EXPERIMENT (1)

DETERMINATION OF MELTING POINTS

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**)^{0}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^{o}C accuracy.

A pure, nonionic, crystalline organic compound usually has a sharp and characteristic melting point (usually 0.5-1.0 ^{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 compound s

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)^{0}C, impure samples of the same compounds melt at lower temperatures and over a wider range, for example 145-148^{0}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:

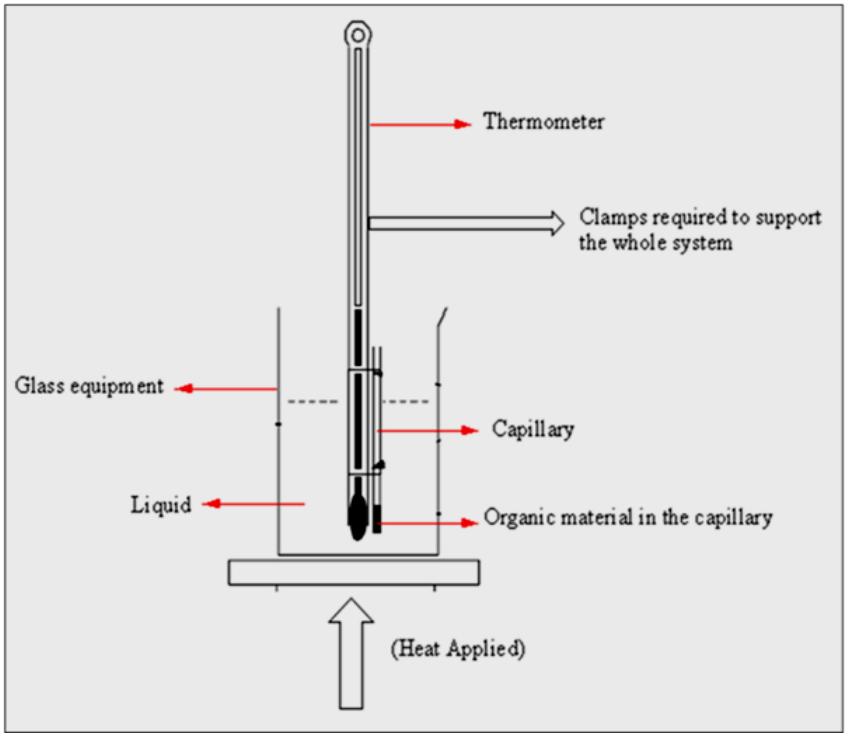


Fig1:Melting point apparats (old fashion model)

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

Properties of oil bath

1)It has a high Boiling point 300^0C rather than water (100^0C)
2) It does not emit toxic gases
3) It is transparence 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)
Thermometer
Bunsen burner
capillary tubes
solid organic compounds
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

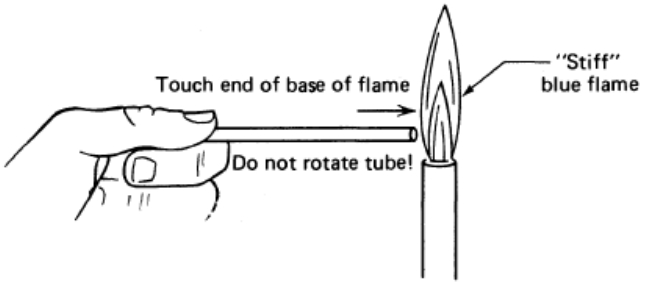
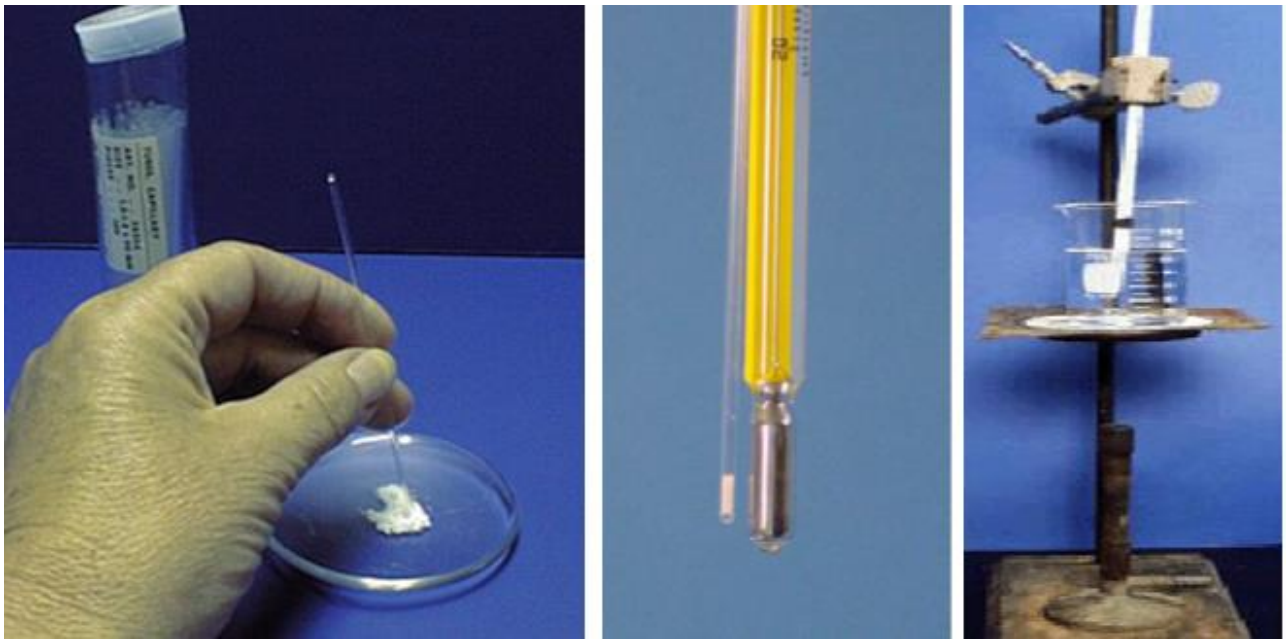


Fig. 32 Closing off a M. P. tube with a flame.

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**

Figure 3. 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 much 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?