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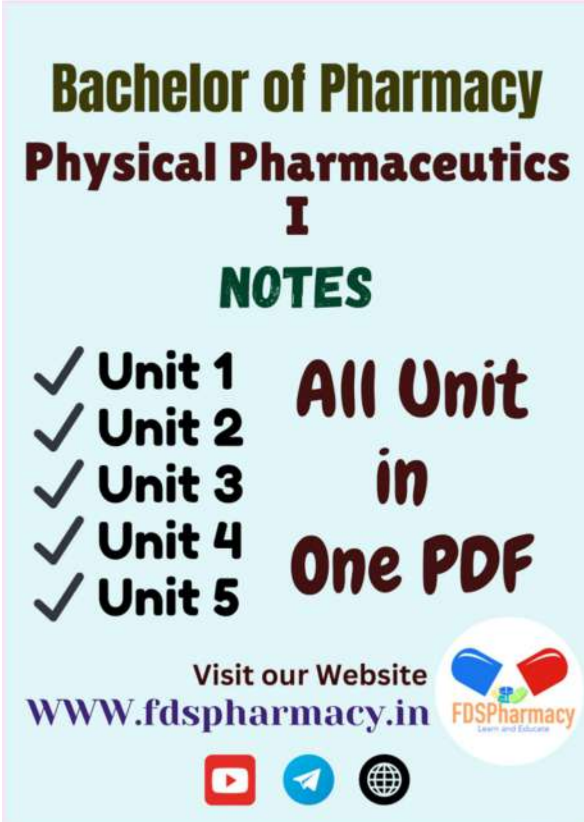
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PHARMACEUTICAL ENGINEERING

UNIT 2

TOPIC :

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

Distillation

Distillation is a physical separation process used to separate components of a liquid mixture based on differences in their boiling points.

- It is commonly used in industries, laboratories, and daily applications for purifying liquids, recovering solvents, or separating liquid mixtures.

Principle of Distillation

- Every liquid has a specific boiling point.
- When a mixture is heated:
  - The component with the lower boiling point vaporizes first.
  - The vapor is then passed through a condenser, where it is cooled and converted back into liquid form (distillate).
- By controlling heating and condensation, different components of the mixture can be separated effectively.

Applications of Distillation

- Purification of chemicals – removal of impurities.
- Alcohol production – separation and concentration of ethanol.
- Isolation of active ingredients in pharmaceutical preparations.
- Extraction of aromatic compounds (e.g., volatile oils).
- Separation of crude oil into useful fractions like petrol, kerosene, diesel, etc.

Types of Distillation

Different methods of distillation are used depending on the nature of liquid mixture and the difference in boiling points of components. Common types include:

1. Simple Distillation
2. Flash Distillation
3. Fractional Distillation
4. Distillation under Reduced Pressure (Vacuum Distillation)
5. Steam Distillation
6. Molecular Distillation

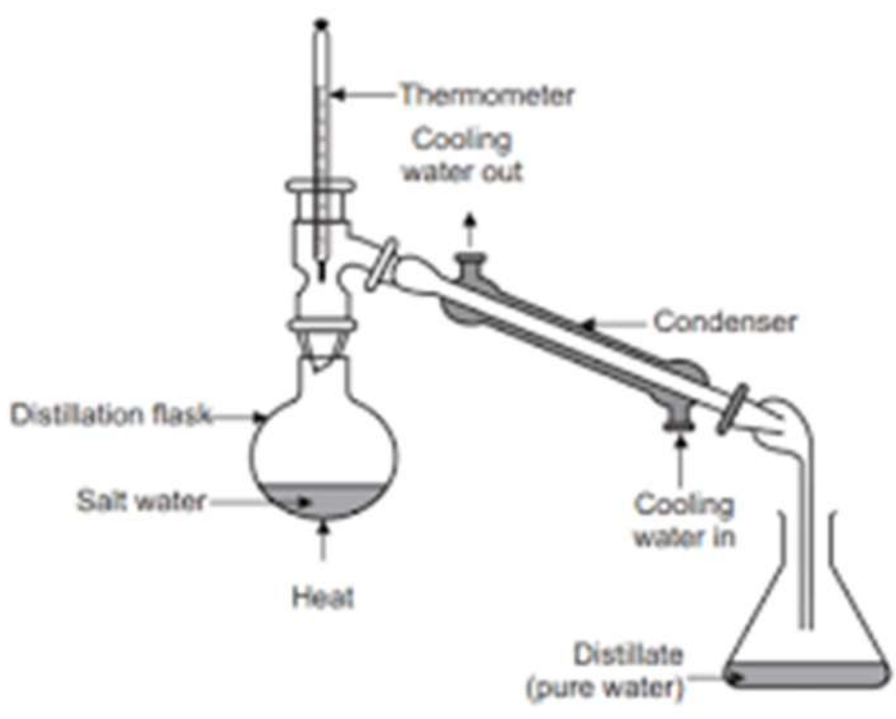
Simple Distillation

Principle

- Simple distillation works on the difference in boiling points of liquid components.
- The liquid with the lower boiling point vaporizes first when the mixture is heated, leaving behind higher-boiling components.
- The vapor is then cooled in a condenser and collected as liquid in a receiving flask.
- This method is effective only when the boiling point difference between components is at least 20–30 °C.

Construction

- Distillation Flask – Holds the liquid mixture.
- Heat Source – Provides heat to boil the mixture.
- Condenser – Cools the vapors into liquid.
- Receiving Flask – Collects the condensed liquid (distillate).
- Thermometer – Monitors the vapor temperature for accurate separation.



Working

1. The liquid mixture is placed in the distillation flask and heated.
2. The component with the lower boiling point vaporizes first.
3. Vapors pass through the condenser, where they are cooled and condense into liquid.
4. The condensed liquid is collected in the receiving flask.
5. The process continues, separating components based on their boiling points.

Advantages

- Simple and easy setup.
- Cost-effective method.
- Requires minimum energy compared to other methods.

Disadvantages

- Not suitable for mixtures with close boiling points.
- Inefficient for heat-sensitive substances (may decompose).

Applications

- Water purification (removal of dissolved salts/impurities).
- Purification of organic solvents.
- Alcohol distillation (e.g., ethanol).

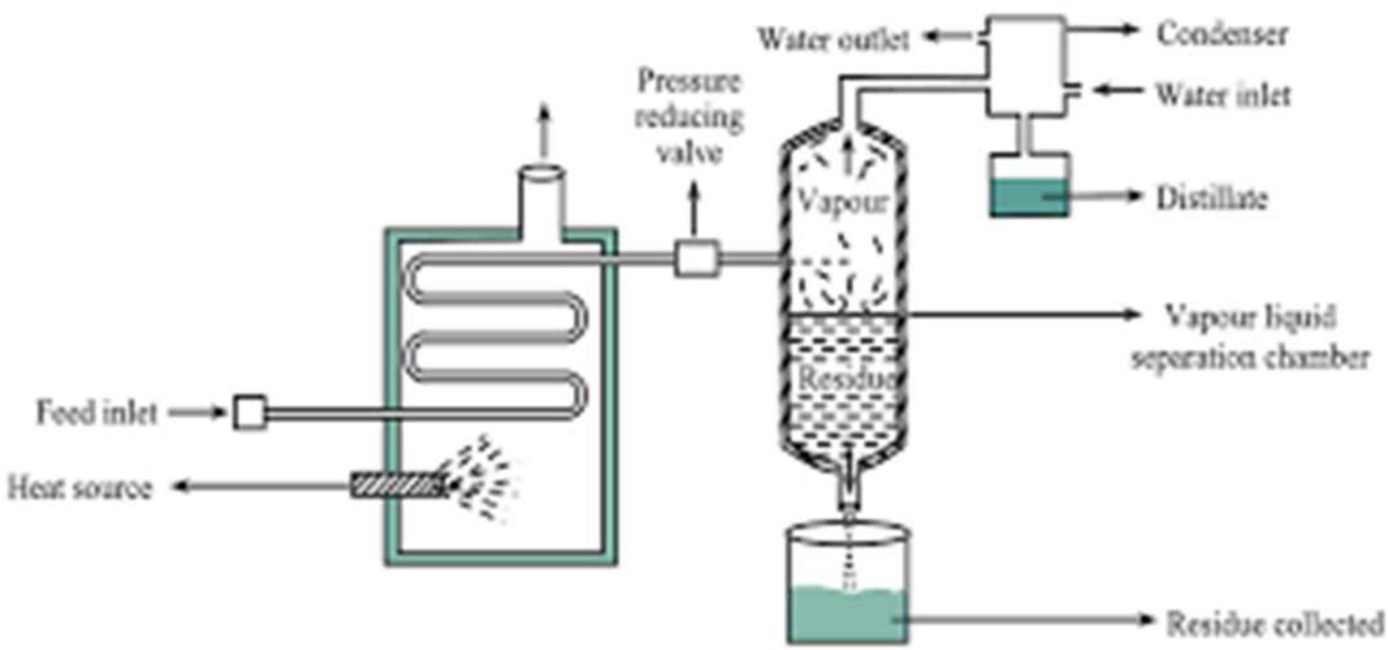
Flash Distillation

Principle

- Flash distillation is based on the partial vaporization of a liquid mixture when it is subjected to sudden pressure reduction or heating.
- When a liquid mixture is flashed (heated or pressure dropped), only a portion vaporizes instantly.
- The vapor formed becomes enriched with the more volatile component, while the remaining liquid is richer in the less volatile component.
- The system quickly reaches vapor–liquid equilibrium.

Construction

- Feed Tank – Holds the liquid mixture.
- Heater/Heat Exchanger – Heats the feed mixture to the desired temperature.
- Flash Chamber – Large vessel where sudden vaporization (flashing) occurs when the heated liquid enters at lower pressure.
- Condenser – Cools and condenses the vapor into liquid distillate.
- Receiving Tanks – Collects the condensed vapor (distillate) and the remaining liquid separately.



Working

1. The liquid feed is first heated in a heat exchanger.
2. The heated mixture is then introduced into a flash chamber where pressure is suddenly reduced.
3. Due to this, a portion of the liquid vaporizes instantly (flashing).
4. The vapor formed is richer in the low-boiling component.
5. The vapor is passed through a condenser and collected as distillate.
6. The remaining liquid in the chamber is richer in the high-boiling component and is collected separately.

Advantages

- Simple and continuous operation.
- Economical for large-scale separations.
- Useful for mixtures where only partial separation is needed.

Disadvantages

- Not suitable for mixtures requiring high purity separation.
- Efficiency depends strongly on temperature and pressure control.
- Limited to systems with large volatility differences.

Applications

- Used in petroleum refining for crude oil separation.
- Recovery of solvents in the chemical and pharmaceutical industries.
- Separation of natural gas liquids.
- Useful for separating heat-sensitive substances quickly (less heating time).

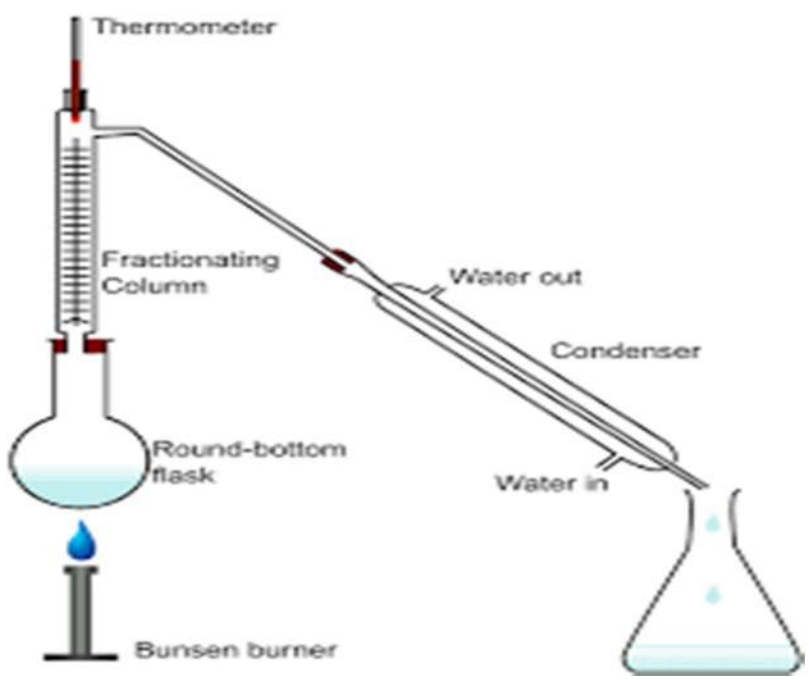
Fractional Distillation

Principle

- Fractional distillation is based on the different boiling points of components in a liquid mixture.
- When the mixture is heated, the component with the lowest boiling point vaporizes first.
- A fractionating column with trays/packing provides repeated condensation and vaporization, allowing better separation even when the boiling points are close.
- Each stage of condensation–vaporization enriches the vapor in the more volatile component, leading to effective separation.

Construction

- Distillation Flask – Contains the liquid mixture to be separated.
- Heat Source – Provides controlled heating.
- Fractionating Column – A long vertical column packed with beads, plates, or glass tubes that allow repeated condensation–vaporization cycles.
- Condenser – Cools the vapor into liquid.
- Receiver Flasks – Collect separated fractions at different boiling points.
- Thermometer – Monitors vapor temperature for accurate fraction collection.



Working

1. The liquid mixture is heated in the distillation flask.
2. Vapors rise into the fractionating column.
3. Inside the column, vapors partially condense and revaporize multiple times.
4. Each cycle enriches the vapor with the more volatile component.
5. Vapors exit the top of the column and are condensed in the condenser.
6. Different components (fractions) are collected in receiver flasks at their respective boiling point ranges.

Advantages

- Can separate mixtures with close boiling points (difference < 25 °C).
- Provides higher purity of separated fractions compared to simple distillation.
- Continuous collection of multiple components.

Disadvantages

- More complex and costly setup.
- Requires more time and energy.
- Not suitable for highly heat-sensitive compounds.

Applications

- Separation of crude oil into petrol, diesel, kerosene, etc.
- Preparation and purification of organic solvents.
- Separation of alcohol–water mixtures.
- Used in pharmaceuticals for isolating and purifying volatile active ingredients.
- Production of essential oils and aromatic compounds.

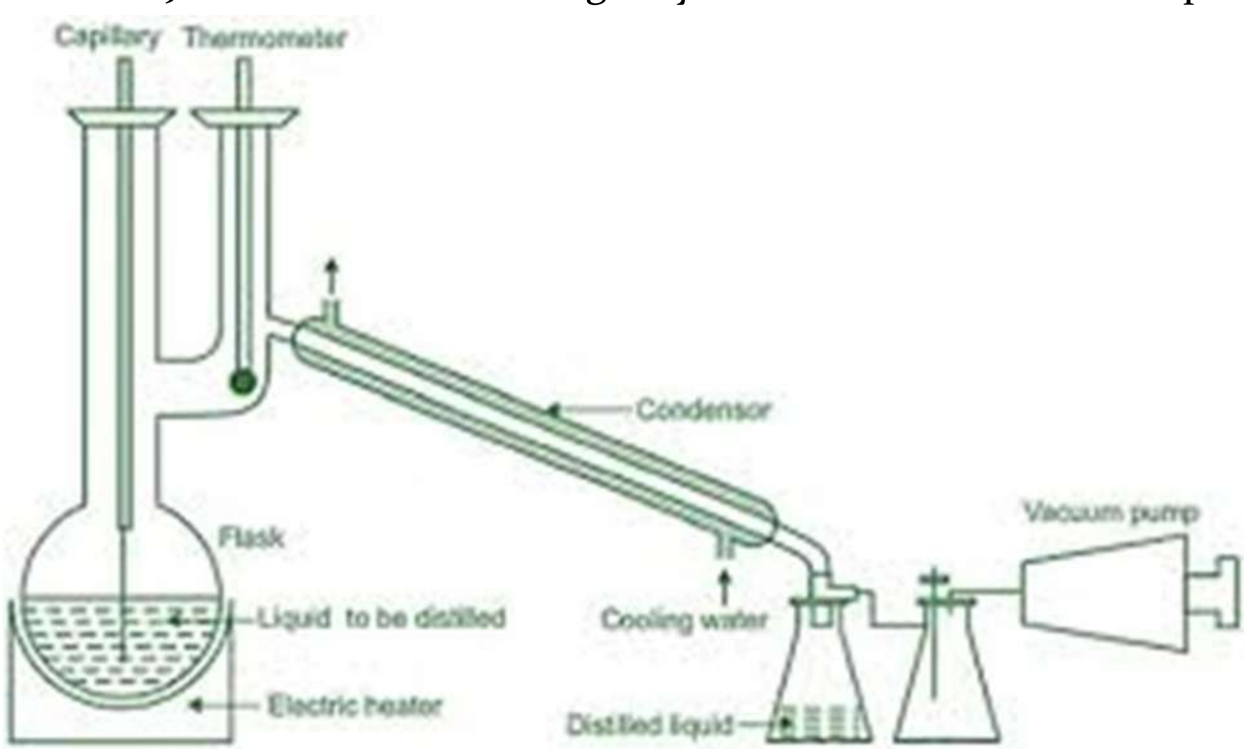
Distillation Under Reduced Pressure (Vacuum Distillation)

Principle

- The boiling point of a liquid decreases when the external pressure is reduced.
- By lowering the pressure (below atmospheric pressure), a liquid can be distilled at a temperature much lower than its normal boiling point.
- This method is especially useful for heat-sensitive and high-boiling liquids that may decompose at high temperatures under normal distillation.

Construction

- Distillation Flask – Contains the liquid mixture.
- Vacuum Pump/Water Aspirator – Reduces the pressure inside the system.
- Condenser – Cools and condenses the vapors.
- Receiver Flask – Collects the distilled liquid under vacuum.
- Manometer/Vacuum Gauge – Measures the pressure inside the apparatus.
- Sealed Joints – Ensure an airtight system to maintain reduced pressure.



Working

1. The liquid mixture is placed in the distillation flask.
2. Pressure inside the system is reduced using a vacuum pump.
3. Due to reduced pressure, the liquid starts to boil at a lower temperature.
4. The vapors pass through the condenser, where they are cooled and converted back into liquid.
5. The distilled liquid is collected in the receiver flask under vacuum.

Advantages

- Prevents decomposition of heat-sensitive substances.
- Allows distillation of liquids with very high boiling points.
- Saves energy as boiling occurs at lower temperatures.

Disadvantages

- Requires special apparatus (vacuum pump, airtight system).
- More expensive and complex than simple distillation.
- Not suitable for very volatile compounds (may escape with vacuum).

Applications

- Purification of pharmaceutical compounds that are heat-sensitive.
- Distillation of glycerin, oils, vitamins, antibiotics, and hormones.
- Industrial separation of high-boiling petroleum fractions.
- Preparation of essential oils and active ingredients that degrade at high temperatures.

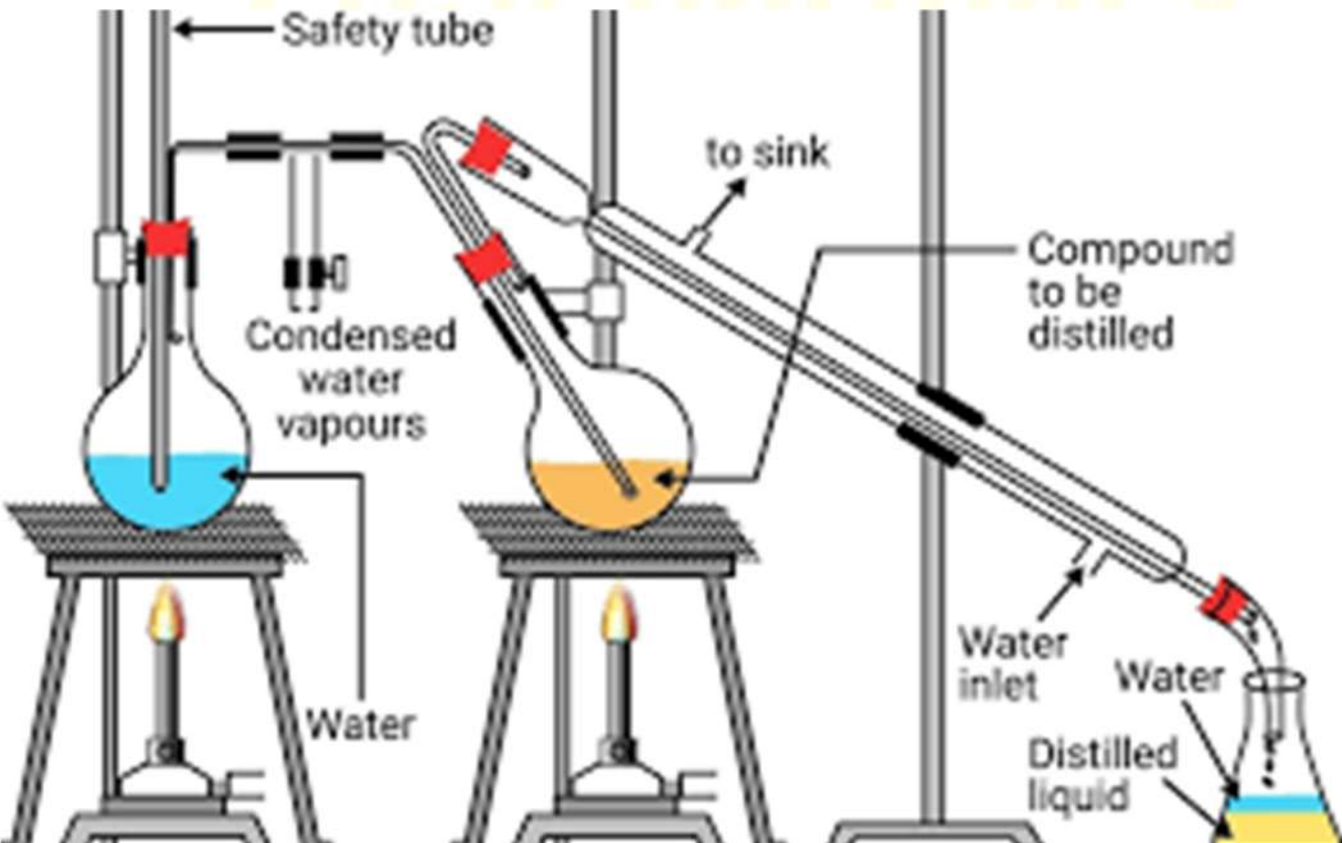
Steam Distillation

Principle

- When steam is passed through immiscible liquids (like water + essential oil), both components exert their own vapor pressures.
- As a result, the mixture boils at a temperature lower than the normal boiling point of either component.
- This prevents decomposition of heat-sensitive substances.

Construction

- Steam Generator – Produces steam.
- Distillation Flask – Contains the immiscible liquid mixture (e.g., plant material + water).
- Condenser – Cools the vapors.
- Receiver Flask – Collects the distillate (mixture of oil + water).
- Separator Funnel – Separates oil layer from water layer (since oils are immiscible with water).



Working

1. Steam is generated in the steam generator and passed into the distillation flask containing the substance to be distilled.
2. The steam lowers the boiling point of the mixture, causing volatile components (essential oils or aromatic compounds) to vaporize along with water.
3. The vapors pass into the condenser, where they are cooled and liquefied.
4. The condensed mixture of water and oil is collected in the receiver flask.
5. Oil is then separated from water using a separating funnel.

Advantages

- Useful for heat-sensitive materials that would decompose at higher temperatures.
- Requires lower energy compared to normal distillation of high-boiling substances.
- Provides a clean and efficient method for separating volatile oils.

Disadvantages

- Limited to substances that are immiscible with water and volatile in steam.
- Not suitable for compounds that are soluble in water.
- Separation process requires additional steps (like separating funnel).

Applications

- Extraction of essential oils from plant materials (e.g., clove oil, eucalyptus oil).
- Separation of aromatic compounds for perfumes and flavors.
- Used in the pharmaceutical industry for isolating active plant ingredients.
- Employed in petroleum refining for separating high-boiling fractions.

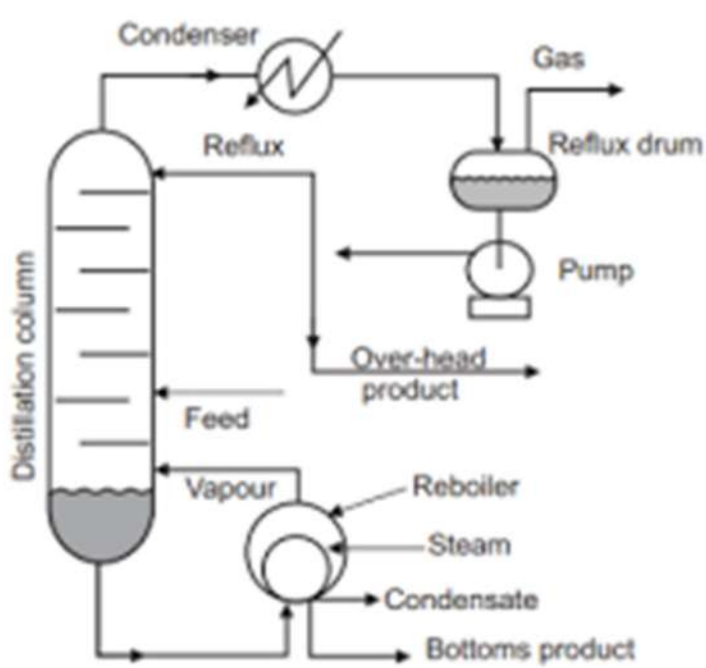
Molecular Distillation

Principle

- Molecular distillation is a special type of vacuum distillation carried out at extremely low pressures (10^{-2} to 10^{-8} mmHg).
- At such pressures, the mean free path of molecules (distance traveled before colliding with another molecule) becomes larger than the distance between evaporating and condensing surfaces.
- Hence, molecules travel directly from the evaporator to the condenser without colliding with other molecules.
- This allows distillation of high-boiling and thermally unstable compounds at very low temperatures, preventing decomposition.

Construction

- Evaporator Surface – Heated surface where the liquid film spreads.
- Condenser Surface – Placed very close (few cm away) and kept cooled.
- Vacuum System – Maintains ultra-low pressure.
- Feed Inlet – Introduces the liquid mixture onto the evaporator.
- Receiver Flasks – Collects the separated fractions (distillate and residue).



Working

1. The liquid mixture is introduced onto the heated evaporator surface.
2. Under high vacuum, volatile molecules evaporate at much lower temperature than their normal boiling points.
3. These molecules travel in a straight line (due to long mean free path) and condense on the nearby cooled condenser surface.
4. The condensed liquid (distillate) is collected in a separate receiver, while the non-volatile residue remains on the evaporator surface.

Advantages

- Prevents thermal decomposition of heat-sensitive materials.
- Enables purification of high-boiling point compounds.
- Operates at low temperatures, saving energy.
- Provides high-purity products.

Disadvantages

- Requires expensive equipment (high-vacuum system).
- Limited to small-scale or specialized operations.
- Not suitable for compounds with very low volatility.

Applications

- Purification of vitamins, hormones, and antibiotics.
- Isolation of essential oils and natural products.
- Separation of fatty acids, esters, and glycerides in the pharmaceutical and cosmetic industries.
- Used in food industry (e.g., purification of omega-3 fatty acids, flavors).