2015 - 2016



# DETERMINATION OF MELTING POINT

Assistant Lecturer Sahar Mohammed Assistant Lecturer Wid Kadhim

### **Physical Properties of Organic Compounds**

The physical properties of a compound include such things as its,

Color, Odor, Refractive index, Density, Solubility, Melting point & Boiling point.

The exact values of the physical properties of a cpd. depend on its **molecular structure**.

**Success** in laboratory often depends on making a good prediction of physical properties from the cpd. structure.

The physical properties of a cpd. depend largely upon which kind of bonds hold it's atoms together in a molecule.

#### There are two kinds of bonds:

1- <u>Ionic Bonds</u>, formed by the transfer of electrons.

2- Covalent Bonds, formed by the sharing of electrons.

## **Phases of Matter**

Matter ordinarily exists in four phases: Solid, Liquid, Gas and Plasma. Plasma is a high – temperature phase not encountered in the typical organic chemistry laboratory.

As the environment around the materials is *changed*, for example by varying the temp. or pressure, the systems undergo a phase transition, that is the change from one state of matter into another.



### There are four Basic types of Intermolecular Forces :

#### 1. Ion - dipole:

Ion is attracted to polar molecule via this force as ex. (NaCl in water).



#### 2. Dipole – dipole: Polar molecules attracted to each other as forces between HCl molecules.





#### **3.** Dipole - induce dipole:

Polar molecules attracted to nonpolar molecules as for ex. ( Oxygen in water )



by dipole - induce dipole interaction .

4. Induce dipole -induce dipole: London dispersion forces also called van der Waal's forces Nonpolar molecules attraction for each other due to electron distortion.



When two atoms come within 5 nanometers of each other, there will be a slight interaction between them, thus causing polarity and a slight attraction.

#### **Hydrogen bonding :**

Hydrogen bonding is an especially strong kind of dipole – dipole attraction in which a hydrogen atom serves as a bridge between 2 electronegative atoms, holding one by a covalent bond & the other by purely electrostatic forces.

For H-bonding to be important, both electronegative atoms must come from the group: F, O, N.



## **Melting Point:**

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

It's the temp. at which the solid begins to change into liquid under a pressure of 1 atmosphere.

The melting point range: is the difference between the temp. at which the sample begins to melt (T1) & the temp. at which the sample is completely melted (T2). Melting point rang =  $T_2 - T_1$ 

## Organic chemist used melting point to:

#### 1- Get an indication of the purity of cpd.

Sample	Pure	Impure	Decomposed
Melting point	Sharpe	Not sharpen	Not sharpen
Melting point range	0.5 - 1 <sup>0</sup> C	Wider range	Wider range

### 2- Help in identification of organic cpd.

Compound name	Compound formula	Compound Melting point
Sodium chloride	NaC1	801 °C
Sodium acetate	CH <sub>3</sub> COO Na	324 °C
Glacial acetic acid	СН3СООН	16.6 °C

# **Mixed Melting Point:**

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





 $\begin{array}{ccc} A & B \\ m.p. = 120 - 122 \ ^{\circ}C & m.p. = 120 - 122 \ ^{\circ}C \end{array}$ 

#### <u>Answer:</u>

We mix them and measure the m.p. for the resultant mixture, If,

m.p. of mixture < m.p. of original & wider m.p. range  $\rightarrow$  different cpd. m.p. of mixture = m.p. of original & same m.p. range  $\rightarrow$  same cpd.

## **General Technique for m.p. Determination: Capillary Tube method.**

#### **Capillary tube**

Thin glass wall Diameter 1mm

Tube tilted to prevent water condensation inside Stages for correct sealing Wrong ! Tube has been overheated

# Filling a capillary tube:

The open end of the tube is pressed gently & repeatedly into the powdered sample several times.



The sample can be moved to the sealed end by turning the tube over & tapping it on the bench.



A sample height of 2-3 mm in the capillary tube is recommended.

Any compound being loaded into a capillary tube <u>must be:</u> 1- Fully dried. 2- Homogeneous.

3- In powdered form.

## **Melting Point Apparatus:**



a- Hot - oil m.p. bath.
b- Thiele apparatus.
c- Long necked flask.
d- Mel-temp apparatus.
e- Digital m.p. measuring apparatus.

Number of Experiment: I Name of Experiment: Determination of the <u>Melting Point</u> of an unknown sample. Aim of Experiment:

 Identification of an unknown cpd. using it's m.p.
 Determination of purity of a cpd. using the m.p. as a physical property.

Procedure:

1- Preparation of the unknown sample as mentioned before.

2- The capillary tube is attached to a thermo meter by a rubber ring in such a way that the closed end is attached to the bottom of the thermometer's bulb.

3- Then both of them are placed in an oil – bath , ( the rubber ring s



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 (T1) & the temperature at which a clear liquid is observed inside the capillary tube (T2) is recorded as the observed

m.p. range.



 $m.p. = (T_1 - T_2)$ 



#### Notes:

- \* For oil bath, glycerin or paraffin can be used, but glycerin is preferred since it's safer than paraffin.
- \* The temperature of the oil bath should rise very slowly.
- \* Gentle heating with continuous stirring is necessary
- \* After completing the experiment, the oil liquid must be allowed to cool before using it again.

# Salt is added on snow covered roads?

Ice melts at ----? What happens to the freezing point if salt is added?

# Among these 3 samples of the same cpd., which of them is the purest one?



m.p. m.p. m.p. 115 - 119°C 118 - 120 °C 121-122°C

# Two of these bottles contain benzoic acid and one m-nitrophenylacetic acid.



9

m.p.=120 -122 °C m.p.=120 -122°C m.p.=120 -122°C

Run mixed melting points to confirm identification :

Mixed 1 and 2 m.p. = 120 - 122°C

Mixed 1 and 3 m.p. = 114 - 117°C

Mixed 2 and 3 m.p. = 115 - 118°C