Experiment no. 1

Distillation a mixture of two miscible liquids by normal distillation

Distillation: - is the process of separating the component or substances from a liquid mixture by selective evaporation and condensation.

There are 3types of distillation including:-

1- Normal Distillation
2- Fractional Distillation
3- vacuum distillations

Purpose:

-To purify a compound by separating it from a non-volatile or less-volatile Material.
- To separate a mixture of two miscible liquids (liquids that mix in all Proportions) with different boiling points.

Normal distillation is usually used only to separate liquids whose boiling points differ greatly (more than 20°C), or to separate liquids from nonvolatile solids or oils.

Boiling point: - the **boiling point** of a liquid is the temperature at which the vapor pressure equals the applied pressure.

Equipment and materials:

1. Round bottom flasks 2. Condenser 3. Thermometer

4. Graduated cylinders 5. Receiver distilling still 6. Hot plate

7. Boiling stone (chips)

Procedure

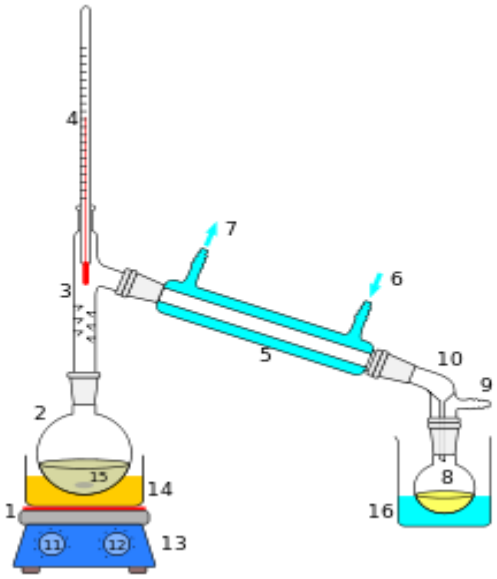
1- Obtain 50 mL of the alcohol (ethanol or methanol)/water solution in a round bottom flask with several boiling chips. (B.p. of ethanol=78 C^o , B.p. of methanol =65 C^0

2- Perform a Normal distillation, recording the temperature with every 5 mL

Of distillate.

4. Record the temperature and watch the time when the first drop of distillate was taken. Collect distillate in a flask or a graduated cylinder.

7. Plot the graph of the collected boiling temperature (Y axis) versus volume (mL) of distillate (X axis).



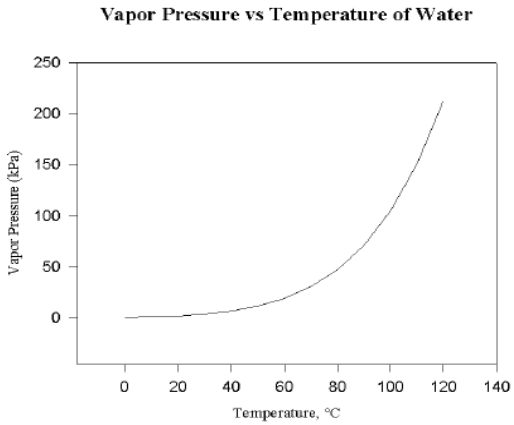


Fig 1. Vapor pressure dependence on temperature for water

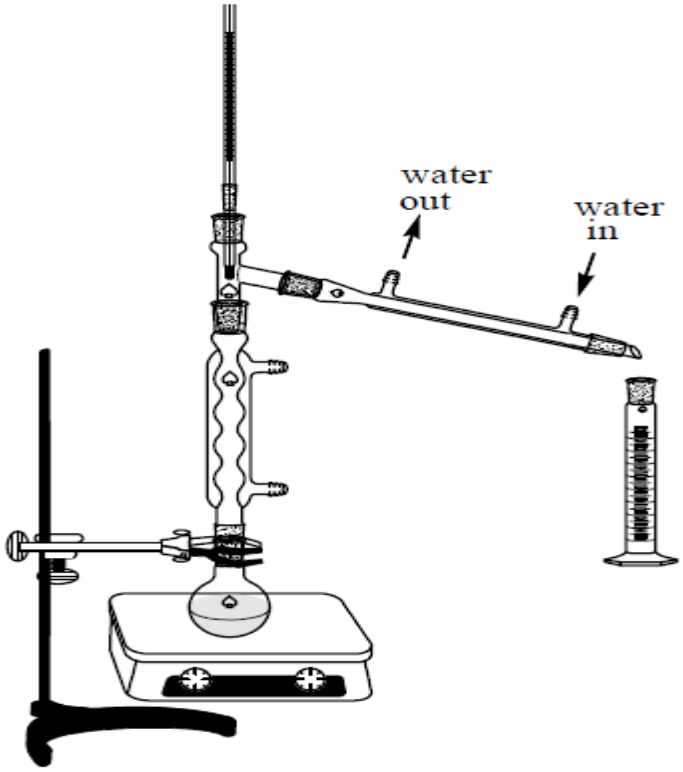
Fractional Distillation

Exp.no.2 Distillation **a mixture of two miscible liquids by Fractional distillation**

This method is usually used to separate or isolated the component of a liquid mixture containing two or more miscible fraction.

1- If the components of the mixture with low difference in the boiling points ,
2- They are closed in their chemical structure like a mixture of (butanol + ethanol or propanol) .
   B.p. methanol = $65C^0$ B.p.Ethanol= $78C^0$

As the solution to be purified is heated, its vapors rise to the fractionating column. As it rises, it cools, condensing on the condenser walls and the surfaces of the packing material. Here, the condensate continues to be heated by the rising hot vapors; it vaporizes once more. However, the composition of the fresh vapors are determined once again by Raoult's law. Each vaporization-condensation cycle (called a *theoretical plate*) will yield a purer solution of the more volatile component.[25] In reality, each cycle at a given temperature does not occur at exactly the same position in the fractionating column; *theoretical plate* is thus a concept rather than an accurate description.

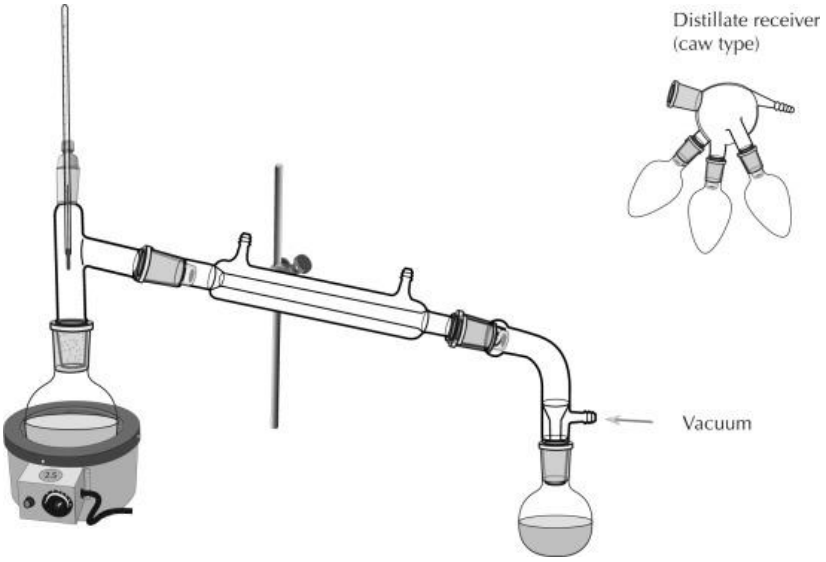


Vacuum distillation

Exp. No.3:- Vacuum distillation under vacuum presser

Also called distillation under reduced pressure, vacuum distillation is used to purify or separate liquids having a boiling point above 150 °C or a lower boiling point but thermally unstable. The vacuum can be created using a water pump or vacuum pump.
It characterized with:-

1- Reducing distillation time.
2- Reducing boiling point.
3- Maintaining organic material specification.



Exp. No. 4 Determination the Melting point of solid organic compound

Melting point Is the temperature at which the solid substance convert to the fused state. (Change the stat from solid to liquid)

Melting point depend on:-

1- Molecular weight of solid organic compound.
2- The purity of solid organic compound (the impure substance has low melting point)

The melting point measured at the first crystal fusing (T1) at heating and then at the first crystal Solid at cooling (T2).

Melting point = (from – to)

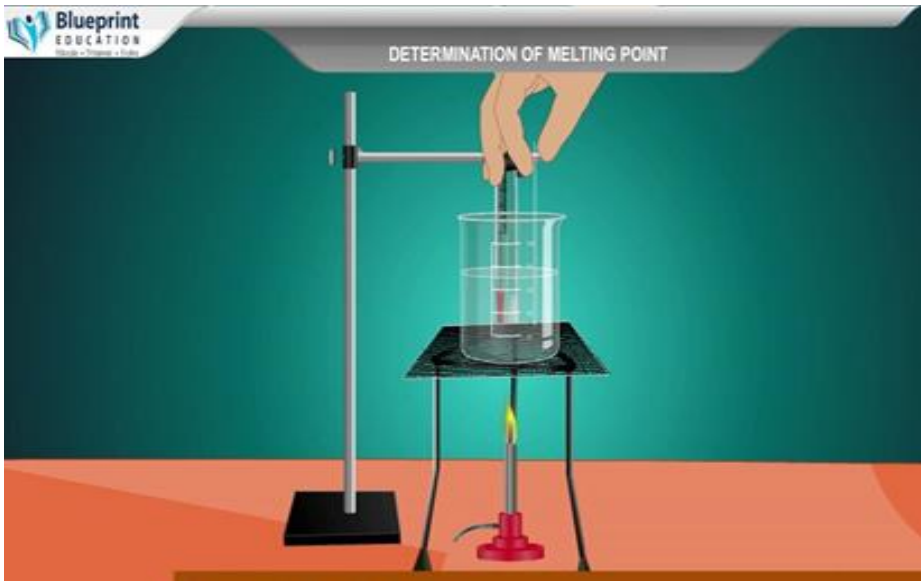
M. P. Naphthalene = 80 .26 C^0

M.P. benzoic acid = 123.3

T1 at the first crystal fused =

T2 at the last crystal disappear =

Melting point = $\frac{T1 + T2}{2}$ =



1- Naphthalene
2- Test tube or capillary tube
3- Beaker
4- Thermometer
5- Hot plate or benzene burner

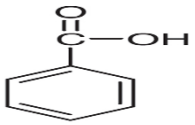
Experiment no.5 Purification of benzoic acid by ReCrystallization

ReCrystallization is a technique for purifying solids that contain small amounts of impurities. This technique is based on the fact that both the solid and the impurities may dissolve in a given solvent, but not to the same extent.

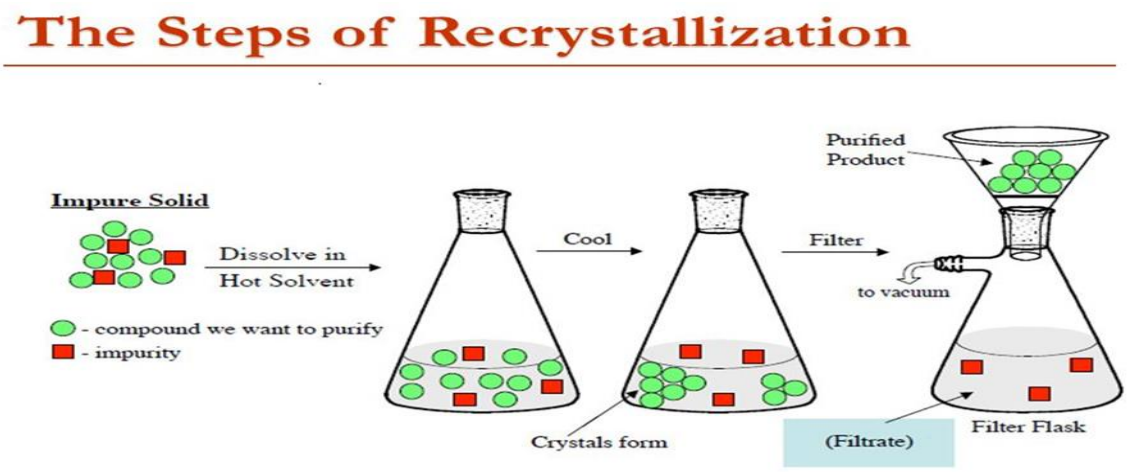
Solubility of solid organic compounds depends on :

1- Ability of solid organic compound dissolving in a liquid (polarity).
2- Temperature degree of the liquid (solvent) .
3- Solubility is a function of concentration.

- First we dissolve the impure solid (Benzoic acid + Charcoal) in a solvent that dissolves benzoic acid, We try to dissolve the maximum amount of sample in the minimum amount of solvent at high temperature (at or near the boiling point of the solvent). After the sample is completely dissolved.
- Filtrate the hot solution
- Cooling after filtration notes the formation of pure crystals
- Drying and weighting



% of pure solid comp.= Wt. of pure comp.
Wt. of Impure comp. X 100



Experiment no. 5 Recrystallization of sold organic compound

Is a technic used for purify chemicals by dissolving both impurities and a compound in an appropriate solvent, either the desired compound or impurities can be removed from the solution, living the another behind.

Solubility of solid organic compound depends on :-

1- Ability of dissolving in a liquid.
2- Temperature degree of the liquid.
3- Functional group in the solid.

The procedure involves:-

(1) Dissolving the impure material in a minimum amount of boiling solvent.

(2) Filtering the hot solution to remove insoluble impurities,

(3) Allowing the solution to cool slowly to deposit crystals of the compound,
(4) filtering the crystals from the solution (called the mother liquor),

(5) Washing the crystals with a little cold solvent to remove the mother liquor,
(6) drying the crystals to remove the last traces of solvent.

Percent recovery from recrystallization is calculated as follows:

$$\%\ \text{recovery} = \frac{\text{mass after recrystallization}}{\text{initial mass}} \times 100\,\%$$

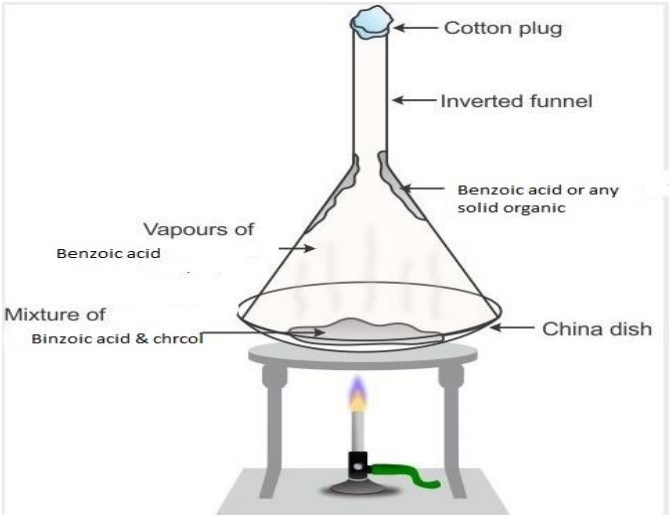
Experiment .No. 6 Purification by Sublimation of solid organic compound

Sublimation is the transition of a substance from a solid state to a gas state without passing through the liquid state.

Sublimation is a technique used to purify substances that have non-volatile

impurities the purified compound can be collected from the cooling

surface which has a non –volatil residue.



Compound which sublimated (Benzoic acid, Naphthalene, camphor)

1-Weight of impure Benzoic acid = 3 g

2-Weight of Watch glass (empty) = g

3-Weight of Watch glass + sublimate = g

% Recovery = Weight of sublimate X 100

Weight of impure benzoic acid

Experiment No.7 Differential Extraction (Extraction of Benzoic acid in Organic Solvent)

Is the method of separation of any organic component present in an aqueous solution.

In this process, we use an organic solvent for which the solubility of the desired compound is more than compared to that in water. Any organic compound present in an aqueous medium, it is separated by shaking it with an organic solvent in which it is more soluble than in water. The organic solvent and the aqueous solution should be immiscible with each other so that they form two distinct layers which can be separated by separating funnel.

It depend on :

1- The reagent which reacts with compound to be extracted like (Sodium hydroxide NaOH , Sodium Carbonate Na_2CO_3 , Sodium bicarbonate $NaHCO_3$).
2- To convert the compound to be extracted to its salt (the fact that salt is soluble in organic solvent.
3- The functional groups existing in the compound to be extracted.