Unit-2

Pharmaceutical Engineering

UNIT-II **10 Hours**

Heat Transfer: Objectives, applications & Heat transfer mechanisms. Fourier's law, Heat transfer by conduction, convection & radiation. Heat interchangers & heat exchangers

Evaporation: Objectives, applications and factors influencing evaporation, differences between evaporation and other heat process. principles, construction, working, uses, merits and demerits of Steam jacketed kettle, horizontal tube evaporator, climbing film evaporator, forced circulation evaporator, multiple effect evaporator& Economy of multiple effect evaporator.

Distillation: Basic Principles and methodology of simple distillation, flash distillation, fractional distillation, distillation under reduced pressure, steam distillation & molecular distillation

Heat Transfer: Objectives, applications & Heat transfer mechanisms. Fourier's law, Heat transfer by conduction, convection & radiation. Heat interchangers & heat exchangers

Heat Transfer:

- Heat is a form of energy. As we know matter is made-up of atoms and molecules, atoms in molecules are always in motion represented by translation, rotation and vibration. This motion leads to generation of heat energy.
- "The movement of heat across the border of the system due to a difference in temperature between the system and its surroundings."

Objectives:

- Formulate basic equation for heat transfer problems.
- Calculate heat transfer between objects with simple geometries.
- Recognize basic heat transfer mechanism and apply appropriate methods for quantification.
- Evaluate the relative contributions of different modes of heat transfer.
- Perform an energy balance to determine temperature and heat flux.
- Apply heat transfer principles to design and size to evaluate performance of heat exchangers

Applications:

- Evaporation: Heat is supplied in order to convert a liquid into a vapour.
- Distillation: Heat is supplied to the liquid mixture for separation of individual vapour component.
- Drying: For drying the wet granules and other solids.
- Crystallization: Saturated solution is heated to bring out super saturation, which promotes crystallization of drugs.
- Sterilization: Autoclaves are used with steam as a heating medium.
- Manufacturing of bulk drugs and dosage forms: Principles of heat transfer is of significance in manufacturing of various bulk drugs, excipients and dosage form.

Heat transfer mechanisms:

Heat can travel from one place to another in several ways. The different modes of heat transfer include:

- Conduction
- Convection
- Radiation

Conduction: (Heat transfer through direct contact between two objects)

- Conduction is the most fundamental mechanism of heat transfer. It occurs when heat is transferred from one atom or molecule to another through direct contact.

- The rate of heat transfer by conduction is proportional to the temperature difference between the two objects and the thermal conductivity of the material.

Convection: (Heat transfer through the movement of a fluid.)

- Convection is the heat transfer that occurs due to the movement of a fluid. When a fluid is heated, it expands and becomes less dense.
- This causes the fluid to rise, and cooler fluid to sink.
- This process creates a current of fluid that carries heat from the hot region to the cold region.

Radiation: (Heat transfer through electromagnetic waves.)

- Radiation is the heat transfer that occurs through electromagnetic waves. All objects emit electromagnetic waves, and the intensity of the waves emitted is proportional to the temperature of the object. When an object absorbs electromagnetic waves, it gains heat.

Fourier's law:

- The law of heat conduction is also known as Fourier's law. It states that "the time rate of heat transfer through a material is proportional to the negative gradient in the temperature and to the area.
- The Fourier's equation of heat conduction is expressed as equation:

$$Q = -\mathrm{k} \times \mathrm{A}\frac{\mathrm{dT}}{dx}$$

Where:
Q- Rate of Change of heat transfer
A= Area of Wall
dT= Change in temperature
dx= Thickness of wall
-k= Constant (proportionally constant)

Note: The negative sign in Fourier's equation indicates that the heat flow is in the direction of negative gradient temperature and that serves to make heat flow positive.

Heat transfer by conduction, convection & radiation.

Conduction:

- The process of transmission of energy from one particle of the medium to another with the particles being in direct contact with each other.

Convection:

- The movement of fluid molecules from higher temperature regions to lower temperature regions.

Radiation;

- Radiant heats is present in some or other form in our daily lives. Thermal radiations are referred to as radiant heat.
- Thermal radiation is generated by the emission of electromagnetic waves. These waves carry away the energy from the emitting body. Radiation takes place through a vacuum or transparent medium which can be either solid or liquid.

Heat Exchangers and Heat Interchangers:

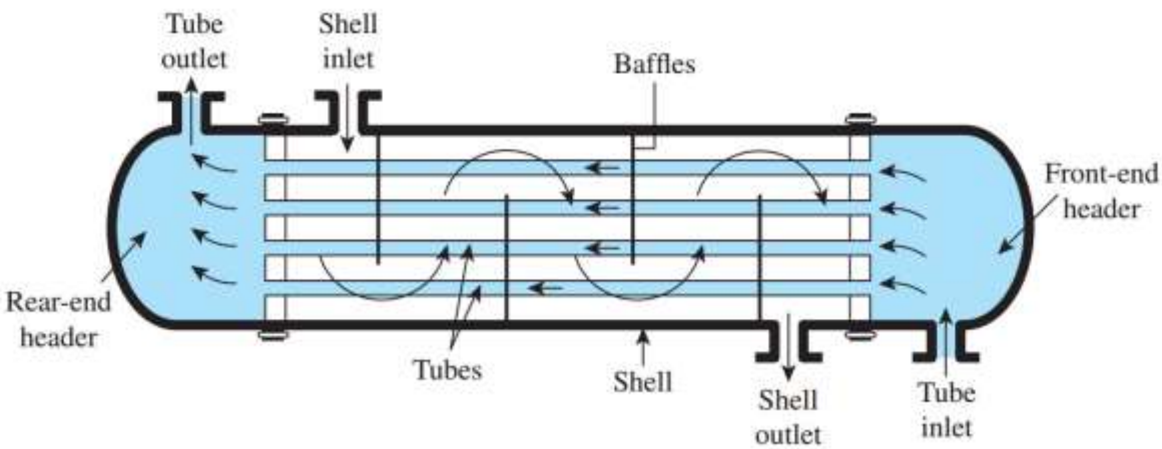
- Heat exchangers are devices that transfer heat between two fluids without mixing them. The fluids can be liquids, gases, or solids.

- Heat exchangers are used in a wide variety of industries, including power generation, chemical processing, and food processing.

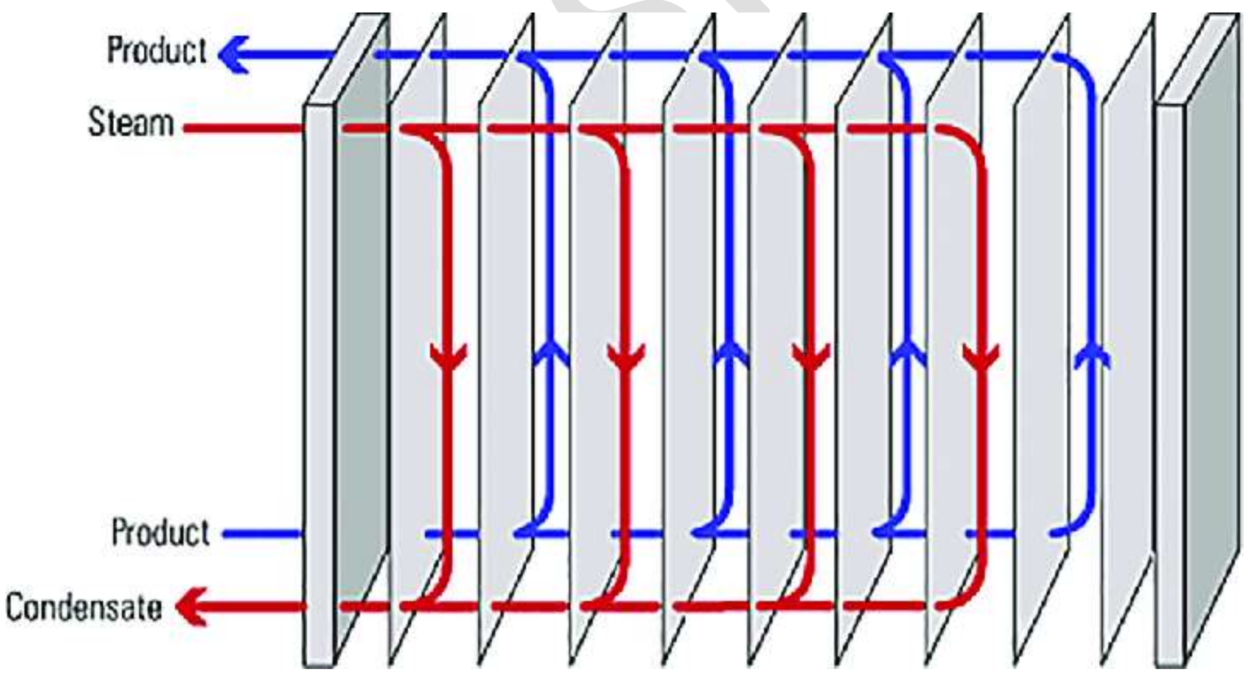
Types of Heat Exchangers:

There are many different types of heat exchangers, but the two most common types are shell-and-tube heat exchangers and plate heat exchangers.

- Shell-and-tube heat exchangers (SPHEs) are the most common type of heat exchanger. They consist of a shell, which contains a number of tubes. The hot fluid flows through the tubes, and the cold fluid flows around the tubes. The tubes are typically made of copper or steel, and the shell is made of steel or stainless steel.



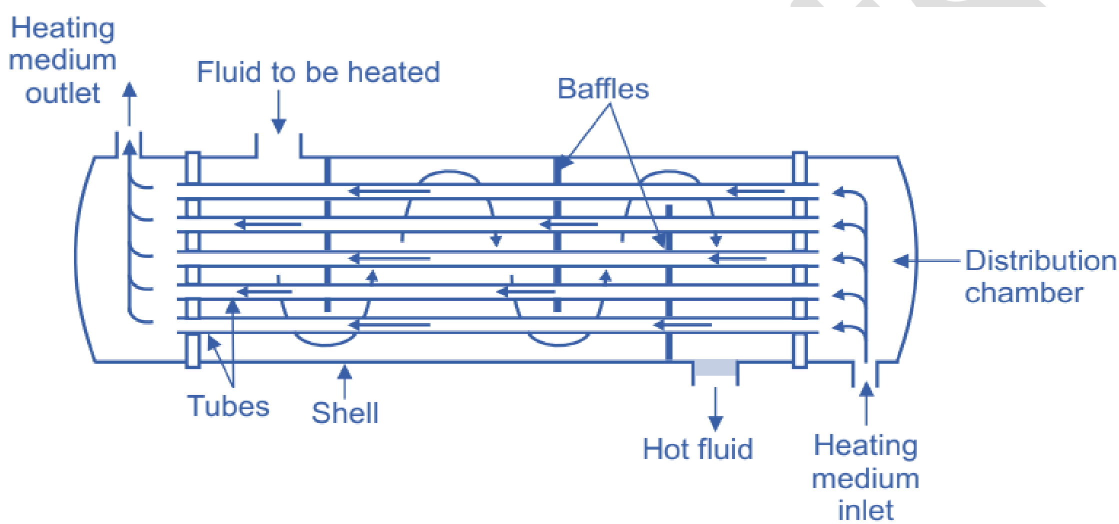
- Plate heat exchangers (PHEs) are a type of heat exchanger that uses a series of plates to transfer heat between the fluids. The plates are made of a thin metal, such as stainless steel or titanium. The fluids flow on opposite sides of the plates, and the heat is transferred through the metal walls of the plates.



Heat Interchangers:

- Heat interchangers are a type of heat exchanger that is used to transfer heat between a fluid and a solid. The solid is typically a metal, which is heated or cooled by the fluid.

- Heat interchangers are used in a variety of applications, such as air conditioning systems, refrigeration systems, and power plants.



Classification of Heat Exchangers:
Heat exchangers are classified according to the following criteria:

- Flow arrangement: The flow arrangement of the fluids can be parallel, counterflow, or crossflow. In parallel flow, the fluids flow in the same direction. In counterflow, the fluids flow in opposite directions. In crossflow, the fluids flow at right angles to each other.
- Number of passes: The number of passes refers to the number of times the fluids flow through the heat exchanger. A single-pass heat exchanger has only one pass for each fluid. A multipass heat exchanger has multiple passes for each fluid.
- Construction materials: The construction materials of a heat exchanger are determined by the fluids that will be used in the exchanger. For example, if the fluids are corrosive, the heat exchanger will need to be made of a corrosion-resistant material, such as stainless steel.

Evaporation: Objectives, applications and factors influencing evaporation, differences between evaporation and other heat process. principles, construction, working, uses, merits and demerits of Steam jacketed kettle, horizontal tube evaporator, climbing film evaporator, forced circulation evaporator, multiple effect evaporator& Economy of multiple effect evaporator.

Evaporation:

- Evaporation is a surface phenomenon, wherein mass transfer takes place from the surface.
- It is the process of vaporization of a liquid. In this process, liquid state of a substance is changing to a gaseous state due to an increase in temperature and/or pressure.

Objectives:

- Getting concentrated product
- An aqueous solution must be removed of its water
- To develop drinking water by evaporating seawater
- It is used to obtain solid-free water for use in chemical boilers.

Applications:

- Making galenical preparations involves evaporation.
- A biological product (such as insulin) is made by evaporating a liquid.
- Plasma and serum (blood products) are prepared through evaporation.
- Antibiotics, enzymes, and hormones are prepared by evaporation.
- To make demineralized drinking water, evaporation is used.

Factors Influencing Evaporation:

1. Surface area

- The rate of evaporation is directly proportional to the surface area of the vessel exposed to evaporation.

2. Agitation

- Agitation is necessary for evaporation.

3. Vapour pressure

- Liquids with a low boiling point evaporate quickly due to high vapor pressure.

4. Type of product required

- The selection of the method and apparatus to be used for evaporation depends upon the type of product required. For example, an open pan produces liquid or dry concentrate while a film evaporator yields liquid concentrate.

5. Density

- As the density increases, the rate of evaporation decreases.

6. Time of evaporation

- Exposure to a relatively high temperature for a short time may be less destructive of the active ingredients than a lower temperature with exposure for a longer period. Film evaporators have used a fairly high temperature but the exposure time is very short.

7. Economic factors

- When selecting the method and apparatus the economic factors are important. Evaporators are designed to give maximum heat transfer to liquid.

8. Moisture content

- Some drug constituents decompose more rapidly in the presence of moisture, especially at the raised temperature. Hence, evaporation should be carried out at a low controlled temperature.

9. Temprature:

- The evaporation rate is directly related to the temperature. As the temperature increases, the rate of evaporation also increases. Because the temperature is rising, the water molecules begin to move faster.
- As the kinetic energy is acquired by the molecules and they escape from the surface to the vapor state. The heat is needed to provide the latent heat of vaporization, and in general, the rate of evaporation is controlled by the heat transfer rate.

- The rate of heat transfer depends on the temperature gradient.

Differences between Evaporation and Other Heat Process:

	Evaporation	Other Heating Processes
1.	The residue is concentrared liquid	Drying: Residue is solid
2.	Transforming liquid into a gas (no need to separation)	Distillation: It is the process of separation (separation is compulsory)
3.	Evaporation does not produce bubbles.	Crystallization: purpose of concentrating solution to get ceystals
4.	Transforming liquid into a gas (no need to separation)	Sublimation: Transition of solid into gas
5.	It happened also on room temprature	Boiling: Process of evaporation of a liquid at the boiling point of the liquid.

Steam Jacketed Kettle (Evaporating Pan):

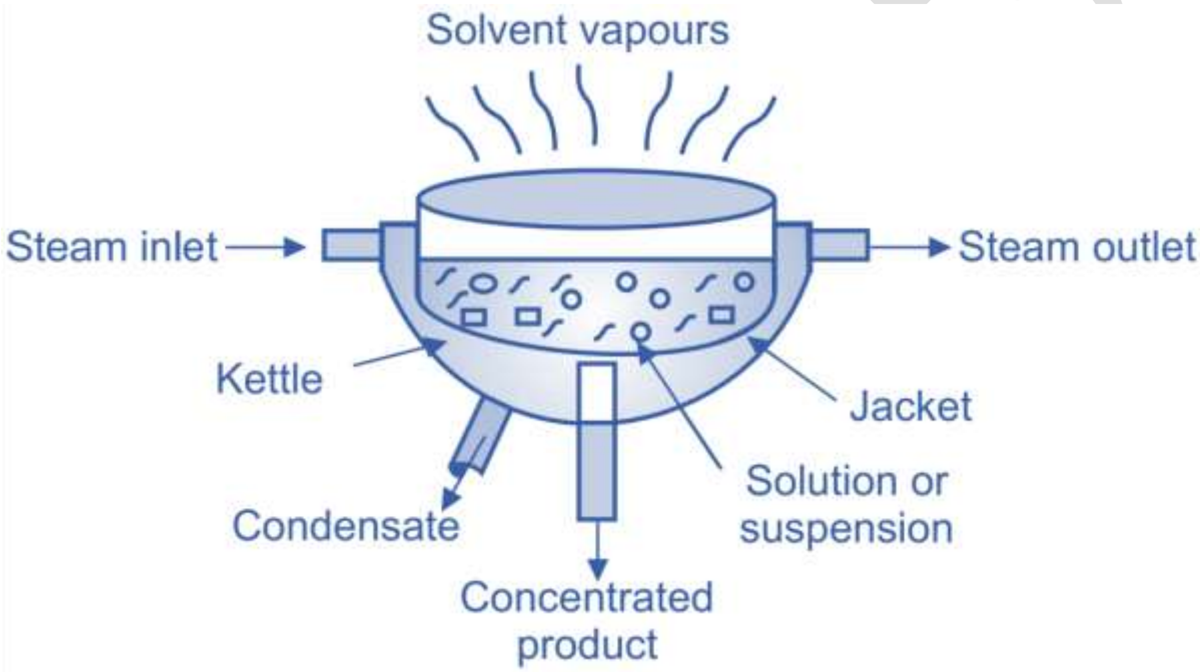
Principles:

The mechanism involved in this evaporation process is conduction and convection so that the heat is transferred by this mechanism to the extract.

Construction:

- In a steam evaporating pan, there is an outer pan called a jacket enclosed by an inner pan called a kettle.
- A space is created between the two pans through which steam passes. The kettle is constructed from several kinds of metal.
- The superior conductivity of copper makes it a great choice for the kettle. Some copper gets dissolved in the preparation if acidic preparation evaporates in the copper kettle.

- To prevent this, tinned copper kettles are used for acidic preparation. In addition to using iron for the jacket's construction, it has a low conductivity.
- Wearing out of iron causes rusting, which is why the iron jacket is tinned or enameled on the inside to prevent it.
- A steam inlet and an inlet for non-condensed gases can be found near the top of the jacket.
- The condensation is drained from the jacket through the bottom outlet. There is an outlet provided at the bottom for discharging product.



Working:

- It consists of a pot filled with evaporable solution, steam fed through the intake and condensate spouting out of the outlet, which heats the content.
- Mechanical stirring is required for larger volumes, while manual stirring is required for smaller volumes. Evaporation begins rapidly at the beginning.
- Vapour must be able to escape the chamber where evaporation occurs in order to prevent fog from forming. The room is fitted with fans to avoid condensation and to speed up evaporation. Fixed or tilted kettles are available.
- Up to 90 liters of capacity can be tilted in a kettle. A bottom outlet allows caught residue to be collected.

Uses:

- Evaporating pan is suitable for concentrating aqueous liquids.
- It is suitable for concentrating thermostable liquors, for example, liquorices extracts.

Merits:

- Evaporating pan is constructed both for small scale and large scale batch operations.
- It is a simple in construction and easy to operate, clean and maintain.
- Its cost of installation and maintenance is low.
- Stirring the contents in pan and removal of the products is easy

Demerits:

- Natural circulation of the product makes poor heat transfer.
- Deposition of solid may cause decomposition of the product.
- It is not suitable for the concentration of thermolabile materials.
- It has no provision to operate under a reduced pressure.
- No provision to recollect the costly organic solvents.

Horizontal Tube Evaporator:

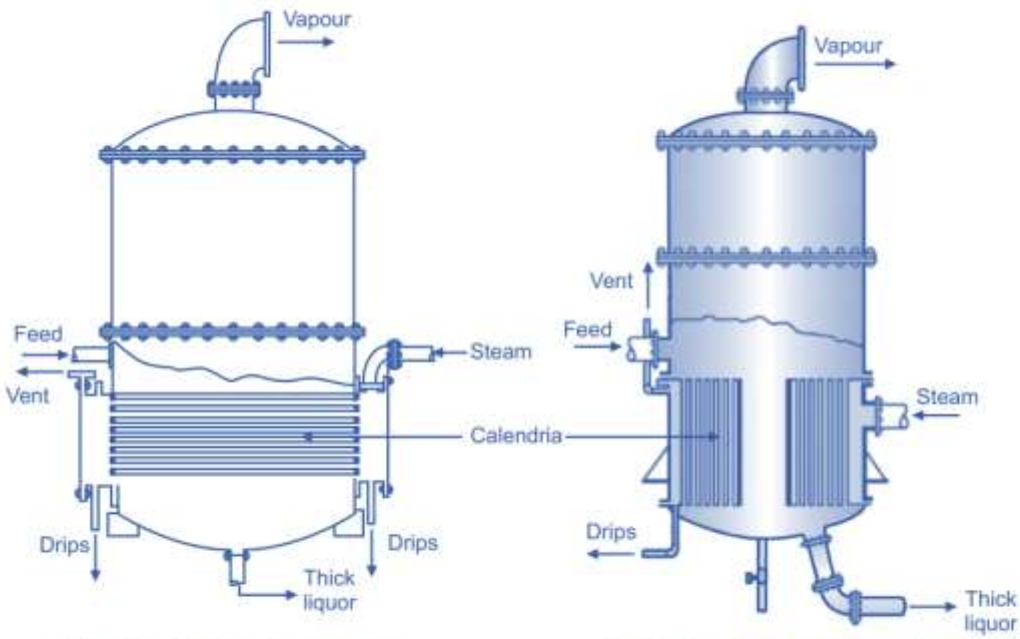
Principles

- The principle mechanism involved in this type of evaporator is that steam is passed through tubes arranged horizontally.
- Heating causes evaporation of the feed outside the tubes discharging concentrate at the bottom and vapours passed out from the outlet at top.

Construction:

- There are two general types of horizontal tube evaporators: rectangular or circular, constructed of stainless steel, aluminium, nickel, carbon, spellerized iron tubing, lead covered copper, and special bronze.
- Between two steam chests, tubes are attached to an arc by means of arc fasteners. Tube sheets are countersunk into four-hole packing plates, which press conical gaskets around tube ends into countersunk holes.

- Providing a secure seal, the tube sheets can be replaced easily. A cylindrical chamber with an exterior diameter of 1 to 3 meters and a height of 2.5 to 4.5 meters has horizontal tubes of 2 to 3 cm diameter running across the bottom.
- The tubes of a vertical tube evaporator are arranged vertically in calendria.
- An evaporator calendria includes many smaller diameter tubes in which the liquid is concentrated as it rises or falls.



(a) Horizontal Tube Evaporator (b) Vertical Tube Evaporator

Working:

- A horizontal tube evaporator warms the liquid surrounding the tube in the bottom of the evaporator body with steam fed through horizontal tubes from a steam chest.
- During the condensed gas process, the steam follows a definite path to the opposite steam chest, where it is withdrawn from the opposite steam chest.
- It is determined by the distribution, size, and shape of the heating surface in the liquid compartment that determines the velocity and path of circulation of the liquid.

Uses:

- It is used in the manufacture of the cascara extract.
- It is used in the manufacture of caustic soda.
- It is used in the manufacture of salts.

Merits:

- It has low cost per unit of heating surfaces.
- It has extreme simplicity.
- Easy renewal of heating surfaces.
- Sectional construction with low maintenance cost.
- Ease of operation.

Demerits:

- Cleaning and maintenance is difficult when compared with steam jacketed kettle.
- It may be used only when rigorous boiling can be obtained with natural circulation.
- It is not suitable for viscous liquids.
- It is not suitable when scaling or salting liquids are involved.

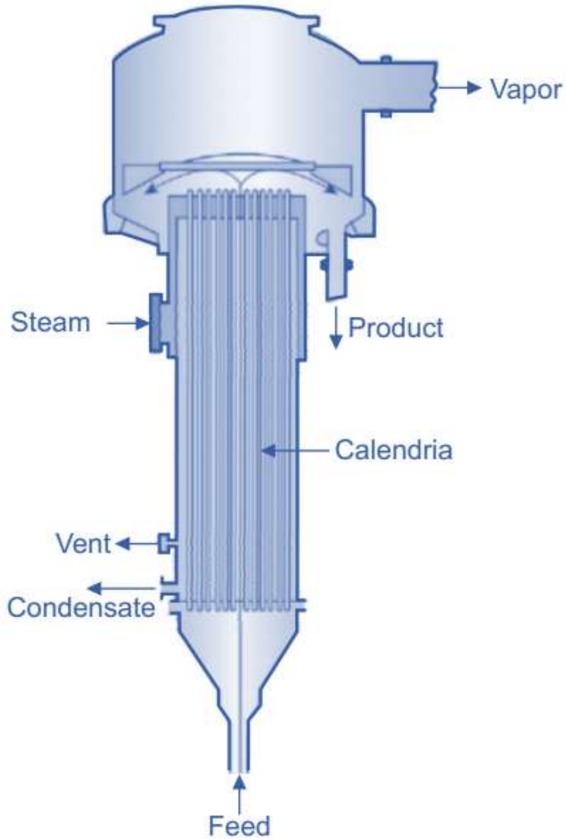
Climbing film evaporator:

Principles:

- The theory of climbing film evaporator is that the ascending force of the steam produced during the boiling causes liquid and vapours to flow upwards in parallel flow.
- At the same time the production of vapour increases and the product is pressed as a thin film on the walls of the tubes, and the liquid rises upwards.

Construction:

- A Rising Film Evaporator (RFE) is a vertical shell and tube heat exchanger with a vapour liquid separator mounted at the top.
- Tubes carrying the steam internally are placed vertically in the bottom of the cylindrical evaporator chamber.
- The length of the boiling tubes is typically not more than 23 ft (7m). This type of unit is known as the Roberts evaporator



Working:

- The preheated liquid feed (to be evaporated) is introduced from the bottom of the unit. The height of the liquid column is maintained low, i.e., 0.6 or 1.2 metres above the bottom tube sheet. Steam enters into the spaces outside the tubes through the inlet. Heat is transferred to the liquor through the walls of the tubes.
- The liquid becomes vapour and forms smaller bubbles, which tend to fuse to larger bubbles.
- These are of the width of the tubes, thereby the bubbles trap a part of the liquid (slug) on its way up in the tubes. As more vapour is formed, the slug of liquid is blown up in the tubes facilitating the liquid to spread like a film over the walls.
- This film of liquid continues to vaporise rapidly. Finally, the mixture of liquid concentrate and vapour eject at a high velocity from the top of the tubes.
- The entrainment separator not only prevents entrainment but also acts as a foam breaker. The vapor leaves from the top, while the concentrate is collected from the bottom.

Uses:

- A climbing film is used for effluent treatment.
- It can be used in production of polymers and for juice concentration and food processing.
- It is used in thermal desalination of sea water.
- It has many applications in pharmaceuticals especially for solvent recovery.
- It is used as reboilers for distillation columns.

Merits:

- It is easier to clean the tubes.
- Thermosiphon action eliminates the need for circulation pump.
- Trace quantities of suspended particles in the feed are tolerated.
- Can operate under reasonable vacuum.
- Multiple effect arrangement provides steam economy.
- It is relatively inexpensive evaporator.

Demerits:

- It requires large floor space and is heavy.
- It has poor heat transfer at low temperature differences.
- Not suitable for thermolabile materials.
- It evaporates products of low viscosity and have minimal fouling tendencies

Forced Circulation Evaporator:

Principles:

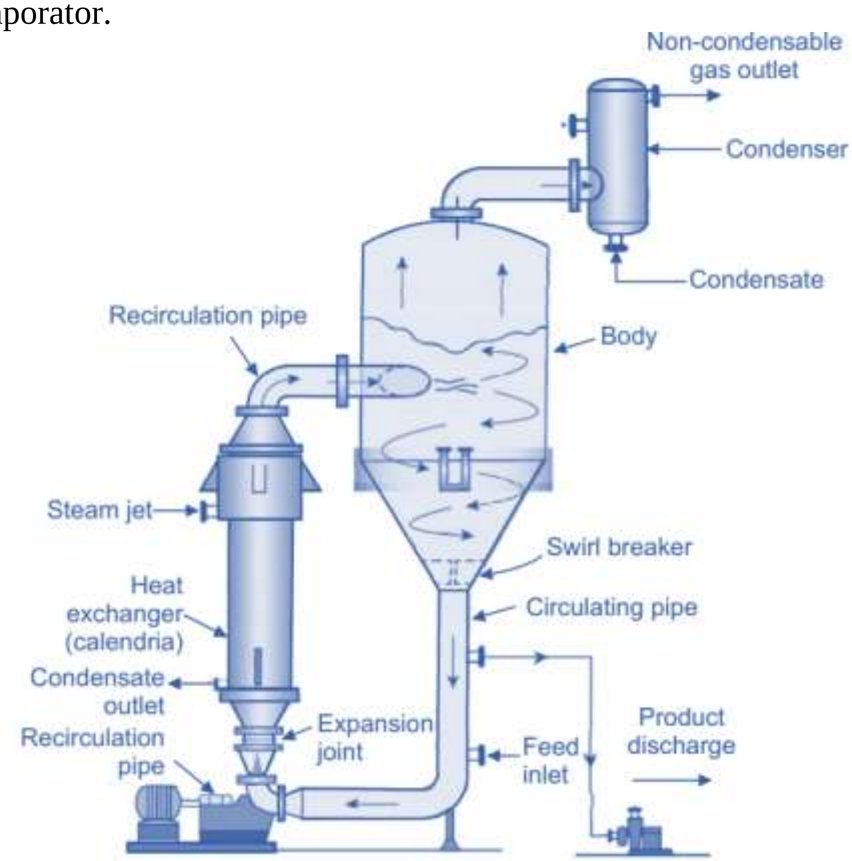
- In forced circulation evaporator liquid is circulated through the tubes at high pressure by means of a pump. Hence boiling does not take places because boiling point is elevated.
- Forced circulation of the liquids also creates some form of agitation

Construction:

- In these types of evaporators pumps are fitted to circulate the contents heating in it. The forced circulation evaporator consists of steam jacketed tubes held between two tube sheets.
- The tube measures 0.1 m inside diameter and 2.5 m long. The parts of the tubes projects in to the vapor head which consists of a deflector. The vapour head is

connected to a return pipe which runs downwards and enters in to the inlet of a pump.

- The liquid is circulated by means of a pump. As it is under pressure in the tubes the boiling point is raised (but no boling take places) and it enters into the body of the evaporator.



Working:

- Steam is introduced in to the calendria. Pump sends the liquid to the tube with a positive velocity. As the liquid move up through the tube it gets heated and begins to boil.
- As result the vapour and liquid mixture rushes out of the tubes at a high velocity. This mixture strikes the deflector in a manner that effective separation of liquid and vapour takes place.
- The vapour enters the cyclone separator and leaves the equipment. The concentrated liquid is circulated through the pump for further evaporation.
- Finally the concentrated product is collected at the bottom from discharge outlet.

Uses:

- Forced circulation evaporator is used to concentrate thermolabile substance solutions.
- It is used for concentration of insulin and liver extracts.

- It is well suited for crystallizing operation where crystals are to be suspended at all times.
- It is used for effluent treatment in pharmaceutical industries.

Merits:

- There is a rapid movement of liquid due to high heat transfer coefficient. Salting, scaling and fouling are not possible due to forced circulation.
- These evaporators are suitable for thermolabile substances because of rapid evaporation.
- It is suitable for the viscous preparation because pumping mechanism is used.
- These evaporators are also used for liquids with high solids content.
- Circulation evaporators are fairly compact and are easy to clean and operate.

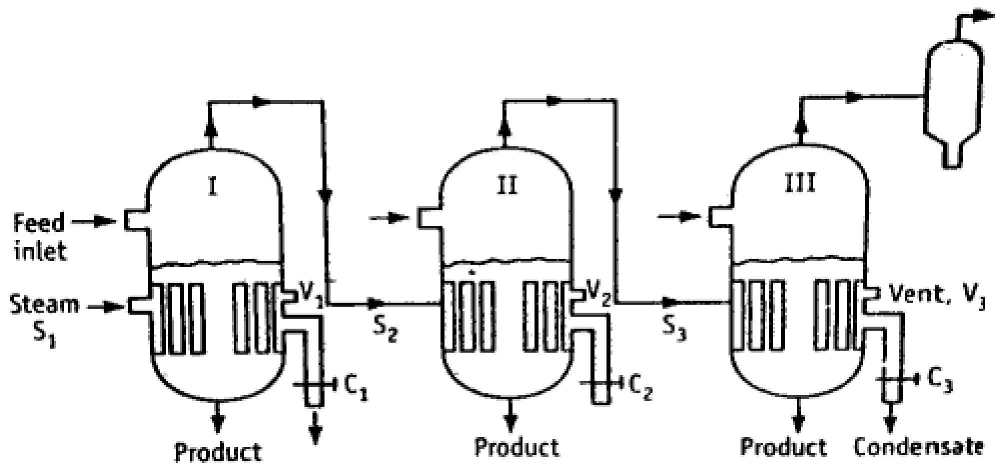
Demerits:

- The hold up time of liquids is high.
- The equipments is expensive as well as power requirement is high.
- It has high maintenance costs.

Multiple Effect Evaporator

Construction:

- Three evaporators, i.e., triple effect evaporator, are used to construct the multiple-effect evaporator.
- Other aspects of the vertical tube evaporator's construction are unchanged.
- Using vapor from the first evaporator to heat the second evaporator is illustrated in Figure. As with the 2nd evaporator, the 3rd evaporator is also heated by the vapor from the 2nd evaporator vacuum pump.
- The last evaporator is connected to a vacuum pump.



Working:

- An evaporator with three effects (3-stages) is a tube-type forced circulation evaporator in which strong steam is used for the first effect to evaporate the solvent from the feed.
- In the second effect, the vaporized solvent is used to evaporate the feed at atmospheric pressure. In the third effect, the evaporation of the concentrating feed from the second stage is used to evaporate solvent from the second stage.

- The evaporated solvent from the third effect is finally condensed with cooling water on the other side of the steam condenser. In all three processes, condensate is collected in condensate receiving tanks.
- The pure solvent in this condensate can be reused in the subsequent process.

Uses:

- The vapor pressure falls, the second effect of a multiple-effect evaporator lowers the boiling point.
- Multi-effect evaporation refers to this reusing of latent heat from vapor molecules.

Merits:

- Operation on a large scale and in continuous mode is possible.
- As compared to single effects, it is highly economic.
- There can be attached about five evaporators.
- Utilizing input energy as efficiently as possible (steam economy)
- Requires less energy to run the entire system

Demerits:

- It has the disadvantage that it requires a lot of headroom.
- In a falling film evaporator, the tubes have a greater pressure drop.
- Temperature sensitivities can be affected by hydrostatic heads located at the bottom of tubes.
- Costly
- Time consume

Economy of multiple effect evaporator.

- The economy of multiple effect evaporators is greater than single effect because vapors generated in the first effect are used as a heating medium for the second effect and so on. Thus, efficiency increases as steam consumption decreases and evaporative capacity increases.
- The performance of multiple effect evaporators is measured in terms of its capacity and economy.
- The capacity of the evaporator is defined as the number of kilograms of water vaporized per hour.
- Its economy is the number of kilograms of water vaporized from all the effects per kilogram of steam used.
- For a single effect evaporator, the steam economy is approximately about 0.8 (<1). The capacity is about n-times that of a single effect evaporator, and the economy is about 0.8 n for n-effect evaporators. However, pumps, interconnecting pipes, and valves required for the transfer of liquid from one effect to another effect increases both the equipment and the operating costs.

Distillation: Basic Principles and methodology of simple distillation, flash distillation, fractional distillation, distillation under reduced pressure, steam distillation & molecular distillation.

Distillation:

- Distillation is a process of separating the components or substances from a liquid mixture by using selective boiling and condensation.
- A typical distillation apparatus consists of a container for the liquid mixture (the boiler), a heating source, a condenser, and a receiving flask.
- The liquid mixture is heated in the boiler until it boils.
- The vapors rise up the condenser, where they are cooled and condensed back into liquid form. The condensed liquid is collected in the receiving flask.

Simple Distillation:

- Simple distillation is the most basic type of distillation.
- Simple distillation is a unit operation in which two liquids with different boiling points are separated.

Principle:

- This process can be repeated until all the fractions of the original mixture are separated. In order for simple distillation to perform, the two liquids' boiling points must have a difference of at least 25 °C (or about 77 °F).
- Simple distillation is a process of heating and cooling liquids in order to separate and purify them. As the liquid being distilled is heated, the vapours that form are richest in the component of the mixture that boils at the lowest temperature.
- Purified component boils, and thus turns into vapours, over a relatively small temperature range (2 or 3 °C).

Working of Simple Distillation

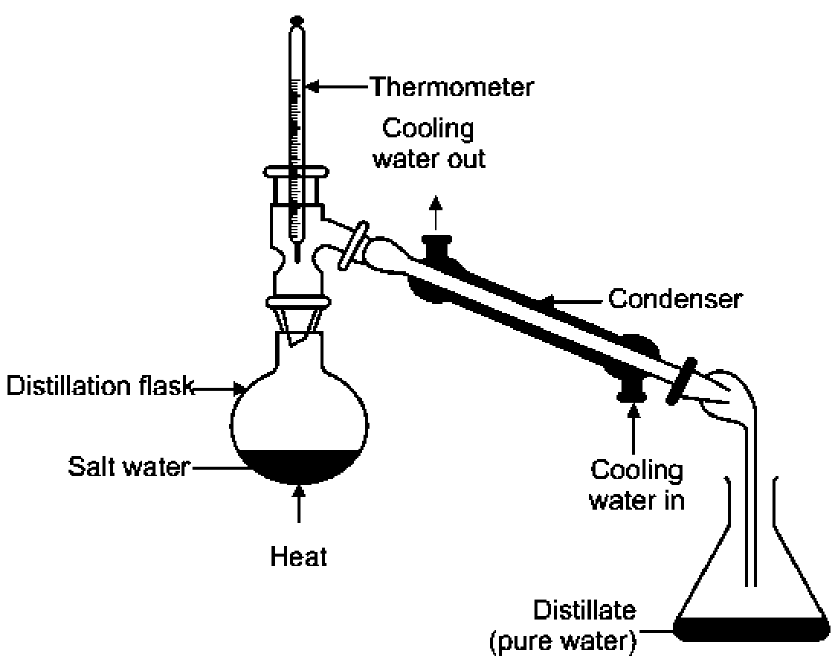
1. Calibration of thermometer: Calibration can be done by placing the thermometer in an ice bath of distilled water. Allow the thermometer to reach thermal equilibrium. Now remove from ice water and place it in a beaker of boiling distilled water and again allow it to reach thermal equilibrium. If the temperatures measured does deviate from the expected values by more than two degrees then use it for recording temperature in the distillation process.

2. Filling the distillation flask: The flask is filled with not more than two-thirds of its volumes to have sufficient space above the liquid surface so that when boiling begins the liquid will not be propelled into the condenser. This is important in the viewpoint of the purity of the distillate. Porcelain chips should be placed in the distillation flask to prevent superheating of the liquid and to cause a more controlled boiling, eliminating the possibility of liquid bumping into the condenser.

3. Heating the distillation flask: The distillation flask is heated slowly until the liquid begins to boil. The vapours rise through the neck of the distillation flask and pass through the condenser and condense and drip into the collection receiver, Fig.1. Generally, the rate of distillation is approximately 20 drops per minute. Distillation must occur slowly enough that all the vapours condense to liquid in the condenser. Many organic compounds are flammable and if vapours pass through the condenser without condensing, they may ignite as they come in contact with the heat source.

4. Condensation of vapours: As the distillate begins to drop from the condenser, the temperature changes steadily. When it is stable, a new receiver is used to collect all the drops that form over a two to a three-degree range of temperature. As the temperature begins to rise further, a third receiver is used to collect the distillate. This process is repeated; using a new receiver every time the temperature stabilizes or begins changing until all of the distillate has been collected in discrete fractions. All fractions of the distillate should be saved until it is shown that the desired compound has been effectively separated by distillation.

Diagram of Simple Distillation:



Advantages:

- It is simple, cheap, easy and economic method.
- It requires less energy.
- This process requires single run and thus is comparatively faster.

Disadvantages:

- The final product may contain impurities.
- Azeotropic mixtures cannot be separated by simple distillation.
- Not suitable for mixtures containing thermolabile components.
- The volume of mixture should be not more than 2/3rd of the container.

Applications:

- Simple distillation is primarily used for production of distilled water.
- Many volatile oils are separated by simple distillation.
- It is also used in the separation of organic solvents from mixtures.
- It is used to separate non-volatile components from volatile ones.
- It is used in preparing pharmaceutical spirits

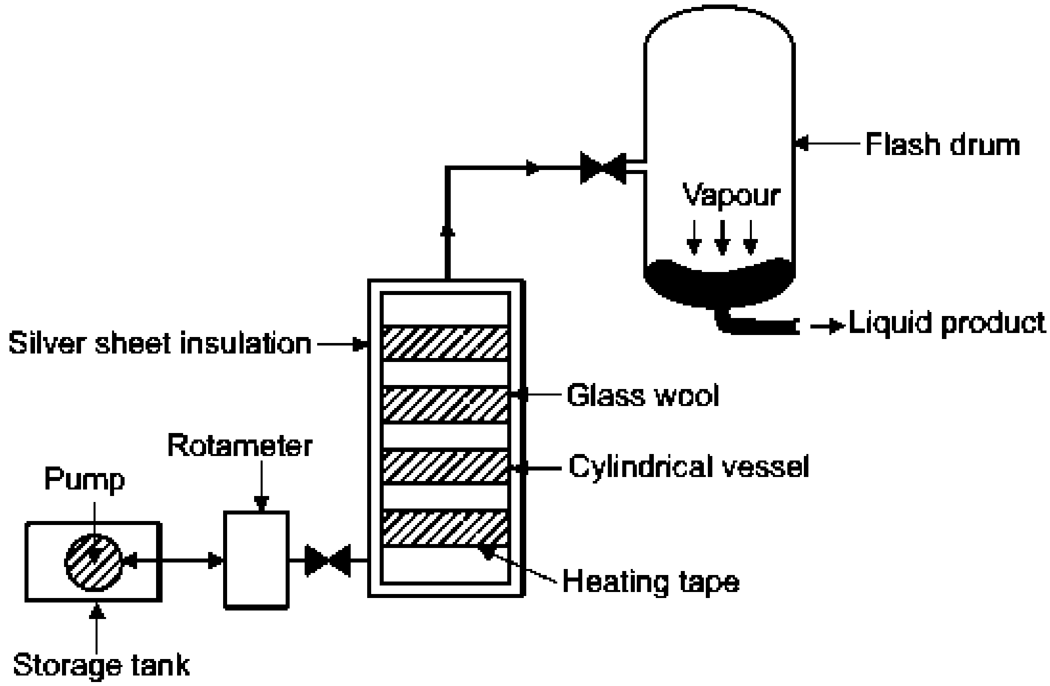
Flash distillation

Principles:

Flash distillation is a separation process that is based on flash vaporization. In this process, a hot liquid mixture passes from a high-pressure zone to a low-pressure zone, causing it to suddenly vaporize. The reduced pressure lowers the boiling point of the liquid, leading to its vaporization. This process is used to separate components of a mixture based on their boiling points.

Methodology of Flash Distillation:

- The Flash distillation unit consists of a pump attached to the feed tank from where it pumps feed at high pressure. This unit has a heating chamber through which the liquid mixture carrying pipe passes.
- The chamber is insulated to avoid heat loss during operation and maintain the desired temperature.
- There is a pressure control valve fitted between the heating chamber and the flash drum.
- The other end of the pipe directly opens into the flash drum. In single-stage flash distillation unit, liquid outlet is provided at the bottom. In case of multi stage distillation, the liquid and vapours are taken to the next unit for further distillation.
- When designing a flash system it is important to provide enough disengaging space in the flash drum. Flash drum can also be designed as cyclone separators.



Application

- It is used in the petroleum industry for refining crude oil.
- It is used in the desalination of ocean water by multi-stage flash distillation.
- It can also be used for separation of heptane from octane.

Advantages:

- It is a continuous process.
- The equipment is smaller than the multi-stage flash distillation.
- The operating costs are low compared to multi-stage flash distillation.

Disadvantages:

- It is not effective in separating components of comparable volatility.
- It is not suitable for two-component systems.

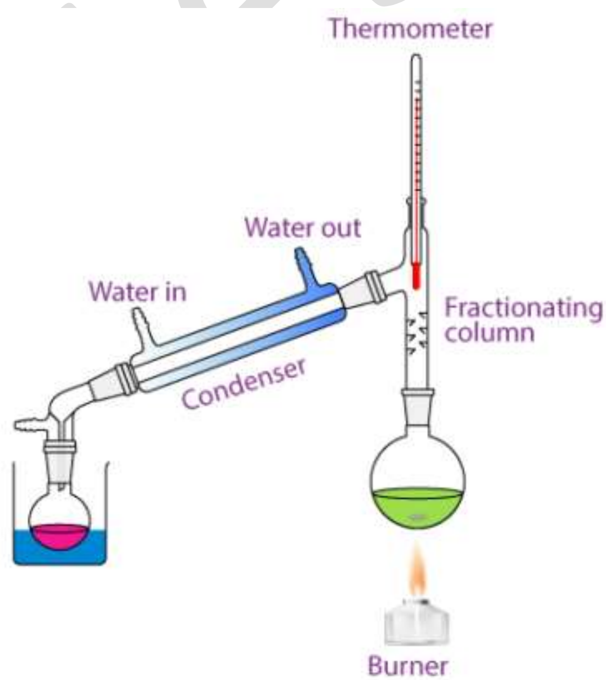
Fractional Distillation:

Principles:

- The basic principle of this type of distillation is that different liquids boil and evaporate at different temperatures. When the mixture is heated, the substance with the lower boiling point starts to boil first and convert into vapors

Methodology of fractional distillation:

- The methodology of fractional distillation involves repeated distillations and condensations. The process requires a few fractional distillation apparatuses, including a distilling flask, condenser, receiver, fractionating column, thermometer, and heat source.
- After setting up the apparatus, a mixture of two miscible liquids A and B is taken, where A has more volatility than substance B.
- The solution is added into the distilling flask while the fractionating column is connected at the tip of the flask.
- Heat is applied, which increases the temperature slowly. The mixture then starts to boil and vapors start rising in the flask. The vapors are from the volatile component A.
- The vapors then start moving through the fractionating column into the condenser where it is cooled down to form a liquid which is collected in the receiver.
- Throughout the process, vaporization and condensation take place repeatedly until the two mixtures are separated completely



Distillation Under Reduced Pressure:

Vacuum Distillation

Principle:

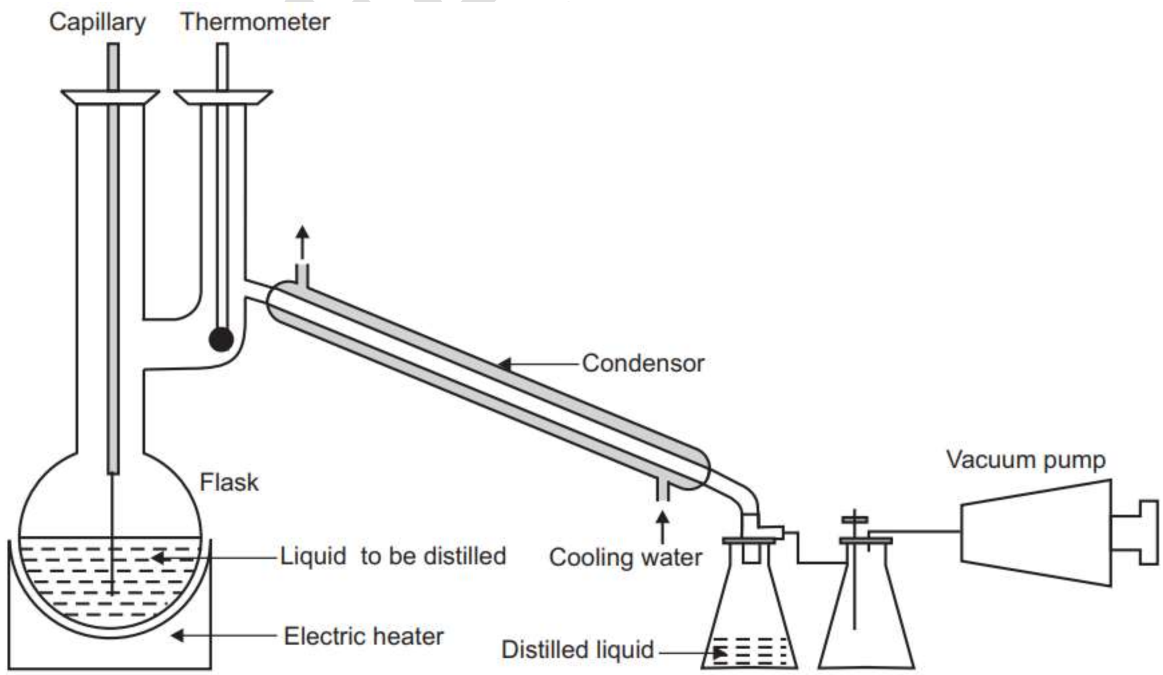
- This distillation method works on the principle that boiling occurs when the vapour pressure of a liquid exceeds the ambient pressure. Vacuum distillation is used with or without heating the mixture.

Construction:

- The vacuum distillation unit consists of a distillation column, condensing distillate, and reboiler.
- Vacuum pumps and vacuum regulators are added to distillation columns to maintain the column at a vacuum. Many mixtures can be distilled at much more economical temperatures with the use of these vacuum distillation columns.

Methodology (Working):

A vacuum distillation is also called as low temperature distillation. The target product in this distillation could either be the remaining product, the distilled product or a purified product. The vacuum pressure associated with a distillation depends on the product to be distilled. For example, volatile substances like those used in oil refineries are likely to undergo
vacuum distillations at above 1 Torr, perhaps 20-50 mmHg.



Applications:

- The products of normal distillation are further distilled using vacuum distillation.
- The high boiling point hydrocarbons, such as lubricants and waxes, are separated at economical temperatures.
- Vacuum distillation is also used in the separation of sensitive organic chemicals and recovery of organic solvents.

Advantages:

- Vacuum distillation reduces the number of stages needed in distillation.
- The product output per day is very high.
  It requires lower temperatures at lower pressures.
- It has very high capacity to handle liquid mixtures giving high yield and highest purity.
- Vacuum distillation can improve a separation by prevention of product degradation because of reduced pressure leading to lower tower bottoms temperatures.

Disadvantages:

- High energy costs of vacuum pumps.
- Pressure and energy losses due to any leaks or cracks.
- Large column diameters needed for the process to be efficient.

Steam Distillation:

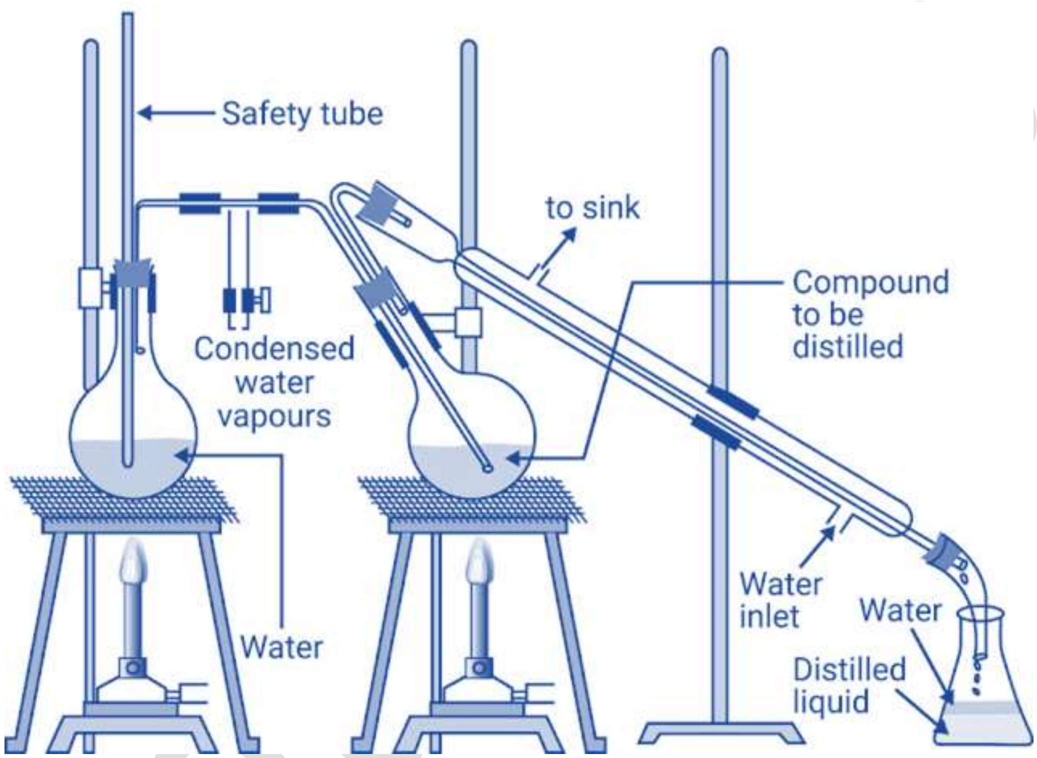
Principle:

- The principle behind steam distillation is a way of separating miscible liquid based on their volatilities.
- The boiling point of the products is so minimized that it permits the constituents to get vaporized.

Construction:

- The general assembly of laboratory scale vacuum distillation units.
- It consists of a metallic can to generate steam.
- This metallic can is fitted with a cork having two holes.
- A safety tube is passed through one of the holes and a bent tube is passed through the other.
- The safety tube should reach almost the bottom of the flask.
- In case the pressure inside the steam generator becomes too much, water will be forced out of the safety tube and the pressure will be released.
- When steam starts coming out from the safety tube; it indicates that the steam can is almost empty.

- The other end of the bent tube is connected to the flask containing non-aqueous liquid (for example, turpentine) through a rubber bung.
- This tube should reach almost the bottom of the flask.
- The flask is connected to a condenser through a delivery tube as shown in the Fig..
- The condenser is connected to a receiver flask using an adaptor.
- The steam can and flask are heated by a suitable method.



Methodology (Working):

- The non-aqueous liquid or oil to be purified or extracted is placed in the flask.
- A small quantity of water is added to it.
- Fill the steam can with water.
- The steam can and the flask are heated simultaneously.
- The steam generated in the steam can pass to the flask through the bent tube.
- A uniform flow of steam passes through the boiling mixture in the flask.
- The mixture gets heated.
- The steam carries the volatile oil and passes into the condenser.
- Cool water passes through the condenser.
- The condensed liquid is collected into the receiver as distillate.
- The distillate is the mixture of immiscible liquids.
- The immiscible liquids form separate layers which can be easily separated using a separating flask.

Advantages:

- Useful for thermolabile substances.

Disadvantages:

- Not suitable when the liquids in the mixture are reactive.

Molecular Distillation:

Principle:

- Molecular distillation is considered as the safest mode of separation and to purify the thermally unstable molecules and related compounds with low volatility and elevated boiling points.
- The process distinguishes the short residence time in the zone of the molecular evaporator exposed to heat and low operating temperature due to vacuum in the space of distillation.

Mean free path: It is defined as the average distance travelled by single molecules in a straight line without any collision. It is expressed as

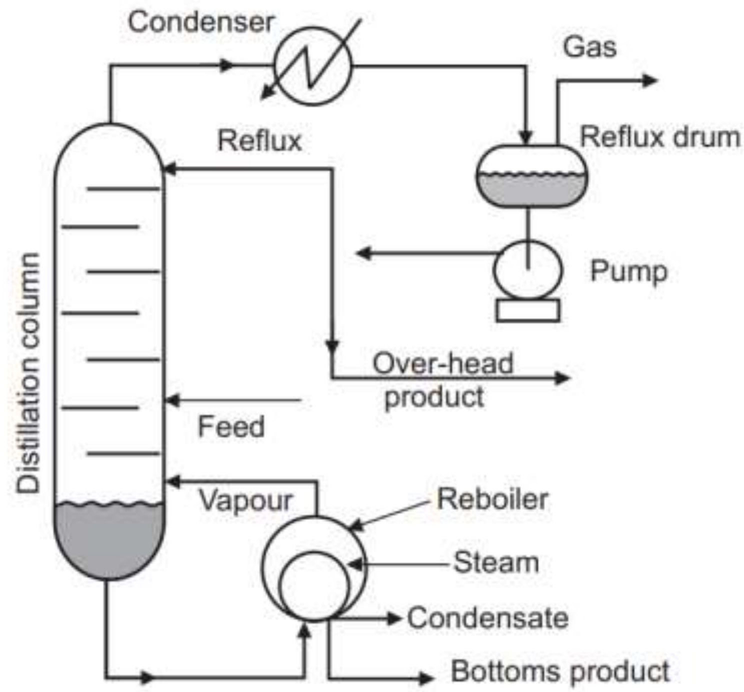
$$\lambda = \eta\sqrt{\frac{3}{p\rho}}$$

Where,

$\lambda = Path\ length$
p= Vapour pressure
ρ= density
$\eta = viscosity$

Construction of Molecular Distillation:

- A simple molecular distillation has a unit that is placed on a hot surface. The distillate moves a very short distance before it gets condensed.
- If the substance is not too viscous, it will drip from the point on the glass condensing surface and run down to the receiving point.
- The sophisticated apparatus with a different design will have the liquid distilled down on a heated surface close to the condenser.
- The movement of a film prevents a build-up of non-volatile materials on the surface of the material to be distilled as this might cause the distillation to stop.



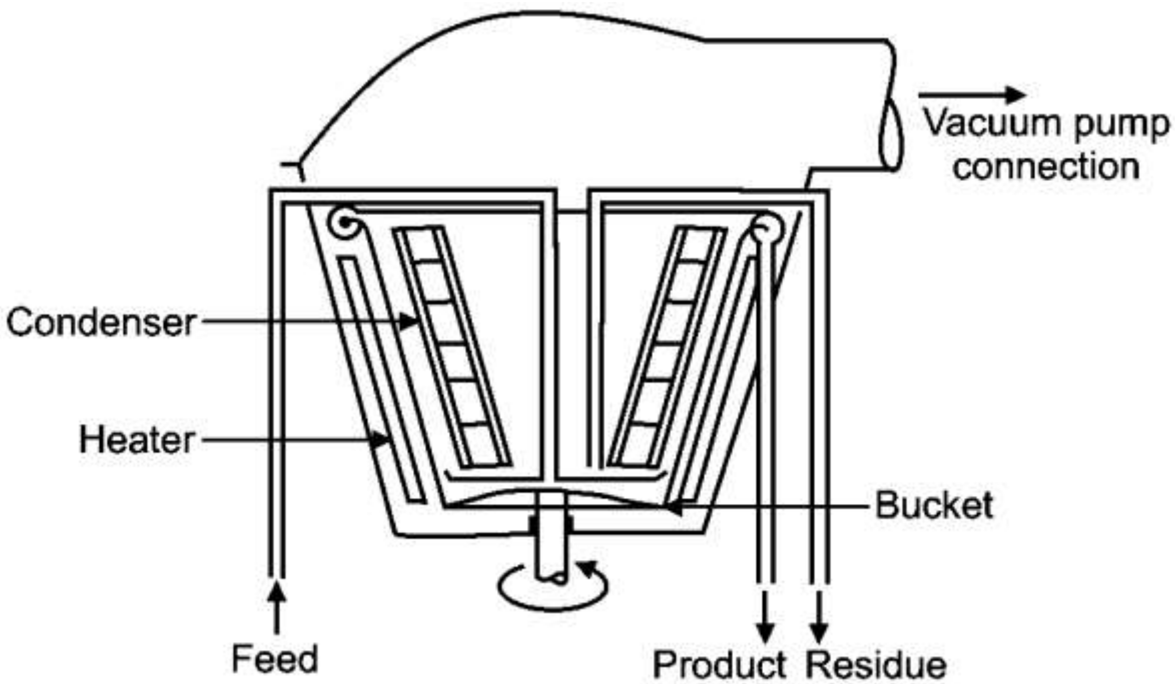
Working of Molecular Distillation:

- The molecular distillation process is carried out at very low pressure so that the distance between the hot and condensing surface is less than the mean free path of the molecules. Each of the units is a single-stage but has several units in series.
- Molecular distillation is applied to thermally sensitive high molecular weight materials.
- The contact times in commercial units may be low as 0.001 seconds. The film thickness is of the order of 0.05 – 0.1 mm.
- In vacuum operations, air ingress is very much possible, whereas in pressure operations vapor emissions are likely to occur.
- The distillation process is inherently hazardous with flammables and the presence of a huge volume of flammables in reboilers, in column internals and adjacent piping pose huge explosion hazards in these distillation units.
- The concentration gradient between the top and bottom of the column has a bearing on safety.
- The concentration of impurities in the column can lead to hazards.

Centrifugal Molecular Distillation:

- Vacuum is applied at the center of vessel then rotate on high speed
- **Now feed is introduced into centre of vessels through feed inlet.**
- Due to high speed rotation centrifugal force create, due to centrifugal force liquid move outward over the surface of bucket and form a film.

- The vapour molecules moves its mean free path then it strikes the condenser then product inlet.
- Residue is collected through residue outlet and re-circulated through feed



Advantages of Molecular Distillation

- Toxicity: Avoids the problem of toxicity of solvents used as the separating agent.
- Thermal stability: Minimizes losses due to thermal decomposition.
- Continuous process: It can be used in a continuous feed process to harvest distillate without having to break the vacuum.
- Stability: The vacuum allows oils to be processed at minimal temperatures, reducing the risk of oxidative damage.
- Purity: Separating the oil's components by weight allows contaminants to be reduced far below industry standards.

Disadvantages of Molecular Distillation

- Cost: The cost for this complicated technology is relatively high.
- Natural form: The starting natural triglyceride form is lost in the distillation process.

Applications of Molecular Distillation

- It is used for the separation of vitamins and polyunsaturated fatty acids.
- Molecular distillation is used industrially for the purification of oils.
- It is also used to enrich borage oil in γ-linolenic acid (GLA) and recover tocopherols from deodorizer distillate of soybean oil.

- It can be used for the production of synthetic and natural vitamin E,
- Capsicum red pigment containing 1% to 2% of the solvent can be separated after two-stage molecular distillation.
- It is used for the separation of strong spices like volatile substances.
- It is used for highly heat-sensitive materials.