

Laboratory of Organic Chemistry

1st stage

2022-2023

Name of experiments:

- 1- Determination of Melting Point.
- 2- Determination of Boiling Point.
- 3- Recrystallization.
- 4- Sublimation.
- 5- Extraction.

Experiment (1):

Determination of Melting Point

Aim of experiment:

- a) To determine the purity of a substance using melting point as physical property.
- b) Identification of an unknown compound using its melting point.
- c) To learn how to obtain an accurate melting point using a Mel-Temp apparatus.

The melting point of a compound is the temperature at which the compound changes from a solid to a liquid state. Experimentally, melting point is actually recorded as the range of temperatures in which the first crystal starts to melt (T₁) until the temperature at which the last crystal just disappears (T₂).

Melting point range = (T₂–T₁)

A pure compound usually has a sharp and characteristic melting point (usually 0.5-1 °C range).

The factors affecting the melting point:

1- Impurities

Impurities decrease 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.

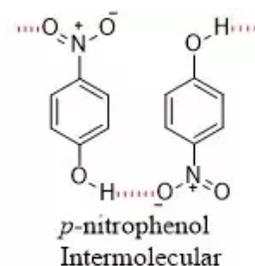
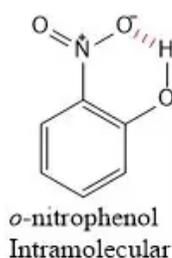
(This phenomenon is used in making the freezing mixture by adding salt to ice)

2- The nature of the organic compounds (bonding)

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.

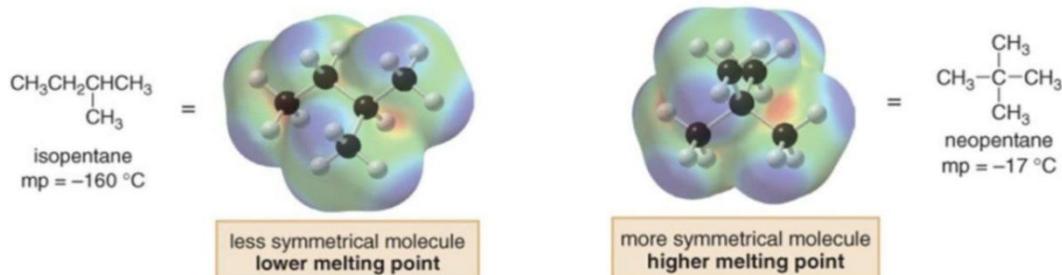
While the **Covalent compounds** consist of molecules rather than ions. Since the attraction between molecules is weaker than is the attraction between ions, covalent compounds usually have a lower melting point than ionic compounds. Moreover, some covalent compounds have polar molecules in which one end is more electronegative than the other, such polar compounds have a higher melting point than nonpolar molecules.

Hydrogen bonding: the compounds having **intermolecular** hydrogen bonding have more melting points than compounds having **intramolecular** hydrogen bonding.



3-Shape of Molecules

Molecular shape influences the melting point of a substance. The tighter the molecules are packed, the higher will be the melting point, for example (neopentane) give a higher melting point than that of (isopentane).



4- 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.

5- pressure

When the pressure increase the melting point increases due to the direct relationship between temperature and pressure.

Note : The rate of heating is the most critical factor affecting experimental results, and should be carefully monitored, particularly close to the expected melting point.

apparatus / Materials:

- Solid organic compounds (Benzoic acid)
- Capillary tubes
- Mel-Temp apparatus

Procedure

- 1- Take a capillary tube and sealed an open end of capillary tube by inserting the tip into a Bunsen flame and turning the tube in your fingers.
- 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.
- 3- Move the powder to the closed end of the capillary tube by tapping it on the table. Repeat until the compound occupies 2-3 mm of the capillary tube end.
- 4- Place the capillary tube in the Mel-Temp apparatus chamber, with the closed end pointed down.
- 5- Record the melting range, which begins when the sample first starts to melt and ends when the sample is completely melted.
- 6- Repeat the experiment with a new capillary tube and fresh quantity of the substance.
- 7- Calculate the average temperatures to get the experimental melting point of the solid.

$$\text{Melting point} = (T_1 + T_2) / 2$$

Sample preparing



H.W:

1. The sample must be in fine powdered form before measuring the melting point, why ?
2. Why could the rate of heating influence the melting point ?
3. Why this method not used for finding the melting points of inorganic compounds ?

Lecturer
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