

Determination of Melting points and Boiling points

A. Melting point (M.P.)

Purpose:

The purpose of this experiment is to determine the melting points of organic compounds and check the purity of compound.

Theory:

The **melting point (m.p)**°C of a compound is the temperature at which it changes from a solid to a liquid. Since this requires that the intermolecular forces that hold the solid together have to be overcome. The melting point is a physical property (melting point, boiling point, density, solubility, etc.) often used to identify compounds. Usually, chemists can only obtain a melting range of a 2 - 3°C accuracy.

A pure, non-ionic, crystalline organic compound usually has a sharp and characteristic melting point (usually 0.5-1.0 °C range). A mixture of very small amounts of miscible impurities will produce a depression of the melting point and an increase in the melting point range.

Melting point range: The interval between the temperature at which a solid sample just begins to turn to liquid and the temperature at which the entire sample becomes liquid. or range of temperatures in which the first crystal starts to melt until the temperature at which the last crystal just disappears.

What are the factors that affect the melting point?

1- Molecular weight

Melting points are higher for higher molecular weight compounds. The reason the melting point increases with the weight is that it takes more energy to separate larger molecules from a crystalline structure than it takes to separate smaller ones.

2- The nature of the organic compounds

Ionic compounds, often form crystal lattices in which each ion is surrounded by ions of the opposite charge, because of the strength of this electrostatic attraction,. Breaking up an ionic crystal lattice requires considerable energy as a result, ionic compounds have high melting points, While the **Covalent compounds** consist of molecules rather than ions. Since the attraction between molecules is weaker than is the attraction between ions, covalent compounds usually have a lower melting point than ionic compounds. Moreover, some covalent compounds have polar molecules in which one end is more electronegative than the other, such polar compounds have a higher melting point than nonpolar molecules.

3-Impurities

Impurities decrease the melting point. ice-salt mixtures. contaminants normally lower the melting point and broaden the melting point range Pure samples usually have sharp melting points, for example(149.5-150)°C, impure samples of the same compounds melt at lower temperatures and over a wider range, for example 145-148°C.

4-Shape of Molecules

Molecular shape influences the melting point of a substance. If the shape of the molecules allows them to be packed together in a compact fashion, For example, symmetrical molecules give compound (4- n -propyl benzoic acid) a higher melting point than that of (4- iso -propyl benzoic acid) , the molecules of which do not pack well,

Types of measuring apparatus of melting point:

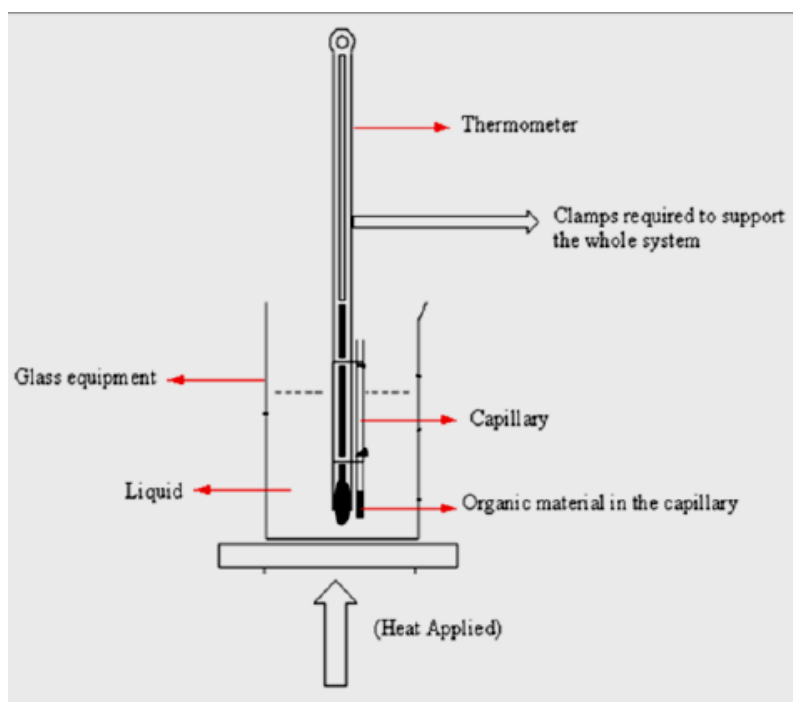


Fig1:Melting point apparats (old fashion model).



Fig 2: Melting point digital apparatus (new fashion model).

Properties of oil bath

- 1) It has a high Boiling point 3000C rather than water (1000C)
- 2) It does not emit toxic gases
- 3) It is transparency and you can see the M.P tube through it. It stays pretty clear on heating.
- 4) It has high density rather than water.
- 5) It does not decompose.

apparatus / Materials:

oil bath(beaker contain oil) capillary tubes.
Thermometer solid organic compounds.
Bunsen burner Rubber bands(ring).

Procedure:

- 1- Take a capillary tube and sealed an open end of capillary tube by inserting the tip into a Bunsen flame near the base of the flame and turning the tube in your fingers using benzen burner.

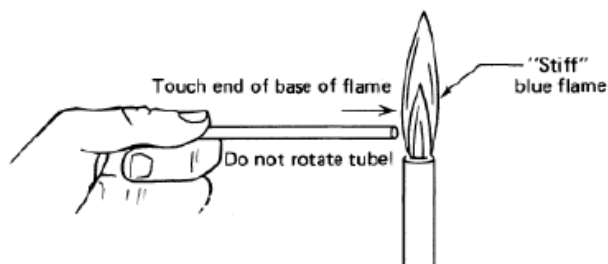


Fig. 3. Closing a M.P. tube by using the flame.

2- Place a small amount of the compound (Benzoic acid) in a clean surface. Push the open end of the capillary tube into the compound as shown in Fig: 3a .

3- Move the powder to the closed end of the capillary tube by tapping it on the tables . Repeat until the compound occupies 1-2 mm of the capillary tube end Fig:3a.

5- Attach the capillary tube to a thermometer using a thread. And align the bulb of the thermometer with the closed end of the capillary tube as illustrated in Fig: 3b.

6- put an oil in a beaker and place it over a piece of wire gauze placed over a tripod stand.

7-. Heat the beaker slowly while constantly stirring the contents using a stirrer *to insure* a uniform temperature throughout. Fig: 3c .

8- Note the temperature at which the compound melts and Record the temperature at which the solid in the capillary tube melts

3a 3b 3c

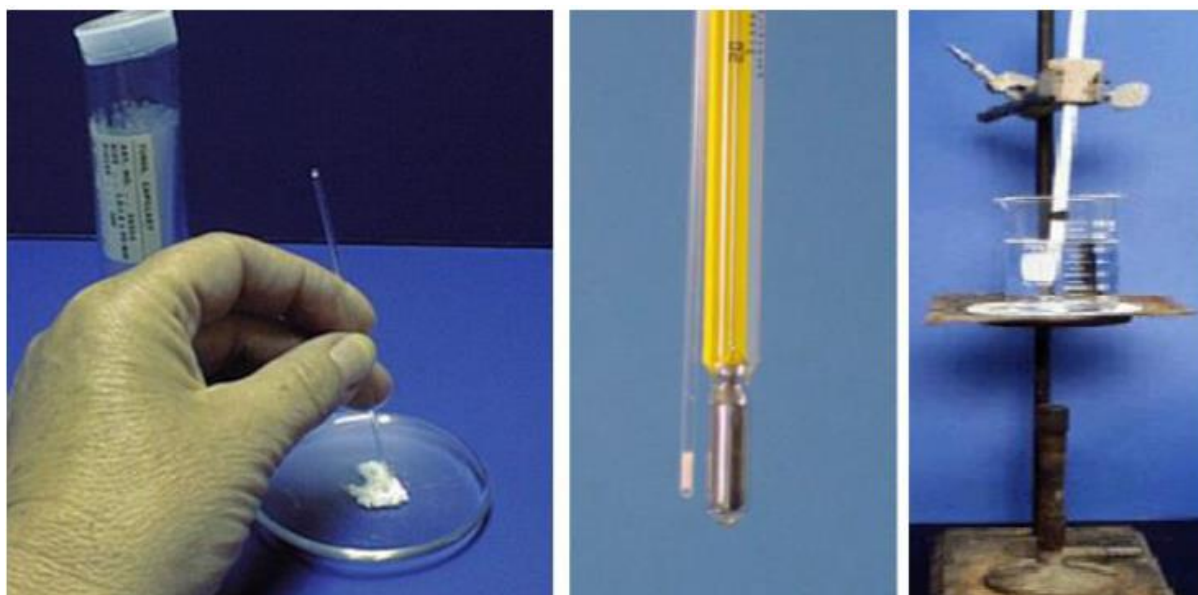


Fig. 4. The set-up for the procedure

Discussion

The amount of sample that you use and the heating rate through the melting range are critical. If you use too many sample or heat too rapidly you will not get accurate results. Be patient and do it right the first time.

Once the sample in the capillary melts, don't use it again. If you have to repeat a measurement for any reason, you must start with a fresh sample in a new capillary.

Questions:

1. Why the content of the beaker (oil bath) is stirring ?
2. Why this method not used for finding the melting points of inorganic compounds?
3. Why could the rate of heating influence the melting point.

B. Boiling point (B. P.)

Purpose:

The purpose of this experiment is to determine the boiling points of various organic compounds and to use these to identify unknowns.

Boiling point Definition:

The boiling point of an organic liquid is the temperature at which its vapour pressure equals the atmospheric pressure over the liquid or it is the temperature at which the vapour and liquid phases are in equilibrium at a given pressure. the boiling point is considered as a criterion of purity of a compound and is useful for identification of organic compounds. Similar to the melting point the boiling point may be sharp or may vary over a temperature range. Pure liquids have sharp boiling points while mixtures show a boiling point range. The atmospheric pressure plays an important role in determination of the boiling point correctly. Reduction of the pressure leads to a decrease or a depression in the boiling point and vice versa.

Experimental Procedures

Boiling Point:

- 1- Obtain a liquid unknown from your instructor. Record the sample number.
- 2- Attach a clean and empty test tube to a thermometer with sewing thread. Put an empty capillary tube in the test tube so that the open end of capillary is down.
- 3- Ensure that the temperature of the paraffin oil is below 50 °C. Place 2-3 mL of sample in the test tube.
- 4- Turn on the hot plate and use a clean glass rod to stir the paraffin oil to ensure a uniform heat distribution.

- 5- Record the temperature when rapid air bubbles come out from the capillary. At this stage, the vapor pressure of the unknown inside the capillary is higher than the atmospheric pressure.

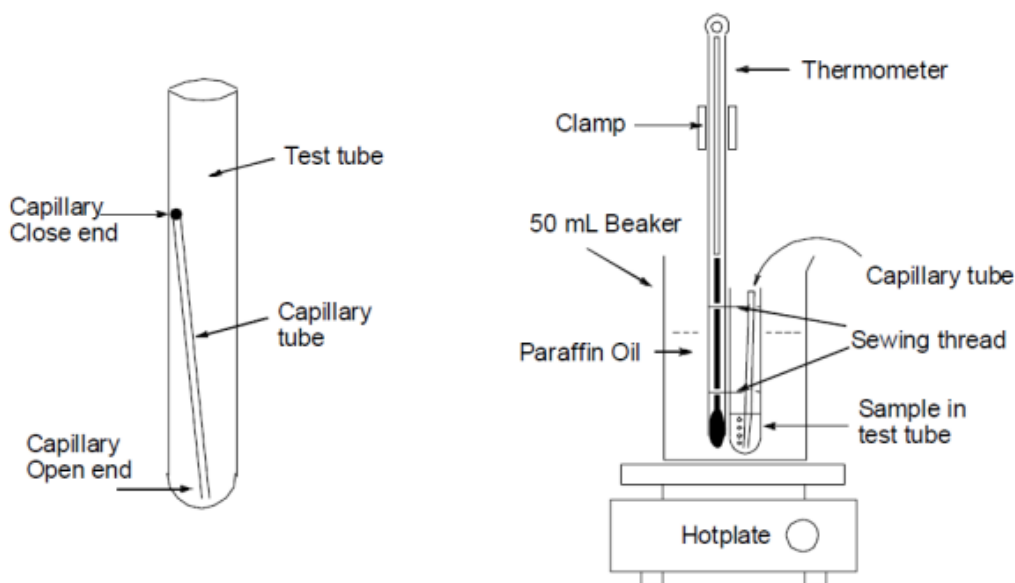


Fig. 5. Apparatus set up of Boiling point determination experiment.

- 6- Turn off the hot plate and carefully insert a ceramic tile between the beaker and the hotplate. Alternatively, you may replace the hot plate with the one that has not been used. However, the thermometer bulb and the content in the test tube should be submerged in the paraffin oil at all times.
- 7- As the temperature decreases, air bubbling will gradually slow down. Record the temperature when you see the last bubble come out and some liquid goes into the capillary.

Questions

- 1- What effect has a change in The atmospheric pressure on the boiling point ?
- 2- What is the definition of boiling point ?
- 3- What is the purpose of determining the boiling point ?