

Exp. 1**Determination of Melting Point**

The melting point is the temperature degree at which there is an equilibrium between liquid and solid states of the compound.

The organic materials have specific and characteristic melting points, which can be used to determine the identity of the compound, also, its purity. The existence of any impurity decreases the melting point. Pure organic compounds have sharp melting points and they melt within a range 0.5-1°C, while impure compounds melt over a range of several degrees.

In a crystalline solid the particles acting as structural units-ions or molecules are arranged in some very regular, symmetrical way; there is a geometric pattern repeated over and over within a crystal.

Melting is the change from the highly ordered arrangement of particles in the crystalline lattice to the more random arrangement that characterizes a liquid. Melting occurs when a temperature is reached at which the thermal energy of the particles is great enough to overcome the intracrystalline forces that hold them in position.

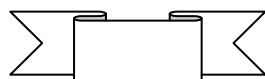
An ionic compound forms crystals in which the structural units are ions. Solid like sodium chloride, for example, is made up of positive sodium ions and negatives chloride ions alternating in a very regular way. There is nothing that we can properly call a molecule of sodium chloride. A particular sodium ion does not "belong" to any one chloride ion; it is equally attracted to six chloride ions. The crystal is an extremely strong, rigid structure, since the electrostatic forces holding each ion in position are powerful. These powerful interionic forces are overcome only at a very high temperature; sodium chloride has a melting point of 801°C. Crystals of other ionic compounds resemble crystals of sodium chloride in having an ionic lattice, although the exact geometric arrangements may be different.

A non-ionic compound, one whose atoms are held to each other entirely by covalent bonds, forms crystals in which the structure units are molecules. It is the forces holding these molecules to each other that must be overcome for melting to occur. In general, these intermolecular forces are very weak compared with the forces holding ions to each other. To melt sodium Chloride we must supply enough energy to break ionic bonds between Na^+ and Cl^- . To melt, methane, CH_4 , we do not need to supply enough energy to break covalent bonds between carbon and hydrogen; we need only supply enough energy to break CH_4 molecules away from each other, methane melts at -183°C .

Procedure

The substance is crushed with the sealed end of a test tube on a filter paper or with a pestle in a clean mortar, and the powdered solid placed to a depth of 1 cm in a capillary tube closed at one end. The tube then placed alongside a thermometer by a rubber ring, in a beaker of paraffin or oil or glycerol. The beaker then slowly heated, and the rate of heating decreased as the expected melting point is approached, the crystals then collapse to a clear liquid.

Recorded the range between the temperature at which the powdered solid begins to collapse (T_1), and the temperature at which a clear liquid is observed in the capillary tube (T_2).



Exp. 2**Determination of Boiling Point**

The boiling point is the degree at which a liquid starts to boil and transfer to a gas state under the systematic pressure. At this point the vapour pressure of the substance is equal to the atmospheric pressure.

The organic materials possess specific and characteristic boiling points, which is a good guide to its purity and may be useful for identifying the liquid.

The determination must be done at a known pressure because boiling point unlike melting point is affected considerably by changes in pressure. Reduction of the pressure leads to a decrease in the boiling point and vice versa.

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.

Although the particles in a liquid are arranged less regularly and are free to move about than in a crystal, each particle is attracted by number of other particles. Boiling involves the breaking away from the liquid of individual molecules or pairs of oppositely charged ions. This occurs when a temperature is reached at which the thermal energy of the particles is great enough to overcome the cohesive forces that hold them in the liquid.

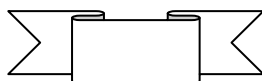
In the liquid state the unit of an ionic compound is the ion. Each ion is held strongly by a number of oppositely charged ions. There is nothing we could properly call a molecule. A great deal of energy is required for a pair of oppositely charged ions to break away from the liquid; boiling occurs only at a very high temperature.

In the liquid state the unit of a non-ionic compound is the molecule. The weak intermolecular forces here dipole-dipole interactions and van der Waals forces are more readily overcome than the strong interionic forces of ionic compounds, and boiling occurs at a very much lower temperature. The bigger the molecules, the

stronger the van der Waals forces. Liquids whose molecules are held together by hydrogen bonds have high boiling points.

Procedure

In a small tube put 2 ml from the organic liquid. Put in the small tube, a capillary tube with its closed end outside the liquid. The small tube is then placed alongside a thermometer by a rubber ring, in a beaker contains glycerol. The beaker then slowly heated. When we approach the boiling point, a rapid stream of bubbles will be released from the capillary tube (inside the organic liquid). Write the temperature degree when the liquid starts to rise in the capillary tube.



Exp. 3**Filtration**

Dissolving a solid substance involves the separation of the structural units ions or molecules, from each other, and the solvent molecules occupy the space between them.

The filtration is an important procedure after completing the reaction either to separate the solid product as a precipitate, or to get rid of insoluble impurities or reactant materials. The desired soluble compound is recovered from the filtrate by evaporating solvent.

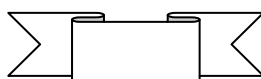
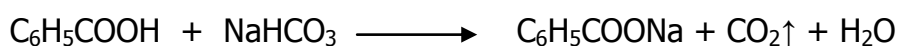
The liquid is poured into a filter paper fitted in a funnel and either the precipitate is collected or the filtrate that contains the desired soluble compound is collected.

In this method we take the advantage that one compound in the mixture is readily soluble in a given solvent whereas the remainder of the mixture may be relatively insoluble.

Procedure

An impure mixture containing about 0.5 gm benzoic acid and 0.5 gm glucose is to be dissolved in about 10 ml of distilled water. The glucose will dissolve in water while benzoic acid remains precipitated. After filtration, benzoic acid remains as a precipitate on the filter paper while the glucose pass with the filtrate as a solution. The glucose can be recovered from the filtrate by evaporating the solvent (water).

To test that the precipitate on the filter paper is the benzoic acid, we add sodium bicarbonate solution on the precipitate. The benzoic acid will be dissolved due to the formation of soluble sodium benzoate and bubbles of the evolved CO₂ gas will be seen.



Exp. 4**Recrystallization**

It is a very important method to purify solid organic substance. These compounds when isolated from organic reactions are usually impure; they are usually contaminated with small amounts of other compounds that are produced along with the desired compound. The method depends on the difference of substance solubility in different temperatures. Most of the organic substances are more soluble in hot solutions than cold. Therefore, the substance we need to crystallize dissolves in a hot solution, and then filter to remove impurities that does not dissolve, and then allow for crystallization by cooling gradually.

The solvent used for recrystallization should be chemically inert toward the solute, dissolve the solute readily at its boiling point but sparingly at low temperature, dissolve impurities either very easily or not at all, and gives good crystals.

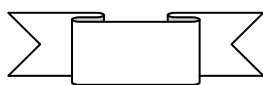
Sometimes samples to be purified may contain soluble colored impurities that may cause the solution and the crystals to be colored. These colored impurities can be removed by adding charcoal. Charcoal has a large active surface on which the colored impurities adsorbed.

If crystallization does not take place scratch the sides or the bottom of the flask below the surface of the solution with a glass rod, or add a small crystal of the pure compound, or evaporate some of the solvent to induce the crystallization process.

Procedure

Put 1 gm of the compound in a beaker then add 30 ml water, and heat until the compound dissolves. If the substance is colored add few grams charcoal. Filter the hot solution, cool, then pure crystals will be formed. Some times it is important to warm the funnel and the filter paper to prevent crystallization of the substance on

the funnel. Separate the crystals by filtration, then wash with water to remove any impurities.



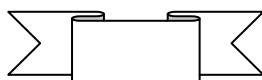
Exp. 5**Sublimation**

It is the direct transfer of a substance from the solid state to the gaseous state. The vapour pressure of the substance should be high at this point of transfer. The substance after sublimation will be very pure, and loses some of its weight.

Procedure

Put in a crucible small amount from the substance, cover the crucible with a filter paper has many small holes on its surface. Put a glass funnel over the filter paper and the crucible, and put a piece of cotton as a plug in the top of the funnel pipe. Heat the crucible gently and watch the substance crystals precipitate on the walls of the funnel, any impurities will remain in the crucible. Collect the pure crystals and weigh it, then count the yield percentage.

$$\text{Yield \%} = \frac{\text{weigh of the product}}{\text{weigh of the sample}} \times 100$$



Exp. 6**Extraction**

Extraction is one of the important methods for separating and purifying organic substances. It is used on a large scale in extraction organic compounds from seeds and leaves of plants. It depends on the difference of the organic substance solubility in two different immiscible solutions, like water and chloroform. The solute is distributed between two unmixed liquid phases. Two layers will be formed; the upper layer is for the liquid of the lower density and the lower layer for the liquid of the higher density.

The method is used sometimes to remove an organic compound from a solution when the use of distillation is not convenient.

Water is usually one of the solvents used in this process, and the other solvent is a non-polar organic liquid, or slightly polar. Ether is one of the important organic solvents used extensively as an extracting solvent. It is slightly soluble in water and its efficiency in use can be improved considerably by the addition of a small amount of an ionizable salt like NaCl or K₂CO₃, to the water layer. This leads to an increase in the polar property of this solution that results in a decrease in the solubility of the non-polar compound. Therefore, ethanol, which is completely miscible with water, becomes an immiscible liquid with respect to the water layer when the water is saturated with a salt such as NaCl. This is known as the *salting out* process.

The extraction solvent should be readily dissolve the substance to be extracted, has a low boiling point so that it can be readily removed, doesn't react with the other solvents, immiscible with water.

The ratio of the concentrations of solute in the two solvents is known as the distribution or partition coefficient K .

$$K = \frac{C_A}{C_B}$$

$$K = \frac{\text{conc. of compound in organic solvent}}{\text{conc. of compound in water}}$$

$$K = \frac{\text{solubility of compound in organic solvent (gm/100 ml)}}{\text{solubility of compound in water (gm/100 ml)}}$$

$$K = \frac{C_{\text{org.}}}{C_{\text{wat.}}} = \frac{\text{wt.}_{\text{org.}}/V_{\text{org.}}}{\text{wt.}_{\text{wat.}}/V_{\text{wat.}}}$$

Where:

$C_{\text{org.}}$ = concentration of the solute in the organic layer

$C_{\text{wat.}}$ = concentration of the solute in the aqueous layer

$\text{wt.}_{\text{org.}}$ = weight of the solute in the organic layer

$\text{wt.}_{\text{wat.}}$ = weight of the solute in the aqueous layer

$V_{\text{org.}}$ = volume of the organic solvent

$V_{\text{wat.}}$ = volume of the aqueous solvent

Procedure

Method 1: (Extraction of salicylic acid from its aqueous solution by chloroform)

- 1- put an unknown weight of salicylic acid in a separatory funnel, and add 25 ml of chloroform and 25 ml distilled water.
- 2- close the funnel hole with a stopper, shake the mixture well for 10 minutes, then open the stopper for a while in order to release the excess pressure.
- 3- Put the funnel on a metal ring for about 10 minutes for complete separation of the two layers.
- 4- Separate the lower layer (chloroform) slowly until the aqueous layer is about to enter the hole of the stopcock.
- 5- Pour the upper aqueous layer through the neck of the separatory funnel. To recognize the identity of any layer, mix few drops of the layer with few drops of

water in a test tube. If there are two layers, then the layer is the organic layer, if there is one layer, then that layer is the aqueous layer.

6- Put 10 ml from the organic layer in a conical flask, and add two drops phenolphthalein indicator.

7- Titrate against 0.1 N NaOH solution until the appearance of pink color.

8- Repeat steps 6 and 7 for the aqueous layer.

Calculations

The aqueous layer:

$$N \times V = \frac{\text{wt. } \mathbf{NaOH}}{\text{eq. wt. } \mathbf{salicylic\ acid}} \times 1000$$

$$0.1 \times V = \frac{\text{wt.}}{138} \times 1000$$

$$\text{wt. in 10 ml} \times 2.5 \text{ (enlarging factor)} = \text{wt. in 25 ml} = x$$

The organic layer:

$$0.1 \times V' = \frac{\text{wt.}}{138} \times 1000$$

$$\text{wt. in 10 ml} \times 2.5 \text{ (enlarging factor)} = \text{wt. in 25 ml} = y$$

$$x \times y = \text{weight of salicylic acid.}$$

$$K = \frac{\text{wt.}_{\text{org.}} / v_{\text{org.}} \text{ 25 ml}}{\text{wt.}_{\text{wat.}} / v_{\text{wat.}} \text{ 25 ml}}$$

Method 2: (Extraction of isopropyl alcohol from its aqueous solution by chloroform)

1- Put in a separatory funnel 30 ml of the alcohol (50% volume), and add 10 ml chloroform

2- close the funnel hole with a stopper, shake the mixture well, then open the stopper for a while in order to release the excess pressure.

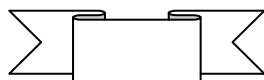
3- Put the funnel on a metal ring fixed on a stand, and allows the mixture to settle for 15 minutes. Two defined layers will be formed.

4- Separate the lower layer (chloroform) slowly until the aqueous layer is about to enter the hole of the stopcock, and put it in a cylinder, and determine its volume.

5- Pour the upper aqueous layer through the neck of the separatory funnel. To recognize the identity of any layer, mix few drops of the layer with few drops of water in a test tube. If there are two layers, then the layer is the organic layer, if there is one layer, then that layer is the aqueous layer.

The aqueous solution may be returned to the funnel, and add 10 ml chloroform and repeat the process.

A distillation process can be done to separate alcohol from chloroform.



Exp. 7**Distillation**

It is the transfer of liquid by heating to a vapour and condensing this vapour. The success of the process depends on the ability difference of the substances to transfer to vapour, and this depends on the forces works between molecules. The difference in the evaporation rang called vapour pressure. If the vapour pressure be equal to the external pressure, the liquid boil. The polar liquids boil at higher degrees than nonpolar liquids that have the same molecular weigh, due to the formation of intermolecular hydrogen bonds.

The **simple distillation** is convenient to separate a liquid contaminated with nonvolatile impurities. Also to separate two substances, if they do not have identical pressure. It is possible to separate two substances if the difference between their boiling points is 30-80°C.

In addition to the simple distillation there are; **vacuum distillation**, which is carried out under reduced pressure. The method is used to separate liquids with high boiling points or are unstable to heat. **Steam distillation** which is used for the purification on non-volatile compounds and which are immiscible with water. **Reflux distillation** which is used to prevent the reactants from loss by evaporation during reaction. **Fractional distillation** which is used to collect separate fractions according to controlled boiling ranges during the distillation of a mixture of a substances.

Procedure of simple distillation

(Separation of acetone from water)

Put 30 ml from a mixture of water and acetone in a boiling flask fitted with a thermometer on its top, and a condenser on its side. add some boiling stone, and heat between 56-62°C, and collect the distilled liquid (acetone). Increase the temperature to 90°C, and collect water. Count the volume percentage for each water and acetone.

$$\text{Volume percentage} = \frac{\text{Volume of substance}}{\text{Volume of solution}} \times 100$$

