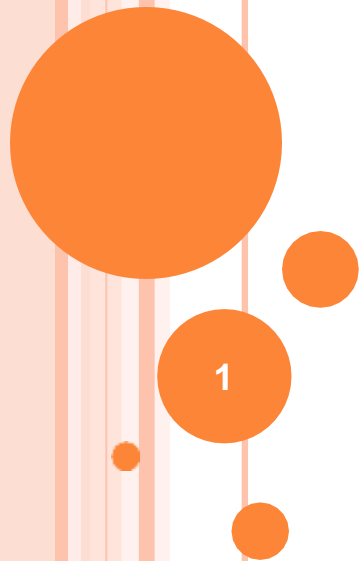


# Flash distillation, Fractional distillation and Distillation under reduced pressure



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## 2. FLASH DISTILLATION

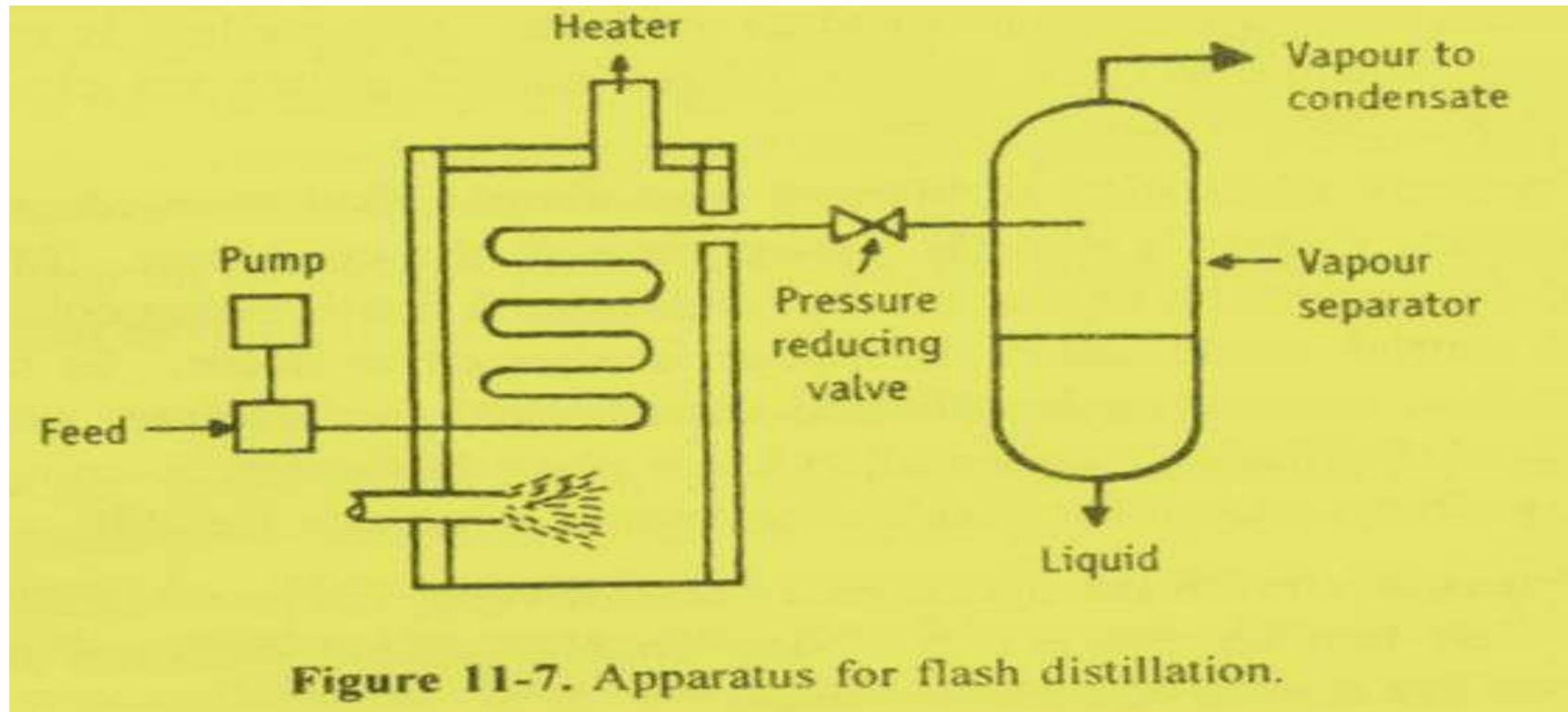
- Flash distillation is defined as a process in which the entire liquid mixture is **suddenly vaporized (flash)** by passing the feed from a **high pressure zone to a low pressure zone**.
- Flash distillation is also known as **equilibrium distillation**, i.e., separation is attempted when the liquid and vapour phases are in equilibrium. This method is frequently carried out as a continuous process and does not involve rectification.

### 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**.
- This process requires certain amount of time. Therefore the liquid and vapour is kept in intimate contact until equilibrium is achieved.
- The liquid fraction is collected separately. The vapour is separated from the liquid and further allowed to condense.

## Construction:

- It consists of a pump, which is connected to a feed reservoir.
- Pump helps in pumping the feed into the heating chamber which contains a suitable heating mechanism.
- The other end of the pipe is directly introduced into the vapour-liquid separator through a reducing valve.
- The vapour outlet is provided at the top of the separator and liquid outlet is provided at the bottom.



## 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**.
- Therefore, **vapour and liquid concentrations at any point remain constant in the unit**.

## 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.

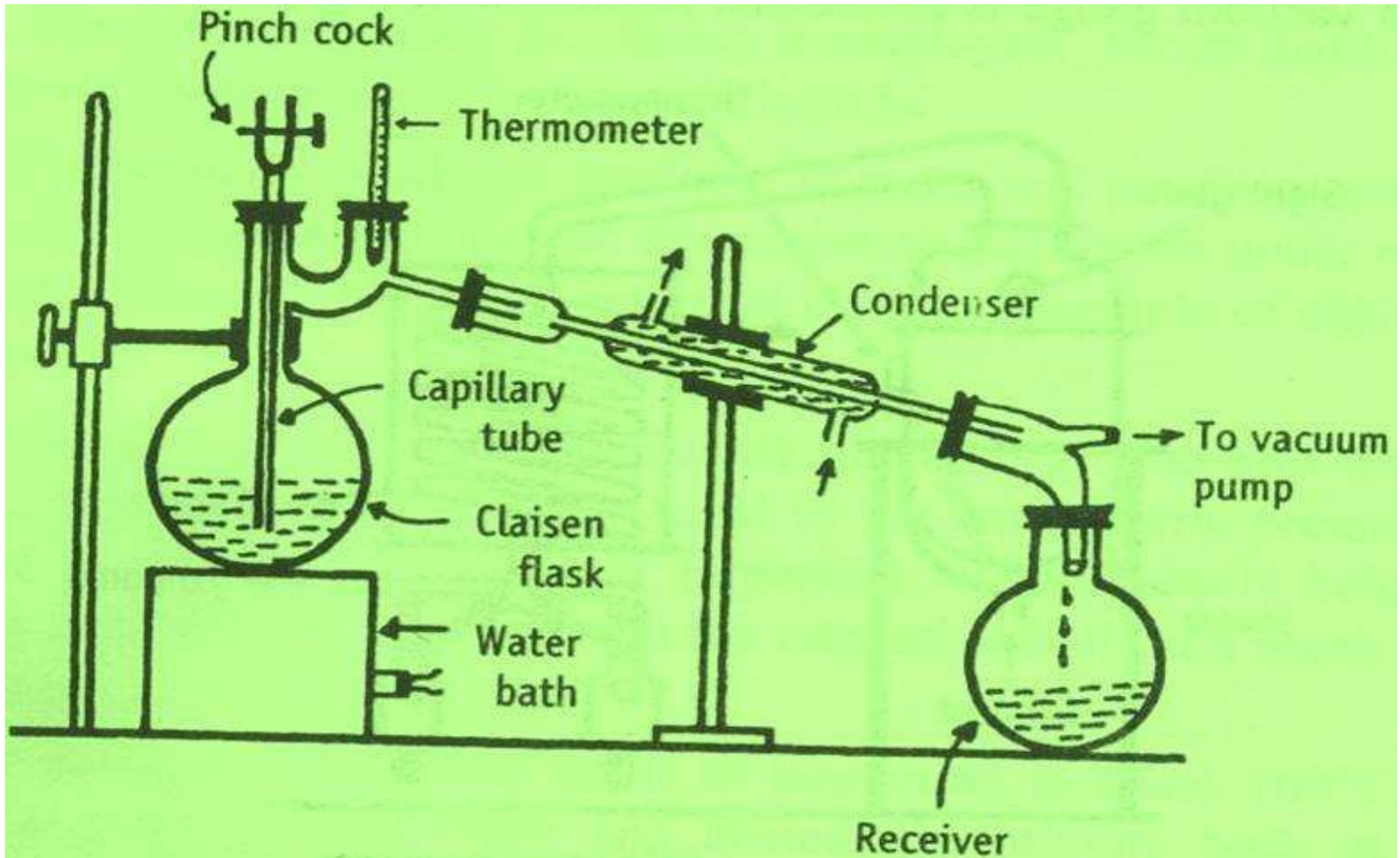
## Advantages:

- ✓ 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.

### 3. Vacuum distillation (distillation under reduced pressure)



**Figure 11-17.** Assembly of apparatus for distillation under reduced pressure (on laboratory scale).

- 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  $100^{\circ}\text{C}$  at an atmospheric pressure is  $101.31\text{ kPa}$  ( $760\text{ mm Hg}$ ). At  $40^{\circ}\text{C}$ , the vapour pressure of water is approximately  $9.33\text{ kPa}$  ( $70\text{ mm Hg}$ ). Hence, the external pressure is reduced to  $9.33\text{ kPa}$  ( $70\text{ mm Hg}$ ) where water boils at  $40^{\circ}\text{C}$ . The net result is the increase in rate of mass transfer into vapour.

The important factor in evaporation is:

**Mass of vapour formed  $\propto$  vapour pressure of evaporating liquid  
external pressure**

According to this formula, water is allowed to evaporate at 40°C and 9.33 kPa (70 mm Hg) pressure, the mass of vapour formed in unit time is approximately 11 times, i.e. **760/70 for water.**

### **Assembling of apparatus:**

- It consists of a **double-neck** distillation flask known as *Claisen flask*.
- Thick walled glass apparatus with interchangeable standard glass joints are used for vacuum distillation.
- In one of the necks of the Claisen flask, a thermometer is fitted. The second neck prevents splashing of the violently agitated liquid.
- Bumping occurs readily during vacuum distillation. **Placing a fine capillary tube** in the second neck of the flask can prevent bumping.
- The capillary tube is dipped in the boiling liquid, so that a **stream of air bubbles is drawn out.**
- Water bath or oil bath is used for heating.
- The Claisen flask is connected to a receiver through a condenser.
- Vacuum pump is attached through an adapter to the receiver. **A small pressure gauge (manometer) should be inserted between the pump and the receiver.**



## Working

- The liquid to be distilled is filled one-half to two-third volume of the flask.
- Small pieces of porcelain are added to the liquid for facilitating distillation and prevent bumping.
- The capillary tube and thermometer are kept in place in the flask.
- The required vacuum is applied.
- The contents are heated gradually.
- The temp. rises and liquid gets vaporised rapidly due to vacuum.
- The vapour passes through the condenser.
- The condensate is collected in the receiver.
- The temp. is noted down, which would be less than the boiling point of the liquid.



## Applications:

*Preventing degradation of active constituents ( $\approx 55^{\circ}\text{C}$ )*

Enzymes - malt extract, pancreatin

Vitamins - thiamine, ascorbic acid

Glycosides - anthraquinones

Alkaloids - hyocyamine to atropine

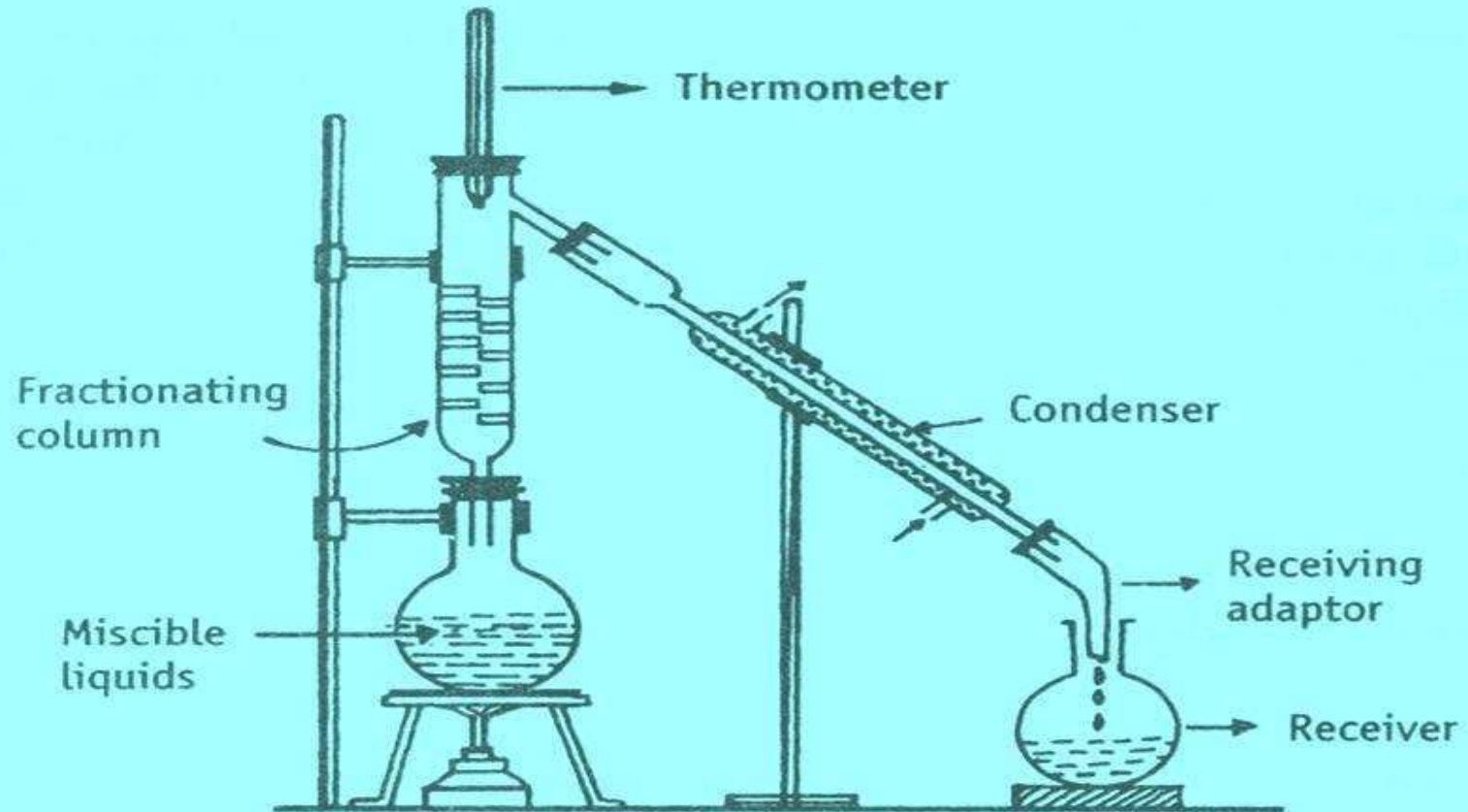
➤ Vacuum distillation is also used in the separation of sensitive organic chemicals and recovery of organic solvents.

## 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.

## 8. FRACTIONAL DISTILLATION



**Figure 11-11.** Assembly of apparatus for fractional distillation (on laboratory scale).

## 8. FRACTIONAL DISTILLATION

- 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.
- 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.

### Simple Distillation Vs Fractional Distillation

Simple Distillation	Fractional Distillation
In simple distillation, vapour is directly passed through the condenser.	In fractional distillation the vapour must pass through a fractionating column in which partial condensation of vapour is allowed to occur.
Condensate is collected directly into the receiver.	Condensation takes place in the fractionating column, so that a part of the condensing vapour returns to the still.

## 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 in 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 miscible liquids such as acetone and water, Chloroform and benzene.

## Disadvantage:

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

## Fractionating columns

- In fractional distillation, special type of still-heads are required so that condensation and re-vaporisation are affected continuously.
- These are known as *fractionating columns*.
- A fractionating column is essentially a long vertical tube in which the vapour passes upward and partially condensed. The condensate flows down the column and is returned eventually to the flask.
- The columns are constructed so as to offer the following advantages simultaneously.
  - (1) It offers a **large cooling surface** for the vapour to condense.
  - (2) An obstruction to the ascending vapour allows easy condensation.

## Fractionating columns Types

A] Packed columns and

B] Plate columns

## Fractionating columns Types

A] Packed columns

