

Determination of Melting Point

The melting point, m.p of a solid crystalline compound is the temperature at which the solid and the liquid phase of the compound are in equilibrium at a certain pressure, usually 1 atmosphere

Or, it is the temperature at which the solid begins to change into liquid under a pressure of one atmosphere

If the resultant liquid is cooled, solidification will occur at the same temperature, i.e the melting point and the freezing point for a pure substance are identical

The melting point range is the difference between the temperature at which begins to melt (T_1) and the temperature at which the sample is completely melted (T_2)

Melting point range = ($T_2 - T_1$)

Organic chemist use melting point to :

- 1- Get an indication of the purity of the crystalline compound.

pure organic compounds usually have a sharp melting point and melt within a range of about 0.5-1 °C.

while, impure substances have no sharp melting points melts over a range of several degrees.

The same idea is applied for pure organic cpd.s if they undergo slight decomposition before reaching their m.p. The decomposition products act as impurities that decrease the m.p & increase the melting point range.

- 2- Help in identification of organic compounds

sodium chloride (NaCl) has melting point of 801 °C.

Glacial acetic acid (CH_3COOH) has a m.p. of 16.6 °C

Mixed melting point

Suppose you have 2 solid samples (A&B) with the same melting point.

How can you know whether the 2 samples are the same or different ?

Answer :

We can mix them and measure the melting point for the resultant mixture. If A and B are different, one will act as an impurity for the other & the measured m.p. will be lower than the original one with higher m.p range.

General Technique if Melting Point Determination :

The method used for the determination of the melting point is called the capillary tube method

Sample preparation

Any compound being loaded into capillary tube must be

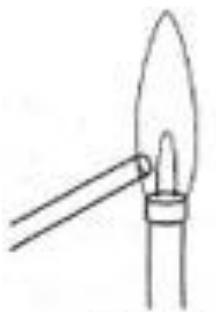
- 1- Fully dry
- 2- Homogenous
- 3- In powdered form

Melting point capillary tubes :

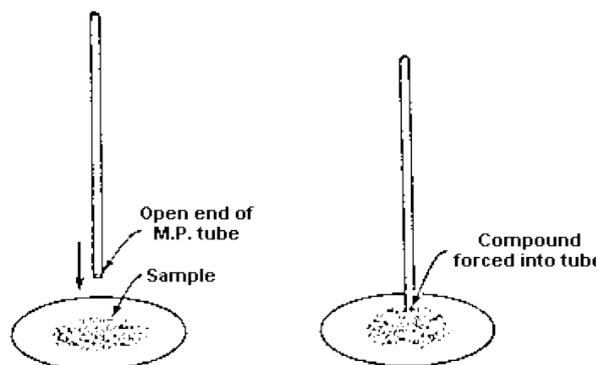
They are thin glass tubes, 1 mm in diameter & are supplied open at both ends or closed at one end or closed at both ends.

Sealing of melting point capillary tubes :

To seal one end of an open capillary tube , just touch the end of the tube into the outer layer of the hot flame of a burner. The end of the tube will collapse in & seal the tube. Make sure that the tube is sealed, i.e. there is not a fine line in the sealed end, & that there is no large globule of glass on the end of the tube



Filling a capillary tube to fill the tube with few milligrams of the solid sample, the open end of the tube is pressed gently & repeatedly into powdered sample several times, the chemical can be moved to the sealed end by turning the tube over end tapping it on the bench. A sample height of 2-3 mm in the capillary tube is recommended for optimum resulted.



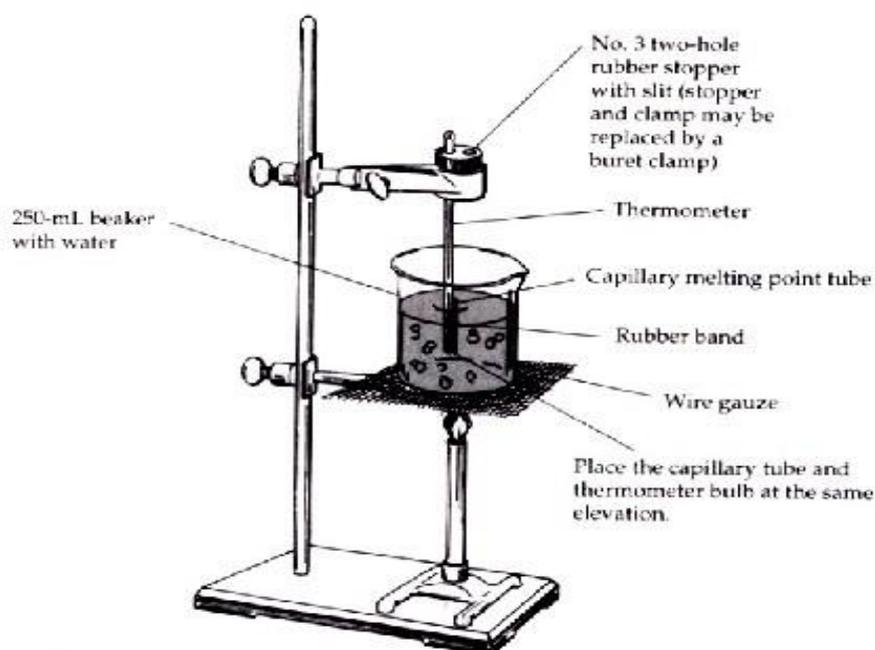
Name of experiment : 1 Determination of melting point of an unknown sample .

Aim of experiment :

- 1- Identification of an unknown compound using its melting point**
- 2- Determination of purity of a compound using m.p. as a physical property**

Procedure

- 1- Preparation of the unknown sample as mentioned before
- 2- The capillary tube is attached to a thermometer by a rubber using in such a way that the closed end is attached to the bottom of thermometer's bulb.
- 3- Then both of them are placed in an oil bath, (the rubber ring should be above the surface of the oil bath).
- 4- Heating is started gradually
- 5- The range between the temperature at which the powdered solid inside the capillary tube begins to liquefy (T_1) and the temperature at which a clear liquid is observed inside the capillary tube (T_2) is recorded as the observed point range.



Apparatus for melting point determination

Question and Discussions :

Q1/ An impure sample melts at a lower temperature and over a wider range, why?

A pure sample melts sharply because the force of attraction between its particles are the same. The presence of a foreign particle (impurity) in a crystal lattice interrupts its uniform structure and the forces of attraction are weakened.

Q2/ In practice, melting point is determined as capillary melting point, why

Determination of m.p is tedious and time consuming. Thus in practice, most melting points are determined as capillary m.p , which can be done quickly with a small amount of sample in a capillary tube.

Q3/ The sample must be in fine powdered form before measuring the melting point, why ?

For efficient and reproducible heat transfer into the sample, so coarse crystalline and non-homogenous samples must be crushed into a fine powder.

Q4/ A sample height of 2-3 mm in capillary tube is recommended, why ?

Since taller samples take extra heat to completely melt & wider range than their shorter counterparts.

