively weak, the melting point will be low.

A pure solid has a sharp melting point and will melt within a narrow range of (1-2) C°.

Soluble impurities affect the melting point of a solid in the following manner:

a. Lower the melting point of the substance. with the upper limit considerably below the true melting point. The presence of an impurity in the molten compound, reduces its vapor pressure thus lowering the melting point of the compound.

b. Broaden the melting point range. Depending on the amount of impurity, the melting process may extend over a range of 2-20 C° or more.

The lattice energy and consequently Melting point depends on:

- 1- Molecular weight.
- 2- Symmetry of the molecules.
- 3- Polarity of the molecules.
- 4- The type of bonding.

Apparatus:

A simple device for determining melting points is shown in **Figure (1)**. It consists of a thermometer fitted through a cork and suspended into a long-necked flask which is three quarters filled with a high boiling and stable liquid like paraffin oil. The thermometer bulb should be about **(1) cm** above the bottom of the flask. The sample in the capillary tube is fastened to the thermometer with a rubber band placed above the level of the oil. The capillary tube should be close to and on a level with the thermometer bulb.

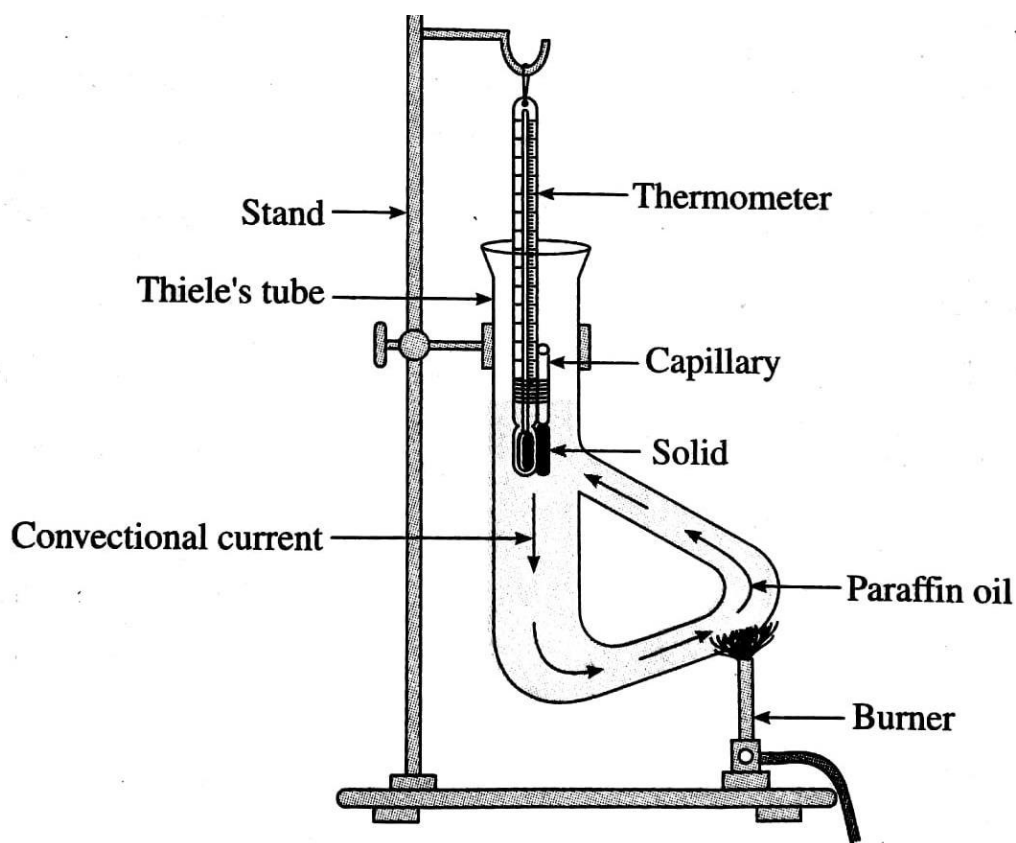


Figure (1) : melting point device

Capillary melting point tubes are about (6-7) cm in length and (1) mm in diameter. They are sealed by rotating one end of the capillary tube in the edge of a small hot flame **Figure (2)**.

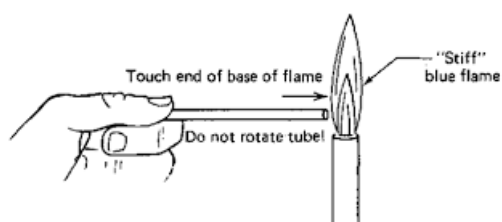


Figure (2): Sealing the end of the capillary tube

The dry solid is ground to a fine powder on a piece of paper with a spatula. The open end of the capillary is then pushed into the powder which is forced down the capillary tube by gently tapping the closed end on the bench top **Figure (3)**. This is repeated several times until the solid is densely packed at the bottom of the tube to a height of 2-3 mm.

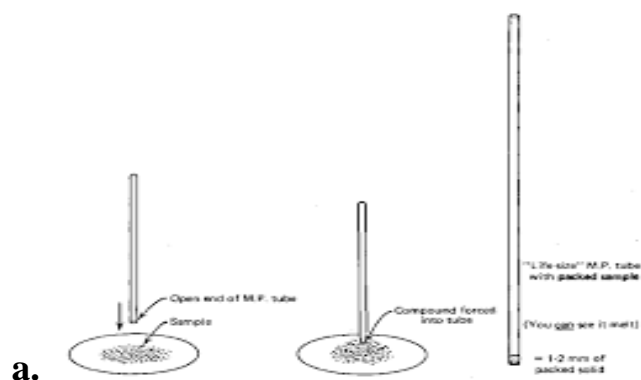


Fig. 3.1. Sealing one end of the capillary tube.

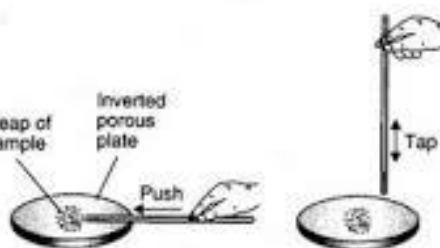


Fig. 3.2. Charging the capillary tube

b.

Figure (3): Filling the capillary tube with the powder of the unknown

Procedure:

To determine the melting point of a solid, a small amount of the powdered substance is introduced into a capillary tube which is then attached to a thermometer and placed in the oil bath.

A. If we have a known organic compound: The bath is heated rapidly to within (20) C° of the expected melting point then slowly, and at a constant rate of (2-3) degrees per minute, close to the melting point. The temperature at which the solid begins to melt, and that at which it is completely liquid, is recorded as the melting point range of that substance.

b. If we have an unknown organic compound:

1. Obtain approximately 0.01 g of the chemical to be tested in a plastic tray. Using a mortar and pestle, grind the sample into a fine powder.

2. It is suggested that a rough and quick trial run is completed to get an approximate melting point for your unknown. Once a rough melting point is determined, allow the machine to cool to at least 15°C below the bottom temperature of the rough melting point.
3. After the rough trial, head the machine very slowly through the melting point. The slower it heats through the melting point, the more accurate the melting point.
4. Record the temperature at which the first liquid droplets appear.
5. Record the temperature at which the last solid particles liquefy.
6. Average the two temperatures to get the experimental melting point of the solid.

$$\frac{t_1 + t_2}{2} = \textit{Melting point}$$

The melting point range is affected by a number of factors in addition to that of purity. Particle size, amount of material used, density of packing in the capillary tube, thickness of the capillary tube and the rate of heating of the liquid bath, are all factors that should be carefully considered to ensure an accurate melting point. The rate of heating is the most critical factor affecting experimental results, and should be carefully monitored, particularly close to the expected melting point.