TOPIC:DISTILLATION

Disclaimer

All the content material provided here is only for teaching purpose

Distillation is an unit operation which involves separation of a vaporizable component from a multi-component system and subsequent condensation of vapours."

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

"Distillation is defined as the separation of the components of a liquid mixture by a process involving vaporization and subsequent condensation at another place."

APPLICATION

- Separation of volatile oils- cloves(Eugenol comprises 72-90%, Vanilin, acetyl eugenol).

  Separation of drugs obtained from plant and animal sources- Vit. A from fish liver oil.

- Purification of organic solvents-absolute alcohol (100%).

  Purification of drugs obtained from chemical process.

Manufacture of official preparations -sprit of nitrous ether, sprit of ammonia, D.water and water for inj.

Quality control methods- Alcohol content in elixir(4-40%).

Raoult's Law

It express a quantitative relationship between the concentration and vapour pressure.

It states that partial vapour pressure of each volatile constituent is equal to vapour pressure of the pure constituent multiplied by its mole fraction in the solution at a given temperature.

Suppose Homogeneous mixture of liquid A and B

Partial vapour pressure of component A in Mixture = $P_A = P°_A * X_A$

Mole fraction of A in solution = X_A

Vapour pressure of A in pure state = $P°_A$

Partial vapour pressure of component B in Mixture = $P_B = P°_B * X_B$

Mole fraction of B in solution = X_B

Vapour pressure of A in pure state = $P°_B$

Total Vapor pressure of Mixture

$$P_T = P_A + P_B$$

$$P_T = P°_A * X_A + P°_B * X_B$$

Positive Deviation & Negative Deviation
(Real Solution)

Positive Deviation:

In some liquids systems, the total vapor pressure is greater than the sum of the partial pressures of the individual components

Ex: benzene and ethanol.

Differ in their polarity, length of hydrocarbon chain and degree of association.

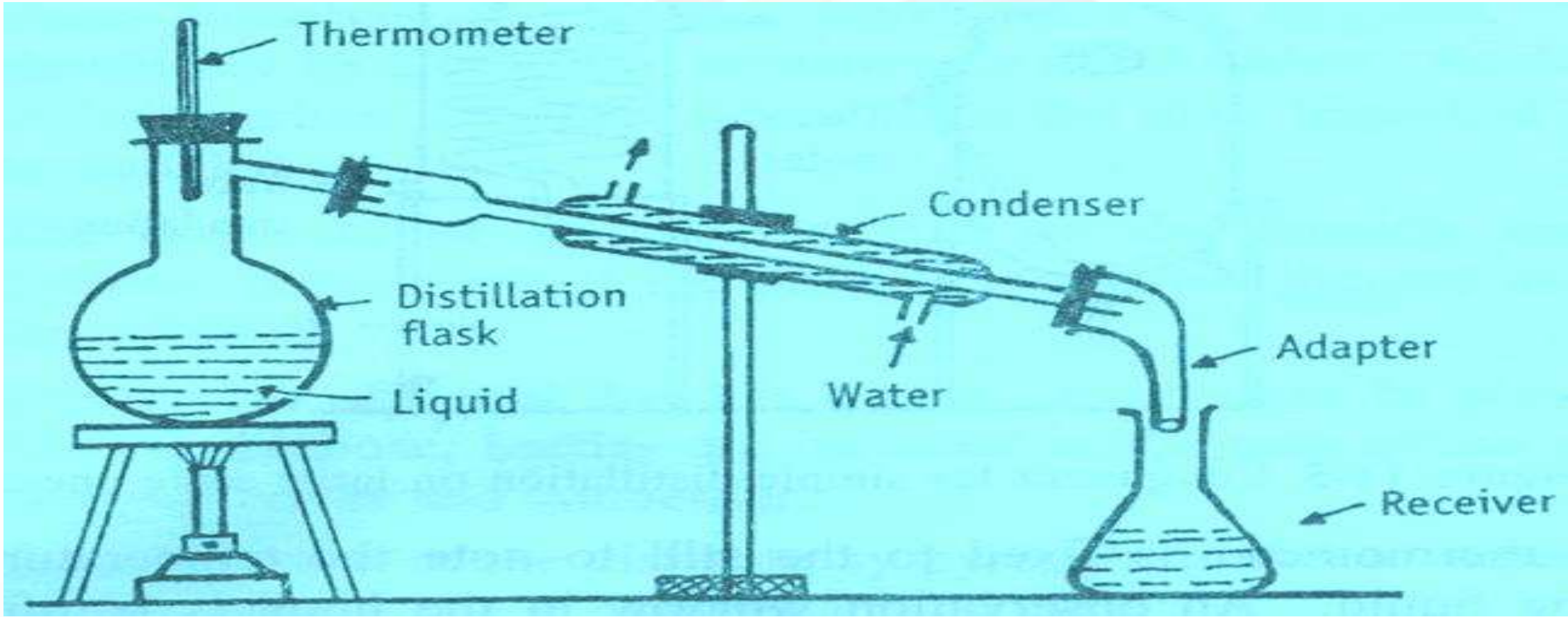
Negative Deviation:

In some liquid systems, the total vapor pressure is lower than that of the sum of the partial pressures of the individual components.

Ex: Chloroform and acetone

Due to hydrogen bonding, salt formation and hydration

Distillation Assembly



General Equipment for Distillation:

STILL :

It is a vaporizing chamber and used to place the material to be distilled.

The still is heated by a suitable means for vaporization of the volatile constituents.

On laboratory scale round bottom flasks made of glass are used so that the progress of the distillation can be noticed.

A condenser is attached to the still using appropriate joints. A trap is inserted between distillation flask and condenser.

CONDENSER :

Used to condense the vapor

It is kept cold by circulating water/air through jacket.

Types:

Single-surface condensers

- Straight Tube
- Bulb type
- Spiral
- Coiled type

Double-surface condensers
Multi-tubular condensers

The condenser is connected to a receiver through a suitable adapter.

Classification of distillation methods

1) Simple Distillation (Differential distillation)
2) Flash Distillation (Equilibrium distillation)
3) Vacuum distillation (distillation under reduced pressure)
4) Molecular Distillation (Evaporation distillation or short path distillation.)
5) Fractional Distillation (Rectification)
6) Aezotropic and extractive Distillation
7) Steam Distillation
8) Destructive Distillation
9) Compression Distillation

SIMPLE DISTILLATION

Simple distillation is a process of converting a single constituent from a liquid (or mixture) into its vapour, transferring the vapour to another place and recovering the liquid by condensing the vapour, usually by allowing it to come in contact with a cold surface.

This process is known **differential distillation**, as distillation is based on the differences in volatilities and vapour pressures of the components in the mixture.

- . **Principle**:
- Liquid boils when its vapour pressure is equal to atmospheric pressure. Simple distillation is **conducted at its boiling point**. The higher the relative volatility of a liquid, the better is the separation by simple distillation. Heat is supplied to the liquid so that it boils. The resulting vapour is transferred to a different place and condensed.

Applications:

•For the preparation of distilled water and water for injection.

•Volatile and aromatic waters are prepared.

•Organic solvents are purified.

•A few official compounds are prepared by distillation. Examples are spirit of nitrous ether and aromatic spirit of ammonia.

•Non-volatile solids are separated from volatile liquids

- Two evaporators are connected together with a piping arrangement so that the vapour from the calandria of first effect (which is heated by steam) is used to heat the calandria of the second effect.
- This means that the calandria of the second effect is used as condenser for the first effect, so that latent heat of vaporisation is used to evaporate more quantity of the liquid instead of its going as waste.
- The vapour from the second effect then taken to a condenser and converted into liquid.

SIMPLE DISTILLATION

•It consists of a distillation flask with a side arm sloping downwards.

•Condenser is fitted into the side arm by means of a cork.

• The condenser is usually water condenser, i.e., jacketed for circulation of water.

•The condenser is connected to a receiver flask using an adapter with ground glass joints.

•On a laboratory scale, the whole apparatus is made of glass.

Procedure:

•Bumping is avoided by adding small pieces of porcelain before distillation.

•A thermometer is inserted into the cork and fixed to the flask.

•The thermometer bulb must be just below the level of the side arm.

•Water is circulated through the jacket of the condenser.

•The contents are heated gradually. The liquid begins to boil after some time.

•The vapour begins to rise up and passes down the side arm into the condenser.

•The temperature rises rapidly and reaches a constant value.

•The temperature of the distillate is noted down, which is equal to the boiling point of the liquid.

•The vapour is condensed and collected into the receiver.

•The flame is adjusted so that the distillate is collected at the rate of one to two drops per second.

Flash Distillation

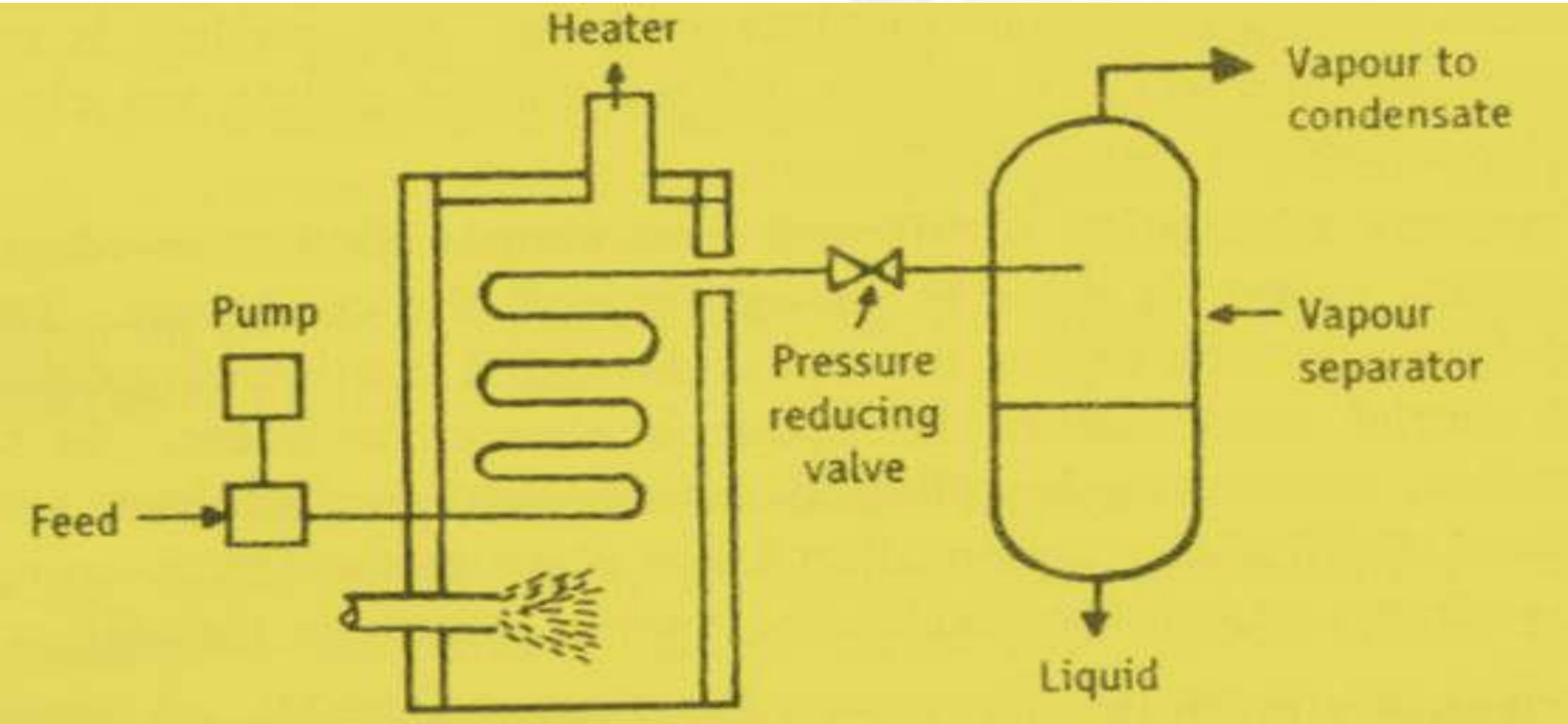


Figure 11-7. Apparatus for flash distillation.

Flash distillation is defined as a process in which the entire liquid mixture is suddenly vaporized (flash) by passing the feed from a high pressure zone to a low pressure zone.

Principle: When a hot liquid mixture is allowed to enter from a high-pressure zone into a low-pressure zone, the entire liquid mixture is suddenly vaporised.

This process is known as flash vaporisation. During this process the chamber gets cooled. The individual vapour phase molecules of high boiling fraction get condensed, while low boiling fraction remains as vapour.

Uses: Flash distillation is used for separating components, which boil at widely different temperatures. It is widely used in petroleum industry for refining crude oil.

Advantages: Flash distillation is a continuous process.

Disadvantages:

It is not effective in separating components of comparable volatility.

It is not an efficient distillation when nearly pure components are required, because the condensed vapour and residual liquid are far from pure.

Working:

•The feed is pumped through a heater at a certain pressure.

•The liquid gets heated, which enters the vapour-liquid separator through a pressure-reducing valve.

•Due to the drop in pressure, the hot liquid flashes, which further enhances the vaporisation process.

•The sudden vaporisation induces cooling. The individual vapour phase molecules of high boiling fraction get condensed, while low boiling fraction remains as vapour.

•The mixture is allowed for a sufficient time, so that vapour and liquid portions separate and achieve equilibrium.

•The vapour is separated through a pipe from above and liquid is collected from the bottom of the separator.

•By continuously feeding into the still, it is possible to obtain continuous flash distillation.

•The operating conditions can be adjusted in such a way that the amount of feed exactly equals the amount of material removed.

VACUUM DISTILLATION

The distillation process in which the liquid is distilled at a temperature lower than its boiling point by the application of vacuum. Vacuum pumps, suction pumps, etc. are used to reduce the pressure on the liquid surface. Distillation under the reduced pressure is based on the principle of the simple distillation with some modifications.

Principle:

•Liquid boils when vapour pressure is equal to the atmospheric pressure, i.e., pressure on its surface. If the external pressure is reduced by applying vacuum, the boiling point of liquid is lowered.

•Therefore, the liquid boils at a lower temperature. This principle is illustrated using an example of water.

• Water boils at an 100°C at an atmospheric pressure is 101.3I kPa (760 mm Hg). At 40°C, the vapour pressure of water is approximately 9.33 kPa (70 mm Hg). Hence, the external pressure is reduced to 9.33 kPa (70 mm Hg) where water boils at 40°C. The net result is the increase in rate of mass transfer into vapour.

Applications:

Preventing degradation of active constituents (≈ 55◦C)

Enzymes - malt extract, pancreatin

Vitamins - thiamine, ascorbic acid

Glycosides - anthraquinones

Alkaloids - hyocyamine to atropine

Disadvantages:

In vacuum distillation, persistent foaming occurs. This may be overcome by adding capryl alcohol to the liquid or by inserting a fine air capillary tube in the second neck of the Claisen flask.

The stream of air is drawn in and breaks the rising foam. The above method is not suitable for the preparation of semisolid or solid extracts by distillation under vacuum.

Molecular distillation

It is defined as a distillation process in which each molecule in the vapour phase travels mean free path and gets condensed individually without intermolecular collisions on application of vacuum. Molecular distillation is based on the principle of the simple distillation with some modifications. This is also called Evaporation distillation or Short path distillation.

Principle:

The substances to be distilled have very low vapour pressures. examples are viscous liquids, oils, greases, waxy materials and high molecular weight substances.

These boil at very high temperature. In order to decrease the boiling point of the liquids, high vacuum must be applied.

The pressure exerted by vapors above the liquid is much lower. At very low pressure, the distance between the evaporating surface and the condenser is approximately equal to the mean free path of the vapour molecules.

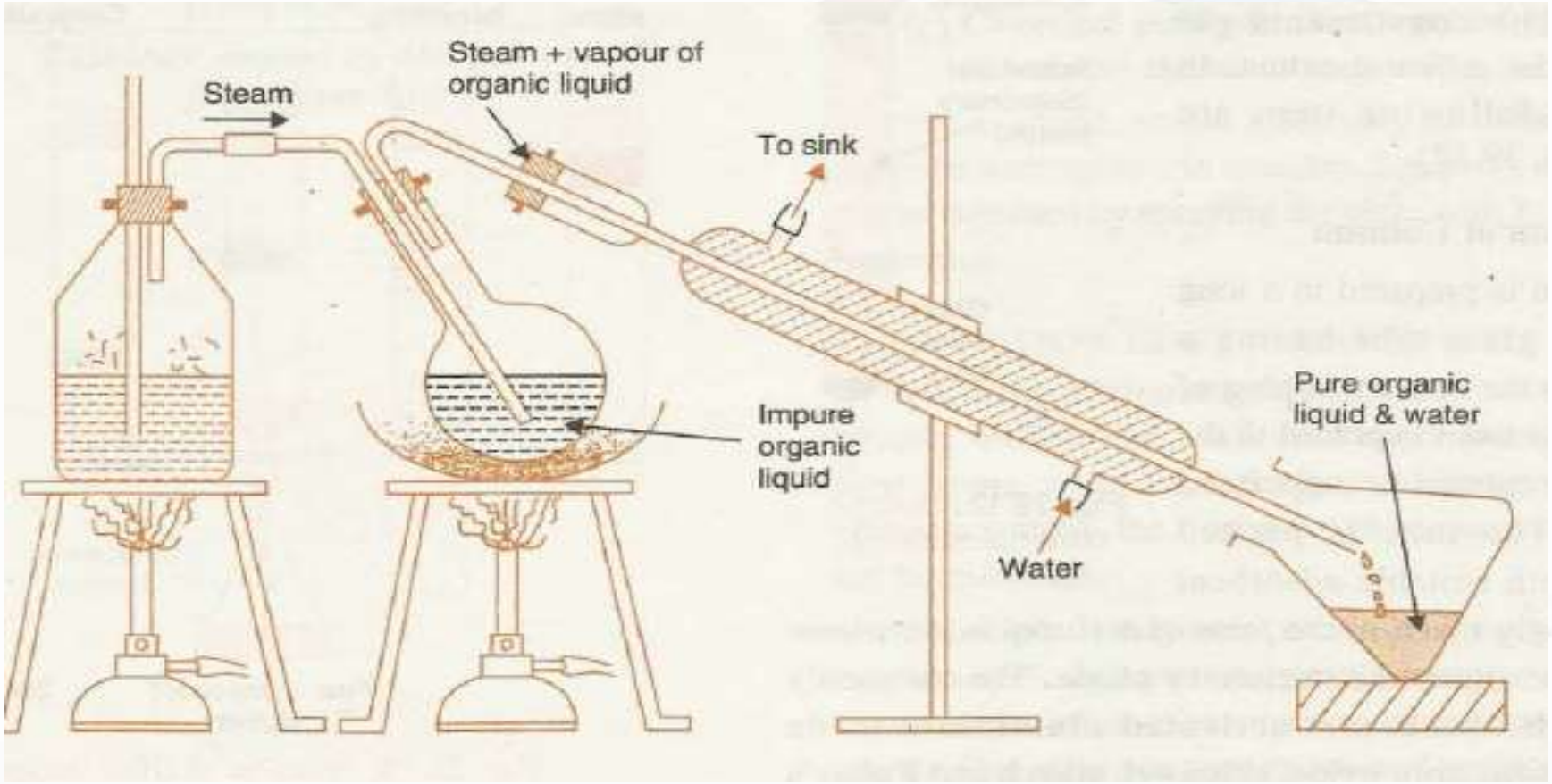
Applications:

Molecular distillation is used for the purification and separation of chemicals of low vapour pressure.

1. Purification of chemicals such as tricresyl phosphate, dibutyl phthalate and dimethyl phthalate.
2. More frequently used in the refining of fixed oils.
3. Vitamin A is separated from fish liver oil. Vitamin's is concentrated by this method from fish liver oils and other vegetable oils.
4. Free fatty acids are distilled at 100°C.
5. Steroids can be obtained between 100°C and 200°C,
6. Triglycerides can be obtained from 200°C onwards.

Proteins and gums will remain as nonvolatile residues. Thus, the above mixture can be separated by molecular distillation.

Steam distillation



Steam distillation is method of distillation carried out with aid of steam.

It is used to separate

- High boiling substances from non-volatile impurities - Separate immiscible liquids

Principle:

A mixture of immiscible liquids begins to boil when sum of their vapour pressure is equal to atmospheric pressure.

In case of mixture of water and turpentine, mixture boils below the boiling point of pure water, though the turpentine boils at a much higher temperature than that of water

Fractional Distillation

- This method is also known as ***rectification,*** **because a part of the vapour is condensed and returned as a liquid**.
- This method is used to separate **miscible volatile liquids**, whose **boiling points are close**, by means of a fractionating column.
- *Fractional distillation* is a process in which vaporisation of liquid mixture gives rise to a mixture of constituents from which the desired one is separated in pure form.

Principle:

- When a liquid mixture is distilled, the partial condensation of the vapour is allowed to occur in a fractionating column.
- In the column, ascending vapour from the still is allowed to come in contact with the condensing vapour returning to the still.
- This results is enrichment of the vapour with the more volatile component.
- By condensing the vapour and reheating the liquid repeatedly, equilibrium between liquid and vapour is set up at each stage, which ultimately results in the separation of a more volatile component

Applications:

Fractional distillation is used for the separation of volatile miscible liquids with near boiling point such as

- Acetone and water
- Chloroform and benzene

Disadvantage:

Fractional distillation cannot be used to separate miscible liquids, which form PURE azeotropic mixtures.

References:

- Sumrahmanyam C.V.S., Setty J.T., Suresh S., Devi K. "PharmaceuticalEngineerining (Unit Operation-II)". Vallabh Prakashan: Delhi. pg no:25-30

- Pharmaceutical Engineering by K.Sanbamurty (New Age International, New Delhi)
- Cooper and Gunn's Tutorial Pharmacy Edited by S.J.Carter (CBS Publishers, Delhi)