



**Shree H. N. Shukla Institute of Pharmaceutical Education and Research**

**B.Pharm**

**Sem 2**

**Subject Name: Pharmaceutical Engineering**

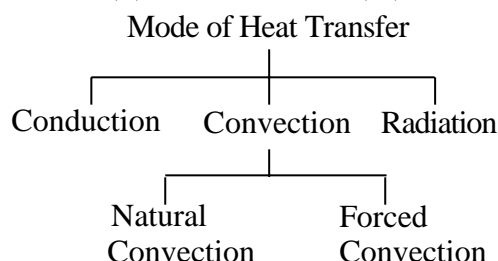
**Subject Code: BP203TP**

**Syllabus:**

1. Heat Transfer: Objectives, applications & Heat transfer mechanisms. Fourier's law, Heat transfer by conduction, convection & radiation. Heat interchangers & heat exchangers.
2. 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.
3. Distillation: Basic Principles and methodology of simple distillation, flash distillation, fractional distillation, distillation under reduced pressure, steam distillation & molecular distillation

**1. HEAT TRANSFER****Classification of heat flow process**

When two objects at different temperatures are brought into thermal contact, heat flows from the object at higher temperature to the object at lower temperature. The mechanisms by which the heat may flow are (i) conduction, (ii) convection and (iii) radiation.

**Conduction**

When heat flow through a body without any observable motion of matter, the type of heat flow is called conduction.

**Mechanism :**

In metallic solids, thermal conduction results from the unbound electrons (which is similar to the electrical conductivity).

In solids that are poor conductor of heat and in liquids the heat is conducted by the transport of momentum of the individual molecules along the temperature gradient.

In gases the conduction occurs by the random motion of the molecules, so that heat is “diffused” from hotter regions to the colder ones.

**Examples:**

Heat flow through the brick wall of a furnace or the metal wall of a tube.

**Convection**

When heat flows by the transfer of matter, the type of heat flow is called convection. In this case the heat flows by actual mixing of warmer portions with the cooler portions of the same material. Convection is restricted to the flow of heat in fluids. Heat flows through fluid by both conduction and convection and it is difficult to separate the two methods because of the eddies set up by the change of density with temperature.

Examples

Transfer of heat by the bodies of turbulent flow and by the current of warm air from a room heater flowing across the room.

**Natural and forced convection**

The forces used to create convection currents in fluids are two types.

*Natural convection:*

When a fluid is heated the warmer part becomes lighter than the cooler part. Due to this difference in density the cooler (higher density) fluid moves down wards and the warmer (lighter density) move upwards and thus forming convection current. Thus heat is transferred with mass. This method of heat transfer is called natural convection.

*Forced convection:*

If the current (or movement of fluid) is caused not by the density difference but by some agitator or by some mechanical devices then the type of heat flow associated with it is called forced convection.

**Radiation**

When heat is transferred through the space by electromagnetic waves the type of flow of heat is called radiation heat flow.

When radiation is passing through empty space, it is not transformed into heat or any other form of neither energy, nor it is diverted from its path. If the radiation falls on a matter the radiation energy will be transmitted (i.e. pass through the matter), reflected or absorbed. Only the energy that is absorbed is converted into heat energy.

**CONDUCTION**

The basic law of heat transfer by conduction can be written in the form of the rate equation:

$$\text{Rate} = \frac{\text{Driving force}}{\text{Resistance}}$$

The driving force is the temperature gradient.

**Fourier's law**

Fourier's law states that the rate of heat flow through a uniform material is proportional to the area perpendicular to the heat flow (A), the temperature drop (dt) and inversely proportional to the length of the path of flow.

Consider an area A of a wall of thickness L. Let the temperature be uniform over the area A on one face of the wall. Both sides of the wall has a temperature gradient. If a thin thickness dL, parallel to the area A, be taken at some intermediate point in the wall, with a temperature difference of dt across such a layer, then Fourier's law may be represented by the equation:

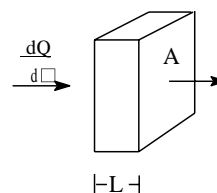
$$\frac{dQ}{d\theta} = \frac{kAdt}{dL}$$

eqn 1

Where k = proportionality constant

If the temperature gradient dt/dL does not vary with time (this case is observed at steady state of heat flow) then the rate of heat flow is constant with time and

$$\frac{dQ}{d\theta} = \text{constant} = q = \frac{kAdt}{dL} \quad \text{eqn. 2}$$



Since normally we know only the temperature at the two faces of the wall hence integrating the Fourier's equation:

$$\frac{q \, dL}{A} = -k \, dt \quad \text{eqn. 3}$$

On integration, if  $t_1$  is the higher temperature than  $t_2$ .

$$q \int_0^L \frac{dL}{A} = - \int_{t_1}^{t_2} k \, dt = -k \int_{t_1}^{t_2} dt \quad \text{eqn. 4}$$

If  $A$  does not vary with  $L$  (i.e. the case of a flat wall) then equation 4 integrates to

$$\frac{qL}{A} = k(t_1 - t_2) = k \Delta t$$

or, by rearranging we get  $q = \frac{k A \Delta t}{L}$  or,  $q = \frac{\Delta t}{L/kA}$  eqn. 5

In equation 5  $\Delta t$  is the driving force and the resistance is  $L/kA$ .

### Thermal conductivity

The proportionality in constant  $k$  in equation  $q = \frac{kA \Delta t}{L}$  is called the *thermal conductivity* (also called the coefficient of thermal conductivity) of the material of which the wall is made.

If  $q$  is expressed in Btu  
 $\Delta t$  in  $^{\circ}\text{F}$   
 $A$  in  $\text{ft}^2$ .  
 $t$  in  $^{\circ}\text{F}$  and  
 $L$  in ft

The unit of  $k$  will be :

$$\frac{(\text{Btu})(\text{ft})}{(\text{hr})(\text{ft}^2)(^{\circ}\text{F})}$$

The numerical value of the thermal conductivity depends upon

(i) The material of which the body is made of

The thermal conductivities of liquids and gases are smaller compared to solids.

For example at  $212^{\circ}\text{F}$  the thermal conductivity of

silver is  $240 (\text{Btu})(\text{ft}) / (\text{hr})(\text{ft}^2)(^{\circ}\text{F})$

water is  $0.35 (\text{Btu})(\text{ft}) / (\text{hr})(\text{ft}^2)(^{\circ}\text{F})$  and

air is  $0.017 (\text{Btu})(\text{ft}) / (\text{hr})(\text{ft}^2)(^{\circ}\text{F})$

(ii) and upon its temperature.

The variation of thermal conductivity with temperature is meager (very small)

but it is assumed that the variation is linear; that is:

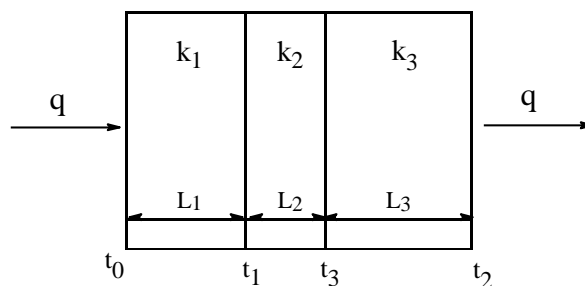
$$k = a + b t$$

where  $a$  and  $b$  are constants and  $t$  is the temperature.

**Compound resistances in series**

Consider a flat wall constructed of a series of layers.

- $L_1, L_2, L_3$  are the thickness of the layers.
- $K_1, K_2, K_3$  are the thermal conductivities of the layers
- Let the area of the compound wall, at right angles to the heat flow be  $A$ .



Let  $t_0, t_1, t_2$  and  $t_3$  be the temperatures at the surfaces of the wall and at each junction according to the figure where  $t_0 > t_1 > t_2 > t_3$ .

Therefore,  $\Delta t = \Delta t_1 + \Delta t_2 + \Delta t_3$

where,  $\Delta t_1 = t_3 - t_0$ .

$\Delta t_2 = t_1 - t_0$ .

$\Delta t_3 = t_2 - t_1$ .

$\Delta t_4 = t_3 - t_2$ .

Again from Fourier's law

$$q_1 + \frac{k_1 A \Delta t_1}{L_1} \quad \text{or,} \quad \Delta t_1 = q_1 \frac{L_1}{k_1 A} = q_1 R_1$$

$$q_2 + \frac{k_2 A \Delta t_2}{L_2} \quad \text{or,} \quad \Delta t_2 = q_2 \frac{L_2}{k_2 A} = q_2 R_2$$

$$q_3 + \frac{k_3 A \Delta t_3}{L_3} \quad \text{or,} \quad \Delta t_3 = q_3 \frac{L_3}{k_3 A} = q_3 R_3$$

Since all the heat passing through the first resistance must pass through the second and in turn, pass through the third, so  $q_1, q_2$  and  $q_3$  must be equal and all of them can be represented by  $q$ . From equation (1-4):

$$\begin{aligned} \Delta t &= \Delta t_1 + \Delta t_2 + \Delta t_3 \\ &= q R_1 + q R_2 + q R_3 \\ &= q (R_1 + R_2 + R_3) \end{aligned}$$

$$\Delta t = q (R_1 + R_2 + R_3) \quad \text{or} \quad q = \frac{\Delta t}{R_1 + R_2 + R_3}$$

If the equivalent resistance of the compound wall is  $R$  then  $q = \frac{\Delta t}{R} = \frac{t_3 - t_0}{R_1 + R_2 + R_3}$

$$\Delta t = q (R_1 + R_2 + R_3)$$

**HEAT FLOW THROUGH A CYLINDER**

Let us consider the hollow cylinder represented by the figure.

$r_1$  = inside radius

$r_2$  = outside radius

$t_1$  = inside temperature

$t_2$  = outside temperature

$N$  = length of the cylinder

$k_m$  = mean thermal conductivity of the material of the cylinder

It is desired to calculate the rate of heat flow through the wall.

Let us consider a very thin cylinder, concentric with the main cylinder with a radius  $r$  where  $r_1 < r < r_2$ .

the thickness of the wall of that cylinder is  $dr$

temperature difference across  $dr$  is  $dt$

Then applying Fourier's law over the thin wall will give:

$$q = \frac{k_m A dt}{dr} \quad \text{where } A = \text{total area of the thin wall}$$

$$= \frac{(2\pi r N) k_m dt}{dr}$$

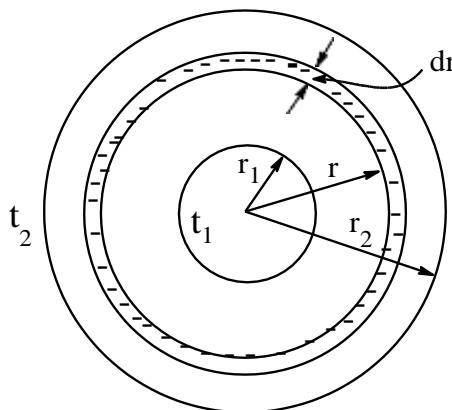
$$\frac{dr}{r} = \frac{2\pi N k_m dt}{q}$$

$$\text{or, } \int_{r_1}^{r_2} \frac{dr}{r} = \frac{2\pi N k_m}{q} \int_{t_1}^{t_2} dt$$

$$\text{or, } \left[ \ln r \right]_{r_1}^{r_2} = \frac{2\pi N k_m}{q} (t_2 - t_1)$$

$$\text{or, } \ln \frac{r_2}{r_1} = \frac{2\pi N k_m}{q} (t_2 - t_1)$$

$$\text{or, } q = \frac{k_m (2\pi N) (t_1 - t_2)}{\ln \frac{r_2}{r_1}}$$



## PRINCIPLES OF HEAT FLOW IN FLUIDS

Heat transfer from a warmer fluid to a cooler fluid, usually through a solid wall separating the two fluids, is found in different heat transfer equipment like heat exchangers, evaporators etc.

The heat transferred may be in the following forms:

- (i) latent heat accompanying a phase change such as condensation, vaporization
- (ii) sensible heat without any phase change.

*Mechanisms of heat transfer through a fluid* : Both by conduction and convection.

### Heat flow from one fluid to another fluid separated by a solid wall

For example a liquid is flowing through a pipe and that liquid is heated by a steam from outside the pipe. In this case heat will be transferred from steam to the liquid. Both steam and the liquid are having Reynolds number above 4000 i.e. they are flowing in turbulent motion. In this case steam will produce a thin film on the outside surface of the pipe and the liquid will form another film at the inside surface of the pipe wall [because the flow is very slow near the solid wall hence in the film viscous flow will prevail]. Beyond these films the steam and the liquid are remaining in turbulent motion i.e. complete mixing is going on.

Heat is transferred through this stagnant film by *conduction* and when it reaches the bulk of the fluid heat is mixed by *forced convection*. Since the bulk of the fluids are in great motion hence the heat transfer within the bulk is very rapid. Since the *thermal conductivities* of the fluids are low hence, although the films are very thin, the *resistance* offered by it to the flow of heat is very large.

### Temperature gradients in forced convection

Let us consider that heat is flowing from a hot fluid through a metal wall to a cold fluid.

- The dotted lines  $F_1F_1$  and  $F_2F_2$  on each side of the solid wall is representing the *boundaries of the films in viscous flow*; all parts of the fluids to the right of  $F_1F_1$  and to the left of  $F_2F_2$  are in turbulent flow.
- The temperature gradient from the bulk of the hot fluid to the metal wall is represented by the curved line  $t_a t_b t_c$ .

The temperature  $t_a$  is the maximum temperature in the hot fluid (i.e. in the bulk of the fluid).

The temperature  $t_b$  is the temperature at the boundary between the viscous and turbulent flow.

The temperature  $t_c$  is the temperature at the actual interface between the fluid and the solid wall.

Similarly for fluid 2 the curved line is  $t_d t_e t_f$ .

- When a thermometer (or any heat sensing instrument) is inserted into the bulk of the fluid it will show a temperature  $t_1$  and  $t_2$  respectively for the two fluids. This  $t_1$  is neither  $t_a$  nor  $t_b$  but this will be an average temperature and  $t_a < t_1 < t_b$ .  $t_1$  is shown as a straight line  $MM$ .

The same remarks will apply for fluid 2 also whose average temperature  $t_2$  is represented by the line  $NN$ .

- The temperature gradient  $t_d t_c$  is caused by the flow of heat in pure conduction through the solid wall and this  $(t_c - t_d)$  smaller than  $(t_1 - t_2)$ .

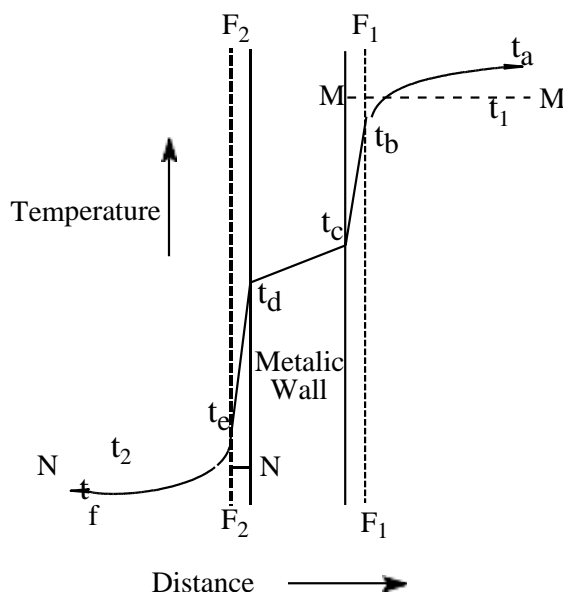


Fig. Temperature gradients in forced convection

### Surface coefficients

Since the thickness of the film is not known, the simple equation of conduction cannot be applied in this case. The difficulty is circumvented by the use of *surface coefficient*.

The *surface coefficient* on the hot side is defined by the relation

$$h_1 = \frac{q}{A_1 (t_1 - t_c)}$$

where,  $h_1$  = surface coefficient of the fluid on the hot side [Btu/(ft<sup>2</sup> °F s)]

$q$  = amount of heat flowing from hot to cold fluid. [Btu / s]

$A_1$  = area of the metal wall on the hot side in a plane at right angle to the heat flow. [ft<sup>2</sup>]

$(t_1 - t_c)$  = temperature gradient. [°F]

If we compare  $q = h_1 A_1 (t_1 - t_c)$  equation with  $q = \frac{k A L}{L}$  then it is evident that

$h_1$  is analogous to  $(k / L)$  and  $(1/h_1 A_1)$  is the resistance term same as that of  $(L / kA)$  and  $h_1$  contains the effect of both the viscous film and of the thermal resistance of the turbulent core that causes the temperature difference  $(t_1 - t_c)$ .

In the same way  $h_2$  may be defined as 
$$h_2 = \frac{q}{A_2(t_d - t_2)}$$

So the resistance imparted by the hot side =  $1 / h_1 A_1$ .

Resistance imparted by the metallic wall =  $L / k A_m$ .

Resistance imparted by the cold side =  $1 / h_2 A_2$ .

### Overall heat transfer coefficient

If these resistances are substituted in the equation for compound resistance in series

$$q = \frac{t_1 - t_2}{R_1 + R_2 + R_3}$$

$$\text{then } q = \frac{t_1 - t_2}{\frac{1}{h_1 A_1} + \frac{L}{k A_m} + \frac{1}{h_2 A_2}}$$

If the numerator and denominator are multiplied with  $A_1$  then

$$q = \frac{A_1(t_1 - t_2)}{1/h_1 + A_1 L / k A_m + A_1 / h_2 A_2}$$

So the *overall heat transfer coefficient*  $U_1$  is defined by the equation

$$U_1 = \frac{1}{1/h_1 + A_1 L / k A_m + A_1 / h_2 A_2}$$

Therefore  $q = U_1 A_1(t_1 - t_2)$  states that the *rate of heat transfer is the product of three factors: overall heat transfer coefficient ( $U_1$ ), temperature drop ( $\Delta t$ ), and area of heating surface ( $A_1$ ).*

For a tubular pipe  $A_1 = \pi D_1 l$  where  $D_1$  and  $l$  are the inner diameter and length of the pipe respectively. Similarly  $A_2 = \pi D_2 l$  and  $A_m = \pi D_m l$ .

So another form of overall heat transfer coefficient :

$$U_1 = \frac{1}{1/h_1 + D_1 L / k D_m + D_1 / h_2 D_2}$$

Analogous equation can be written for  $U_m$  and  $U_2$ .

### Fluids in natural convection

If a fluid is in contact with a heated surface, the fluid immediately adjacent to the tube will tend to rise because of its decreased density and to be replaced by colder fluid. This fluid circulation caused by density differences due to the temperature differences in the fluid is termed natural convection.

The velocity of circulation of the fluid is dependent

on the density differences

on the geometry of the system, i.e. to the size, shape, arrangement of the heating surface and the shape of the heating vessel in which the fluid is enclosed.

For the simple case of a fluid outside a single horizontal cylinder with a large extent of fluid surrounding the cylinder, the heat-transfer coefficient for natural convection has been correlated by an equation containing three dimensionless groups the Nusselt number (Nu), the Prandtl number (Pr) and the Grashof number (Gr):

$$Nu = f(Gr, Pr)$$



$$\text{where } Nu = \frac{hD_o}{k_f}, \quad Gr = \frac{D_o^3 \rho_f^2 g \Delta T_o}{\mu_f^2}, \quad Pr = \frac{c_p \mu_f}{k_f}$$

where  $h$  = average heat transfer coefficient, based on the entire pipe surface

$k_f$  = thermal conductivity of fluid

$c_p$  = specific heat of fluid at constant pressure

$\rho_f$  = density of fluid

$\beta$  = coefficient of thermal expansion of fluid

$g$  = acceleration of gravity

$\Delta T_o$  = average difference in temperature between outside of pipe and fluid distant from wall

$\mu_f$  = viscosity of fluid

When hot bodies lose heat to their surroundings they do so both by radiation and convection. In the lower temperature range convection is more important, in higher temperature range radiation is more important.

### Heat transfer through boiling liquids

Consider a horizontal tube or a group of horizontal tube immersed in a pool of pure liquid with steam or other source of heat inside the tubes.  $\Delta T$  is the difference in temperature between the tube wall temperature and the temperature of the liquid (under the pressure of the vapour space above the liquid).

- When the  $\Delta T$  is very small the rate of heat transfer is nearly similar to that of a non-boiling liquid.
- As the  $\Delta T$  is increased, the coefficient of heat transfer is increased rapidly because the stirring effect of the increasing number of bubbles released produces currents in the liquid that accelerates the heat transfer. This increasing coefficient multiplied with the increasing  $\Delta T$ , results in an even more rapid increase in the total heat transferred per unit area.
- However, if the temperature of the surface is continually increased, a point is found where the heat-transfer coefficient reaches a maximum.
- At higher  $\Delta T$  beyond the maximum value the heat transfer rate is sharply lowered. Actually the bubbles of vapour formed on the heating surface are discharged rapidly to rise through the liquid. This type of boiling is called *nucleate* boiling. At critical  $\Delta T$ , these bubbles coalesce into a continuous film of vapour that insulates the tube, and this reduces the heat transfer rate with increasing  $\Delta T$ .

With polished horizontal tube in reasonably pure water, this critical point is reached at relatively modest value of  $\Delta T$ , possibly 45 to 50°F. With rougher commercial steel tubes the critical  $\Delta T$  is much higher.

In case of nucleate boiling how easily the bubbles will leave the surface depends on the following factors:

- *roughness of the tube and the type of roughness*: For instance a rough surface with small sharp projections makes it possible to detach bubbles from points more easily than from a smooth surface.
- *the tendency of the liquid to wet the tube*: A liquid that wets the tube strongly tends to pinch off the bubbles of gas and liberate them more quickly than a liquid that does not wet the surface easily.
- *the difference in density between the bubble and the liquid*

- *the physical arrangement of the surface*: For instance, a vertical tube with bubbles rising inside it, will always show a much higher critical  $\Delta T$  than a horizontal tube, with bubbles formed on the outside.

### Heat flow through condensing vapours

When a saturated vapour, such as steam, transmits its heat to a metal surface and is condensed, the condensation may take place in either of two entirely distinct forms. One is *film type condensation* and the other is *drop-wise condensation*.

*Film-type condensation*:

In this case the condensed liquid wets the surface on which it is condensing and forms a continuous film of condensate. If the condensate is occurring on the outside of the tube then this film of condensate drops off the underside of the tube. If the tube is vertical then the condensate runs down the whole length of the tube.

For the case of a **horizontal tube in true-film type condensation** on which there is condensing a saturated vapour, free from any non-condensed gas and moving at low velocities, Nusselt has derived the following equation:

$$h = 0.725 \sqrt[4]{\frac{k^3 \rho g \Delta T}{D \mu}}$$

where  $\rho$  = latent heat of vaporisation of vapor, Btu/lb

$\rho$  = density of condensate, lb/ft<sup>3</sup>.

$k$  = thermal conductivity of condensed vapor  $\frac{(\text{Btu})(\text{ft})}{(\text{ft}^2)(\text{h})(^\circ\text{F})}$

$g$  = acceleration due to gravity (ft/hr<sup>2</sup>)

$\mu$  = viscosity of condensate film, (ft-lb-hr) units

$D$  = outside pipe diameter, ft

$\Delta T$  = temperature difference between vapor and metal,  $^\circ\text{F}$ .

For the case of a vertical tube with all other conditions same as above Nusselt has given the equation:

$$h = 0.943 \sqrt[4]{\frac{k^3 \rho g \Delta T}{L \mu}} \quad \text{where } L = \text{the length of the tube.}$$

- The film coefficient between condensing vapors and metal walls increases with increasing temperature of the vapor, because of decreased viscosity of the film condensate.
- Coefficient ( $h$ ) decreases with increasing temperature drop, because increasing temperature drops cause faster condensation and hence thicker liquid films.
- The presence of non-condensable gas accumulate near the heating surface and add to their resistance to that of the liquid film.

### Drop-wise condensation

In this case the condensed liquid does not wet the surface, but collects in drops that may range from microscopic size up to drops easily seen with naked eye. This drops grow for a while and then fall of the surface, leaving an apparently bare area in which new drops form again.

These two types of condensation give widely different film coefficients of heat transfer. Coefficients in the case of drop-wise condensation are much greater than those obtained in the film type condensation provided all other characteristics of the surface are the same.

## RADIATION

Any solid body at any temperature above absolute zero radiates energy. This radiation is an electromagnetic phenomenon and takes place without the necessity of any medium.

The approximate range of wavelengths for infra-red radiation (or heat rays) is 0.8 to 400  $\mu\text{m}$ .

In industries, most of the cases, the thermal radiation corresponds to wavelengths from 0.8 to 25  $\mu\text{m}$ .

### The Black Body

Not all substances radiate heat at same rate at a given temperature. So a theoretical hot body is defined, which is called '**black body**'.

*Definition:* A 'black body' is defined as that body which radiates maximum possible amount of energy at a given temperature.

*Example:* It has been shown that the inside of an enclosed space, at a constant temperature throughout, viewed through an opening so small that the amount of energy escaping through the opening is negligible, corresponds to a black body.

*Practical e.g.* In practice, a convenient black body is made from a tube of carbon plugged at both ends with a small observation hole in the center of one end.

*Industrial e.g.* The inside of furnace at completely uniform temperatures, viewed through a small opening, is a black body. The interior of the furnace and all the objects within the furnace can also be considered black bodies.

### Rates of radiation

If the radiation energy emitted by a hot body is plotted against the wave lengths emitted then graphs of the nature shown in the fig will be obtained.

The total amount of radiation emitted by a black body would be given by integrating the curves of the figure (Effect of temperature on amount and distribution of black-body radiation) The result is the Stefan-Boltzmann law

$$q = b A T^4.$$

where

$q$  = energy radiated per hour (BTU/hr)

$A$  = area of radiating surface (sqft)

$T$  = absolute temperature of the radiating surface  $^{\circ}\text{R}$  (Rankine) =  $t^{\circ}\text{F} + 460$

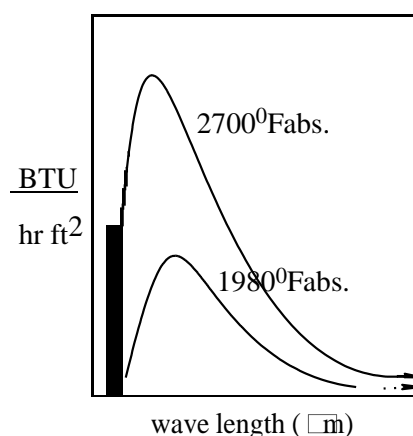
For black bodies the value of  $b = 0.174 \times 10^{-8} \text{ Btu} / (\text{hr sqft } ^{\circ}\text{F}^4)$

No actual body radiates quite as much as the black body. The radiation by any actual body can be expressed as

$$q = \epsilon b A T^4.$$

where  $\epsilon$  is the emissivity of the body.

- The emissivity is a fraction less than 1 and is the ratio of the energy emitted by the body in question to that emitted by a black body at the same temperature.
- When a radiant energy falls on a cooler body either the energy is *reflected* or *transmitted* or *absorbed*. The fraction of the radiation energy falling on a body that is absorbed is represented by  $\epsilon$ , the *absorptivity*, which is always less than 1. If the reflected energy can be neglected then the energy absorbed by any body is equal to the radiation falling on it.
- It can be shown that the absorptivity of a given substance at a given temperature and its emissivity at the same temperature must be equal.



That is,

$$\alpha = \epsilon$$

Since the  $\epsilon$  of a black body is 1 hence the  $\alpha$  of the black body will also be equal to 1. Therefore the black body absorbs all the radiation falling on it – an important property of black body.

- The value of  $\alpha$  for a given surface at a given temperature varies somewhat with the wavelength of the radiation involved. To avoid complication due to this the concept of *gray body* has been introduced. The *absorptivity* of a gray body at a *given temperature* is constant for all wavelengths of radiation.
- When a small black body of area A and temperature  $T_2$  is completely surrounded by a hotter black body of temperature  $T_1$ , the net amount of heat transferred from the hotter body to the colder body is, therefore the algebraic sum of the radiation from the two bodies, so that Stefan's law may be written for this case as...

$$q = b A (T_1^4 - T_2^4)$$

### HEAT EXCHANGERS

Some of the processes that involve heat transfer in pharmaceutical industries are:

- Preparation of starch paste (in steam jacketed kettle)
- Crystallization
- Evaporation
- Distillation

The equipment's used for heat transferring are known as heat exchangers.

### Classification of heat exchangers

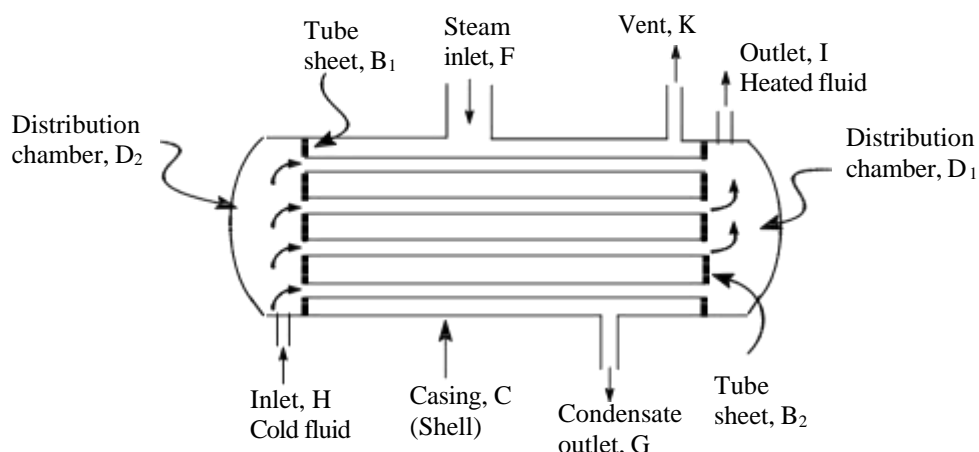
On the basis of transfer of heat, heat exchangers are classified as:

1. *Direct transfer type*: The hot and cold fluids are separated by a metal wall through which the heat is transferred from hot fluid to cold fluid. E.g. shell and tube heater,
2. *Storage type*: First a hot fluid is flown through a porous solid medium to heat the medium, then the cold fluid is flown through the hot solid porous medium to extract the heat from it. This type of heat exchangers is not used in pharmaceutical industries.
3. *Direct contact type*: Hot fluid is passed through the cold fluid and in this case the hot and cold fluids are not separated physically. For example steam is bubbled through a cold liquid.

### Tubular heater

Tubular heaters consist of circular tubes, one fluid flows through the inner tube, while the other flows through the outside space. The heat transfer takes place across the wall of the tube.

## 1. Single Pass Tubular heater:



**Construction:** It consists of a bundle of *parallel tubes*, which are relatively thin-walled. The ends of these tubes are fitted to two *tube-sheets* B1 and B2. The bundles of parallel tubes are enclosed in a cylindrical shell or *casing* (C, made of cast iron) to which the tube-sheets (B1 and B2) are fitted. Two *distribution chambers* D1 and D2 are provided at each end of the casing (C). *Cold fluid inlet* (H) is fitted with distribution chamber D2 and *hot fluid outlet* (I) is fitted with the distribution chamber D1. *Steam inlet* F, *steam outlet* K (called *vent*) and *condensate outlet* G are fitted to the shell.

**Working:** Steam is introduced through the steam inlet F into the space surrounding the parallel tubes. Heat is transferred to the cold liquid inside the tubes and steam is condensed. The condensate is removed through condensate outlet G placed at the bottom of the casing. Non-condensable gases, if any, escapes through the vent K provided at the top of the shell.

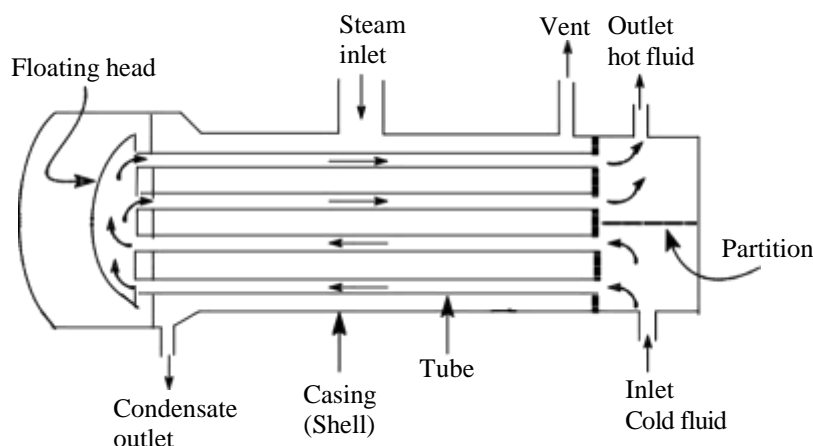
The fluid to be heated is pumped through the cold fluid inlet (H) in to distribution chamber D1, flows through the tubes and collects in the distribution chamber D2. Heat is transferred from the steam to the cold fluid through the metal wall. The hot fluid leaves the heater through outlet (I).

**Advantages:** Large heating surface is packed into a small volume.

**Disadvantages:**

- The velocities of the fluid flowing through these tubes are low because of large cross-sectional area or surface area.
- The expansion of the tubes and shell takes place due to difference in temperatures. This may lead to loosening of the tube sheets from the casing.
- Initial cost and maintenance costs are very high.

## 2. Floating-head two-pass heater

*Construction:*

It consists of a bundle of parallel tubes. They are enclosed in a shell (casing). The right side of the distribution chamber is partitioned and fluid inlet and outlet are connected to the same chamber. The partition is such that both have equal number of tubes. On left side the distribution chamber is not connected to the casing. It is structurally independent, hence known as *floating head*. The ends of the tubes are fitted with the floating head. Casing is provided with steam inlet, vent, and condensate outlet.

*Working:*

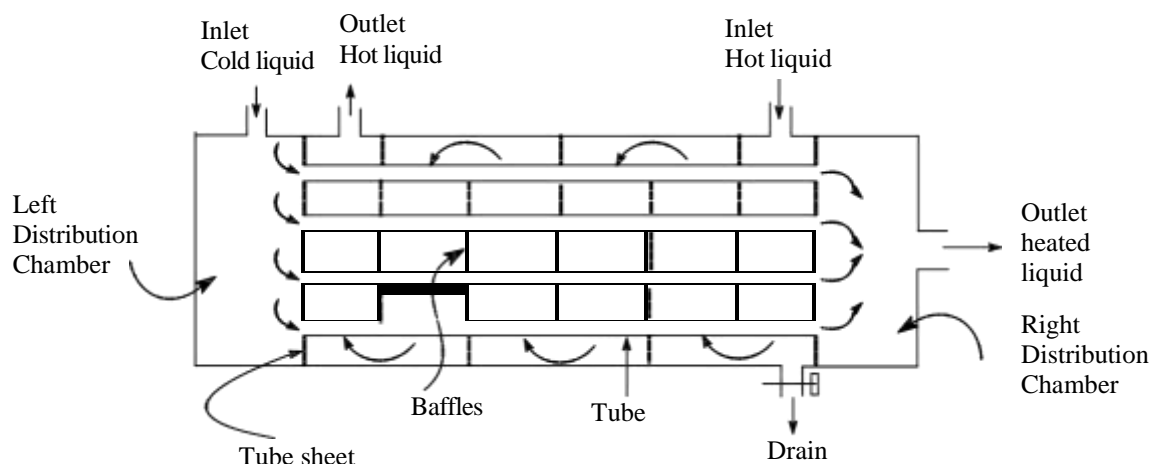
Steam is introduced through the steam inlet of the casing (shell). Steam heats the tubes. The condensate escapes through the condensate outlet fitted at the bottom of the casing. Non-condensable gas, if any, escapes through the vent of the casing provided at the top.

The cold fluid is introduced through the cold fluid inlet into the right-hand distribution chamber. The fluid flows through few tubes present in the lower part of the distribution chamber. The fluid reaches the *floating head* and changes direction and flows through the upper tubes again to the right-hand distribution chamber. The hot fluid is taken out through the outlet.

During this process the fluid in the tubes get heated due to heat transfer through the metallic wall.

*Advantages:* Due to differences in temperature the tubes and shells may expand and the joints may get loose. Since, the floating head part is independent of shell (or casing) hence the problem of loosening is prevented in this type of heater.

## 3. Liquid-to-liquid heat interchanger

*Construction:*

Construction is same as that of single-pass tubular heater, only difference is that it contains *baffles* to lengthen the path of flow of outer liquid. A set of parallel tubes are fitted to two *tube-sheets* at two ends. The tubes and the tube-sheets are placed inside the shell. Cold liquid inlet is fitted to the left-hand side distribution chamber and outlet is fitted with the right-hand side of the distribution chamber. Hot liquid is entering through the right-hand top side of the shell and leaving the shell through the outlet placed on the top, left-hand side of the shell.

*Baffles* consists of circular metal sheet, with one side cut away. Baffles are placed inside the shell at appropriate places. Baffles have perforation on it through which the tubes pass.

*Working:*

The hot liquid is pumped from the left-top of the shell. The fluid flows through the shell (i.e. outside of the tubes) and moves down directly to the bottom (due to baffle), again moves up – like this it flows from the left to right-hand side of the shell.

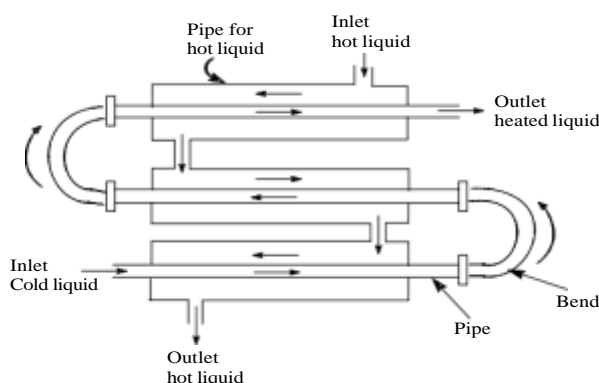
Baffles increases the velocity of the hot fluid outside the tubes, which creates more turbulence. This reduces the film thickness at the outside of the tube and thus increases the film coefficient and thus heat transfer increases.

The baffles also get heated and add to the heat transfer to cold liquid.

The cold liquid is pumped through the inlet at the left-hand side distribution chamber. The liquid passes through the tubes and gets heated. The heated liquid is collected from the right-hand side distribution chamber.

## 4. Double-pipe heat interchanger

N.B. In a liquid-to-liquid heat interchanger, the fluid to be heated is passed only once through the tubes before it is discharged, i.e. single pass. The heat transfer in this case is not efficient.



In double-pipe heat interchanger number of pass can be increased as desired.

*Construction:*

In this case two pipes are used – one is inserted into the other. Cold fluid is passed through inner tube. The outer pipe acts as a jacket for the circulation of hot fluid. All jacketed sections are interconnected.

Normally the number of pipe-sections are few. The length of the pipe is also less. The inner tubes may be made of glass and standard iron. The pipes are connected with standard return bends and the pipes are stacked vertically. The pipes may have longitudinal fins on its outer surface for better heat transfer.

*Working:* The hot liquid is pumped into the jacketed section. It is circulated through the annular spaces between them and carried from one section to the next section. Finally it leaves the jacket. In this process the pipes get heated.

The liquid to be heated is pumped through the inlet of the inner tube. The liquid gets heated up and flows through the bend tube into the next tube-section and finally leaves the exchanger.

*Limitation:* Double-pipe heat interchanger is economical when the heating surface area is less than 9 m<sup>2</sup>.

## SOURCES OF HEAT

1. Steam
2. Electricity: Heating coil made with nichrome wire. Used in heating oven, tray dryer. Air can be heated by passing through heating coil. Water can be heated by using heating element. Heating element is a coil of nichrome wire covered with a nonconductor and enclosed within a steel pipe.
3. Infra red source: Infra Red heating source with carbon filament. Used for drying films, coatings etc. as the materials are conveyed under the IR heat source.

### Steam as heating media

Steam has wide-spread application in pharmaceutical industries. The applications are as follows:

1. Steam has very high heat content. – Under 1 atm pressure saturated steam has 540cal/gm of latent heat.
2. The heat of steam is given up at constant temperature. – When steam condenses no temperature change takes place only the latent heat is given up.
3. The raw material of steam is water. It is cheap and easily available.
4. Steam is clean, odourless and tasteless, so that any accidental contamination of a product are not likely to be serious.
5. Steam is easy to generate, distribute and control.

### Properties of steam

**One kg** of water is taken in a closed vessel one side of which is closed by a moving frictionless piston. By changing the weight of the piston the pressure inside the vessel can be changed. Let us imagine that the piston is giving a **constant pressure, P** and the temperature inside is **0°C**.

1. State-1: Heat (h) is added until water starts to boil. This h amount of heat is required to raise the temperature of water from 0°C to t<sub>s</sub>. So

$$h = ms \Delta t \quad \text{where } m = 1 \text{ kg, } s = \text{specific heat of water} = 1000 \text{ J/kg}^\circ\text{C}$$



$$= 1 \times 1000 \times (t_s - 0)$$

$$= t_s \text{ kJ}$$

2. State-2: If more heat is added,  $q$  fraction of water will be vaporized. If the latent heat of vaporization of water is  $L$  kJ/kg then the steam contains  $qL$  kJ amount of heat and the total heat of water and steam  $= (h + qL)$  kJ.

When steam remains in contact with water that steam is called *wet steam* and  $q$  is known as *dryness fraction*.

3. State-3: If further heat is added, a point will be reached when all the water will be converted to steam i.e.  $q = 1$ . The total heat of the steam is now  $H_s = (h + L)$  kJ. The temperature is still  $t_s$ . Since no water is there in contact with steam hence this type of steam is called *dry saturated steam*.
4. State-4: If more heat is provided to dry saturated steam then the temperature of the steam will increase above  $t_s$  (say  $t_{sup}$ ). This type of steam is called *super-heated steam*.  
The total heat content  $= H_s + H_{sup} = h + L + H_{sup}$ .

### Generation of steam

The steam is generated in central boiler house at high pressure. High-pressure steam may be used to generate electricity by driving a turbine and the low-pressure steam that is exhausted can be used to heat various processes. Under high pressure steam can be carried through pipes to different equipments.

### Distribution of steam

From the boiler the steam is distributed through piping of adequate size and minimum length to minimize heat loss. To reduce heat loss the pipes should be *lagged*, i.e. the pipes are covered with a porous, poor conducting material such as asbestos, kieselguhr or glass wool. The porous medium entraps a stagnant layer of air around the pipe.

An alternative method of reducing heat loss is by covering the pipes with several layers of aluminium sheets. The surface of the aluminium reduces radiation heat loss and the air entrapped within the layer of aluminium sheets reduces heat loss due to convection and conduction.

### Boiler capacity

The output of a boiler is often expressed in pounds of steam delivered per hour. Since this value may vary in temperature and pressure over time, a more accurate and complete expression is that of heat transferred over time, expressed as British thermal units per hour. Boiler capacity is usually expressed as kBtu/hour (1000 Btu/hour).

### Steam Pressure

If pressure increases the temperature of the steam will increase, thus making it super-heated steam. Super-heated steam contains less amount of latent heat compared to dry saturated-steam. Since the latent heat is the useful heat, steam should be used at the lowest pressure that will give a suitable temperature gradient.

## **2. EVAPORATION**

### **Definition**

Theoretically, evaporation means simply vaporization from the surface of the liquid.

Evaporation is an unit operation by which a solvent is evaporated from a solution by boiling the liquor in a suitable vessel and withdrawing the vapor, leaving a concentrated liquid residue.

Objective of evaporation:

To make a solution more concentrated. Generally extracts are concentrated in this way.

Factors affecting evaporation:

#### **(i) Temperature:**

Heat is necessary to provide the latent heat of vaporization, and in general, the rate of evaporation is controlled by the rate of heat transfer. Rate of heat transfer depends on the temperature gradient.

Many pharmaceutical agents are thermolabile. So the temperature that will cause the least possible decomposition should be used.

e.g. Many glycosides and alkaloids are decomposed at temperature below 100<sup>0</sup>C.

e.g. Hormones, enzymes and antibiotics are extremely heat sensitive substances. e.g.

Malt extract (containing enzyme) is prepared by evaporation under reduced pressure to avoid loss of enzymes.

Some antibiotics are concentrated by freeze-drying.

#### **(ii) Temperature and time of evaporation**

Exposure to a relatively high temperature for a short period of time may be less destructive of active principles than a lower temperature with exposure for a longer period.

Film evaporators used a fairly high temperature but the time of exposure is very short.

An evaporating pan involve prolonged heating.

#### **(iii) Temperature and moisture content**

Some drug constituents decompose more rapidly in the presence of moisture, especially at a raised temperature (by hydrolysis). Hence, evaporation should be carried out at a low controlled temperature, although the final drying can be performed at higher temperature when little moisture remains.

e.g. Belladonna Dry Extract is an example of this type.

#### **(iv) Type of product required**

Evaporating pans or stills will produce liquid or dry products, but film evaporators will yield only liquid products. So a dilute extract can be first concentrated in a film evaporator and then the concentrated extract may be dried in an evaporating pan.

#### **(v) Effect of concentration**

As the liquor becomes concentrated, the increasing proportion of solids results in elevation of the boiling point of the solution. This leads to a greater risk of damage to thermolabile constituents and reduction of the temperature gradient.

In general concentrated solutions will have increased viscosity, causing thicker boundary layers, and may deposit solids that may build up on the heating surface that reduce heat transfer.

All these problems may be minimized by turbulent flow condition.

## EVAPORATORS

Evaporators are classified according to the form of the movement,

- (i) Natural circulation evaporators.
- (ii) Forced circulation evaporation
- (iii) Film evaporators

### (i) Natural Circulation Evaporator

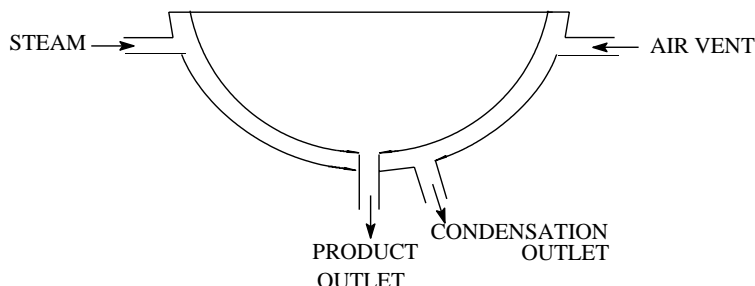
#### EVAPORATING PAN

##### Construction

The apparatus consists of a hemispherical, or shallow pan, constructed from a suitable material such as copper or stainless steel and surrounded by a steam jacket.

The hemispherical shape gives the best surface/ volume ratio for heating, and the largest area for separation of vapor.

The pan may have a mounting, permitting it to be tilted to remove the product, but the shallow form makes this arrangement somewhat unstable, and an outlet at the bottom, is common.



##### Working

The dilute solution is taken in the pan. Steam is introduced through the steam inlet into the jacket to heat the pan. In these evaporators the movement of the liquid results from convection currents set up by the heating process. The concentrated liquid is collected through the outlet placed at the bottom of the pan.

##### Advantages:

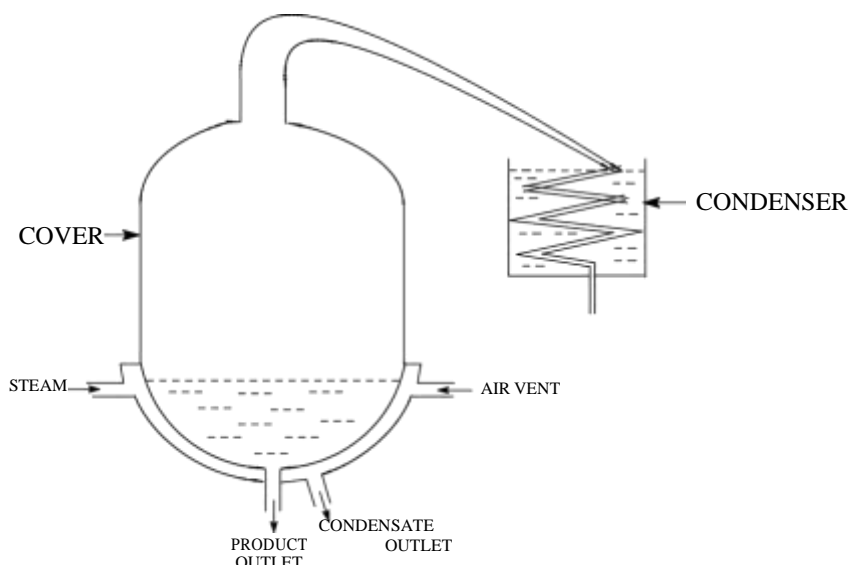
- (a) It is simple and cheap to construct.
- (b) It is easy to use, clean and maintain.

##### Disadvantages:

- (a) Having only natural circulation, the overall coefficient of heat transfer will be poor and solids are likely to deposit on the surface, leading to decomposition of the product and a further deterioration in heat transfer.
- (b) Also many products give rise to foaming.
- (c) The total liquor is heated over all the time, which may be unsatisfactory with thermolabile materials.
- (d) The heating surface is limited and decreases proportionally as the size of the pan increases.
- (e) The pan is open, so the vapor passes to the atmosphere, which can lead to saturation of the atmosphere.
- (f) Only aqueous liquids can be evaporated in these pans.
- (g) Pan evaporation cannot be done under reduced pressure.
- (h) Can only be used for thermolabile products.

**EVAPORATING STILLS****Construction**

It consists of a jacketed-evaporating pan with a cylindrical *cover* that connects it to a condenser. The over all assembly is called *still*. The cover is clamped with the evaporating pan.

**Working**

The dilute liquid is fed into the still, the cover is clamped. Steam is introduced into the jacket. The liquid is evaporated and condensed in the condenser and collected. The product (i.e. concentrated liquid) is collected through the product outlet.

**Advantages:**

- (a) Simple construction and easy to clean and maintain.
- (b) The vapor is removed by condensation which
  - (i) speeds evaporation
  - (ii) reduces inconvenience and
  - (iii) allows the equipment to be used for solvents other than water e.g. ethanol.
- (c) A receiver and vacuum pump can be fitted to the condenser, permitting operation under reduced pressure and, hence, at lower temperature.

**Disadvantages:**

- (a) Natural convection only
- (b) All the liquor is heated all the time
- (c) The heating surface is limited.

**Uses:**

- (i) Aqueous and other solvents may be evaporated
- (ii) Thermolabile materials can be evaporated under reduced pressure.
- (iii) Removing the still head it is convenient for evaporating extracts to dryness.

**SHORT TUBE EVAPORATOR (Basket type vertical short tube evaporator)****Construction and Principle****Construction**

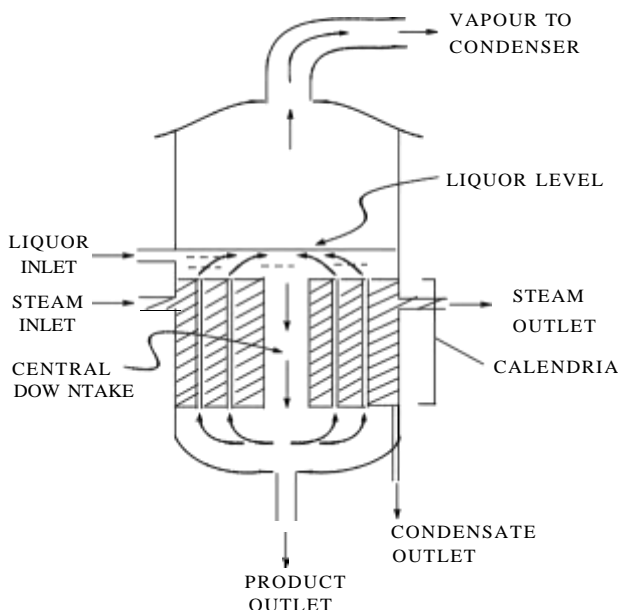
The evaporator is a cylindrical vessel. The lower portion of the vessel consists of a nest of tubes with the liquor inside and steam outside— this assembly is called *calendra*. The specifications of calendria are as follows:

Tube length: 1 – 2 m  
Tube diameter: 40 – 80 mm  
Diameter of evaporator: 2.5 m  
Number of tubes: 1000

The feed inlet is at the top of the calendra.  
The product outlet is placed at the bottom of the evaporator. Steam inlet and outlet is placed from the side of the calendra.

### Working

- The feed is introduced through the feed inlet and the liquor is maintained at a level slightly above the top of the tubes (of calendra), the space above this is left for the disengagement of vapor from the boiling liquor.
- The liquor in the tubes is heated by the steam and begins to boil, when the mixture of liquid and vapor will shoot up the tubes (in a similar manner to that of a liquid that is allowed to boil vigorously in a test-tube).
- This sets up a circulation, with boiling liquor rising up the smaller tubes of the calendra and returning down the larger central downtake.
- The product is collected through the product outlet.



### Advantages

1. Use of tubular calendra increases the heating area, possibly by a factor of 10 to 15 compared to that of an external jacket.
2. The vigorous circulation reduces boundary layers and keeps solids in suspension, so increasing the rate of heat transfer.
3. Condenser and receiver can be attached to run the evaporation under vacuum with nonaqueous solvents.

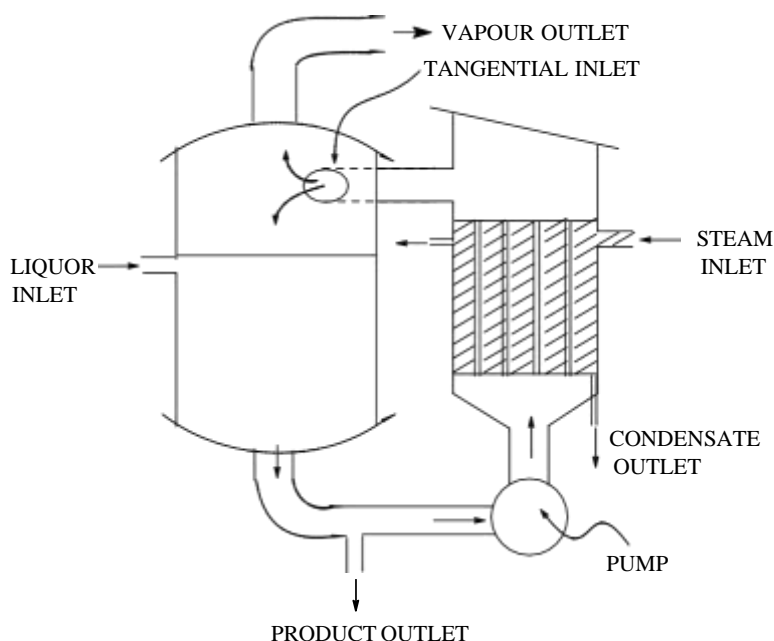
### Disadvantages

1. Since the evaporator is filled to a point above the level of the calendra, a considerable amount of liquid is heated for a long time. The effect of this continual heating can be reduced to some extent by removing concentrated liquor slowly from the outlet at the bottom of the vessel.
2. Complicated design, difficult for cleaning and maintenance.
3. The head (pressure) of the liquor increases pressure at the bottom of the vessel and, in large evaporators where the liquor depth may be of the order of 2 m; this may give rise to a pressure of about 0.25 bar, leading to elevation of the boiling point by 5 to 6°C.

**FORCED CIRCULATION EVAPORATORS**

Forced circulation evaporators are natural circulation evaporators with some added form of mechanical agitation. Different forms of forced circulation evaporators can be designed.

- An evaporating pan, in which the contents are agitated by a stirring rod or pole could be described as a forced circulation evaporator.
- A mechanically operated propeller or paddle agitator can be introduced into an evaporating pan or still.
- Propeller or paddle agitator can be introduced into the downtake of a short-tube evaporator.
- A typical forced circulation evaporator can be shown as follows:

**Construction**

The evaporator consists of a short tube calendria and a large cylindrical vessel (body of the evaporator) for separation of vapor and liquid takes place. The liquor inlet is provided at the side of the cylindrical vessel. A pump is fitted in between the calendria and the body of the evaporator. A tangential inlet for liquid under high pressure is placed at neck of the body of the evaporator. The vapor outlet is placed at the top of the body and it may be passed through a condenser to collect the condensed liquid.

**Working Principle**

Feed is introduced through the liquor inlet. Pump will force the liquid through the calendria. Steam heats the liquid inside the calendria. As it is under pressure in the tubes the boiling point is elevated and no boiling takes place. As the liquor leaves the tubes and enters the body of the evaporator through the tangential inlet there is a drop in pressure and vapor flashes off from the superheated liquor. The concentrated liquid is pumped out through the product outlet and the vapor is collected through the vapor outlet.

**Advantages**

- Rapid liquid movement improves heat transfer, especially with viscous liquids or materials that deposit solids or foam readily.
- The forced circulation overcomes the effect of greater viscosity of liquids when evaporated under reduced pressure.

- Rapid evaporation rate makes this method suitable for thermolabile materials, e.g. it is used in practice for the concentration of insulin and liver extracts.

### FILM EVAPORATORS

Film evaporators spread the material as a film over the heated surface, and the vapor escapes the film.

#### Long tube evaporators (Climbing film evaporators)

##### *Construction and working principle*

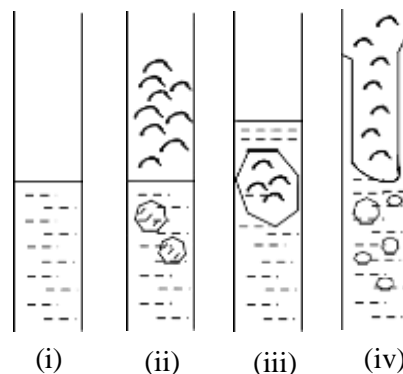
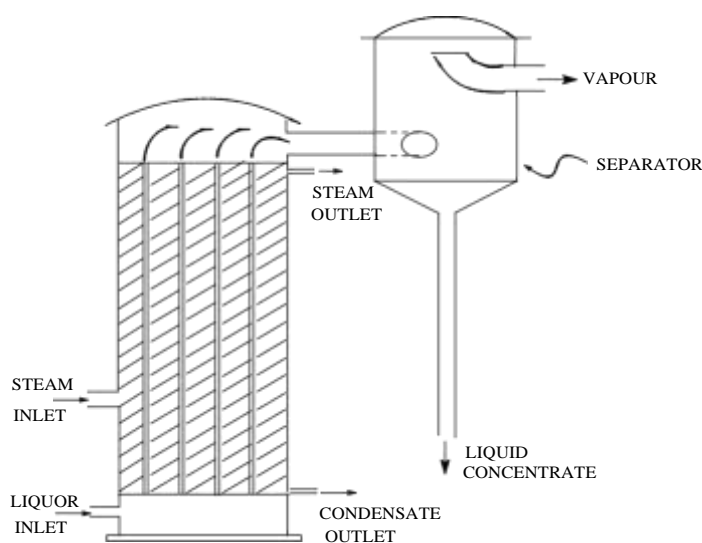
The heating unit consists of steam-jacketed tubes, having a length to diameter ratio of about 140 to 1, so that a large evaporator may have tubes 50 mm in diameter and about 7 m in length.

The liquor to be evaporated is introduced into the bottom of the tube, a film of liquid forms on the walls and rises up the tubes, hence it is called climbing film evaporator.

At the upper end, the mixture of vapor and concentrated liquor enters a separator, the vapor passes to a condenser, and the concentrated liquid to a receiver.

Cold or pre heated liquor is introduced into the tube (fig.-i). Heat is transferred to the liquor from the walls and boiling begins, increasing in vigor (fig.-ii). Ultimately sufficient vapor has been formed for the smaller bubbles to unite to a large bubble, filling the width of the tube and trapping a 'slug' of liquid above the bubble (fig.-iii).

As more vapor is formed, the slug of liquid is blown up the tube (fig.-iv), the tube is filled with vapor, while the liquid continues to vaporize rapidly, the vapor escaping up the tube and, because of friction between the vapor and liquid, the film also is dragged up the tube upto a distance of 5 to 6 metres.



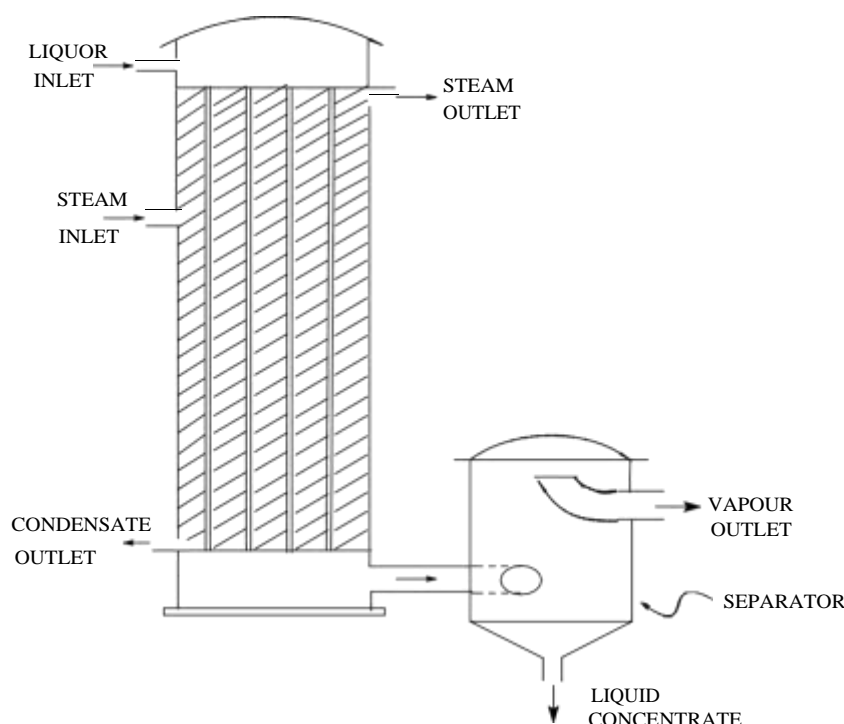
(i) (ii) (iii) (iv)

**Long tube evaporators (Falling film evaporators)***Construction and working principle*

The heating unit consists of steam-jacketed tubes, having a length to diameter ratio of about 140 to 1, so that a large evaporator may have tubes 50 mm in diameter and about 7 m in length.

The liquor to be evaporated is introduced at the top of the evaporator tubes and the liquor comes down due to gravity.

The concentrate and vapor leaves the bottom. They are separated in a chamber where the concentrate is taken out through product outlet and vapor from vapor outlet.

*Advantages of long tube evaporator(s)*

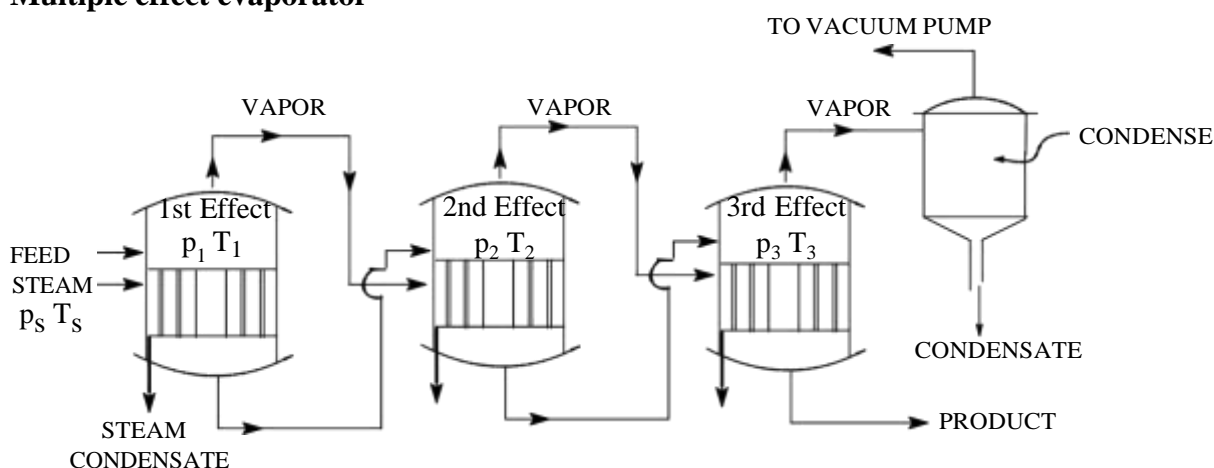
Since the movement of the film is assisted by gravity, more viscous liquid can be handled by falling film evaporator.

- (i) Very high film velocity reduces boundary layers to a minimum giving improved heat transfer.
- (ii) The use of long narrow tubes provides large surface area for heat transfer.
- (iii) Because of increased heat transfer efficiency, a small temperature gradient is necessary with less risk of damage to thermolabile materials.
- (iv) Although the tubes are long, they are not submerged, as in the short-tube evaporator; so that there is no elevation of boiling point due to hydrostatic head.

*Disadvantages*

- (i) Expense to manufacture and install the instrument is high.
- (ii) Difficult to clean and maintain.
- (iii) From the operational point of view the feed rate is critical. If too high, the liquor may be concentrated insufficiently, where as, if the feed rate is too low, the film cannot be maintained and dry patches may form on the tube wall.

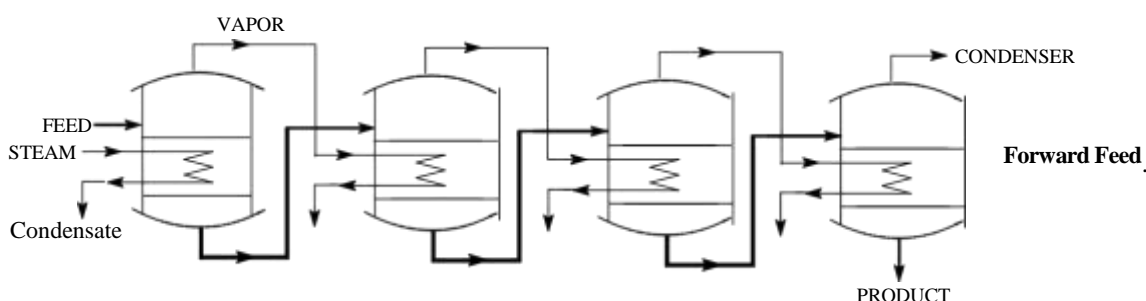


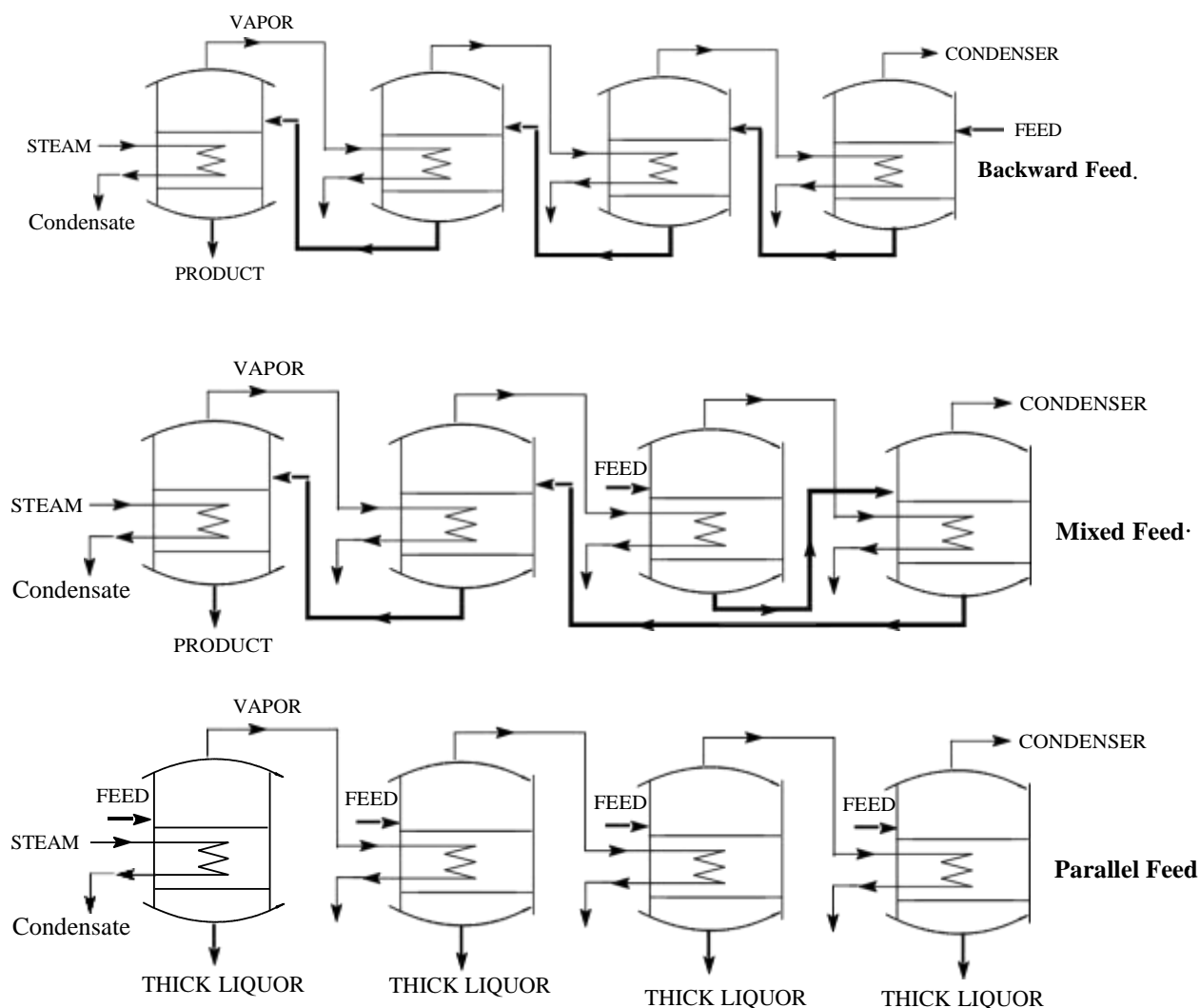
**Multiple effect evaporator**

*Triple-effect evaporator:  $p_s, p_1, p_2, p_3$  vapor pressures,  $T_s, T_1, T_2, T_3$  temperatures where  $p_s > p_1 > p_2 > p_3$ .*

In a single effect evaporator steam is supplied for heating the liquor. The total heat is not transferred from the steam. So the rest of the heat is wasted. To use that heat efficiently, connections are made so that the vapor from one effect serves as the heating medium for the next effect.

- (i) The dilute feed (liquor) enters the first effect, where it is partly concentrated; it flows to the second effect for additional concentration and then to the third effect for final concentration. This liquor is pumped out of the third effect.
- (ii) In the first effect raw steam is fed in which the vapor pressure in the evaporator is the highest,  $p_1$ . the second effect has the intermediate vapor pressure; i.e.  $p_1 > p_2 > p_3$ . This pressure gradient is maintained by drawing the vapor through a vacuum pump and condensing after the final effect.
- (iii) Depending on the lowering of vapor pressure boiling point of the liquids of 2nd and 3rd effect will also be lowered; i.e.  $T_1 > T_2 > T_3$ .
- (iv) In the 2nd effect vapor from the 1st effect ( $T_1$ ) is heating the liquor (having temperature  $T_2$ ). So there is a temperature gradient ( $T_1 - T_2$ ); consequently the liquor will be heated. Similar heating will be there in the 3rd effect also.

**Methods of feeding**



#### Forward feed

##### Advantages:

1. Feed moves from high pressure (in effect-2) to low pressure (in effect – 4) chambers, so pumping of liquor is not required.
2. Product is obtained at lowest temperature.
3. This method is suitable for scale-forming liquids because concentrated product is subjected to lowest temperature.

##### Disadvantages

It is not suitable for cold feed because, the steam input in effect-1 raises the temperature of the feed, and a small amount of heat is supplied as latent heat of vaporization. Therefore, amount of vapor produced will be less than the amount of steam supplied. Lower amount of vapor in effect-1 produces lower amount of vapor in the subsequent effects. Therefore, the overall economy is lower.

#### Backward feed

In backward-feed the feed enters in the last effect and moves towards first effect (i.e IV  $\rightarrow$  III  $\rightarrow$  II  $\rightarrow$  I).

##### Advantages

It is suitable for cold feed, because the heat used for increasing the temperature in IV effect is already used for heating 3 times. This will give more economy.

The method is suitable for viscous products, because highly concentrated product is at highest temperature, hence lower viscosity ( $\square$  higher heat transfer  $\square$  higher capacity)

#### Disadvantages

The liquid moves from low-pressure (IV) to high-pressure chambers (III  $\square$  II  $\square$  I) pumping is required.

#### Mixed feed method

The feed enters in the intermediate effect, moves forward and then backward to effect-I (III  $\square$  IV  $\square$  II  $\square$  I).

#### Advantages

- Liquid moves from high pressure (III) to low pressure (IV), hence no pump is required. Liquid moves from IV  $\square$  II  $\square$  I requires pump.
- Product is obtained from highest temperature (I) hence lowest viscosity.

#### Parallel feed

It is suitable where the feed has to be concentrated slightly.

### ECONOMY OF MULTIPLE EFFECT EVAPORATORS

*Assumptions:* (a) Feed is at boiling point and  
(b) Loss of heat is negligible

#### In effect-1

1 Kg of steam transfers its heat to feed. Since feed is at boiling point so the total amount of heat is used as latent heat of vaporization. Therefore, 1 kg steam will produce 1 kg vapor.

#### In effect – 2

1 Kg vapor from effect-1 will transfer heat to the liquor of effect -2. Here also 1 kg vapor produce 1 kg vapor from the liquor.

#### In effect – 3

1 Kg vapor from effect-II will produce 1 kg vapor in effect-3.

Therefore, 1 kg steam will produce 3 kg vapor.

Now, economy of a single effect evaporator =  $\frac{\text{vapor produced}}{\text{steam used}} = \frac{1\text{kg}}{1\text{kg}} = 1$

And economy of a triple effect evaporator =  $\frac{\text{vapor produced}}{\text{steam used}} = \frac{3\text{kg}}{1\text{kg}} = 3$

So for N number of effects economy will be N times that of a single effect evaporator.

### CAPACITY OF MULTIPLE EFFECT EVAPORATORS

Capacity = total evaporation per hour  
= vapor production rate

Capacity is also expressed in terms of total heat transferred because latent heats are nearly constant all over the ranges of pressure ordinarily involved.

The heat transferred in the three effects can be represented by the following equations:

$$q_1 = U_1 A_1 \Delta t_1.$$

$$q_2 = U_2 A_2 \Delta t_2. q_3$$

$$= U_3 A_3 \Delta t_3.$$

Total capacity will be found by adding these equations, giving:

$$q = q_1 + q_2 + q_3.$$

$$= U_1 A_1 \Delta t_1 + U_2 A_2 \Delta t_2 + U_3 A_3 \Delta t_3.$$

Assuming that all effects have equal areas  $A_1 = A_2 = A_3 = A$  (let) and that average coefficient  $U_{avg}$  can be applied to the system. The eqn (i) can be written as:

$$q = U_{avg} A (\Delta t_1 + \Delta t_2 + \Delta t_3)$$

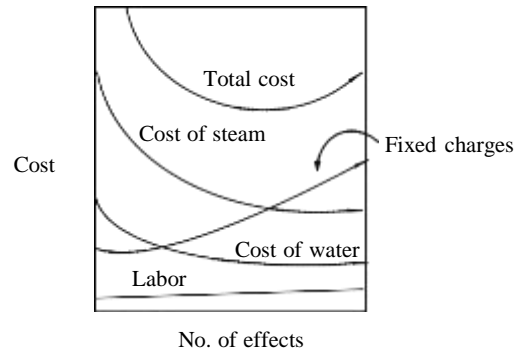
However the sum of individual temperatures drops equals the total over-all temperature drop between the temperature of the steam and the temperature in the condenser; therefore

$$q = U_{avg} A \Delta t$$

This is exactly the same as that of single effect.

#### Conclusion

It follows from this that if the number of effects of an evaporation system is varied and if the total temperature difference is kept constant, the total capacity of the system remains substantially unchanged.



### 3.DISTILLATION

Distillation may be defined as the separation of the constituents of a mixture including a liquid by partial vaporization of the mixture and separate and collect the vapor.

Such separation may include

- (i) one liquid from non-volatile impurities.
- (ii) one liquid from one or more other liquids, with which it may be miscible, partially-miscible or immiscible

N.B.

In practice it is difficult to distinguish between evaporation, distillation and drying.

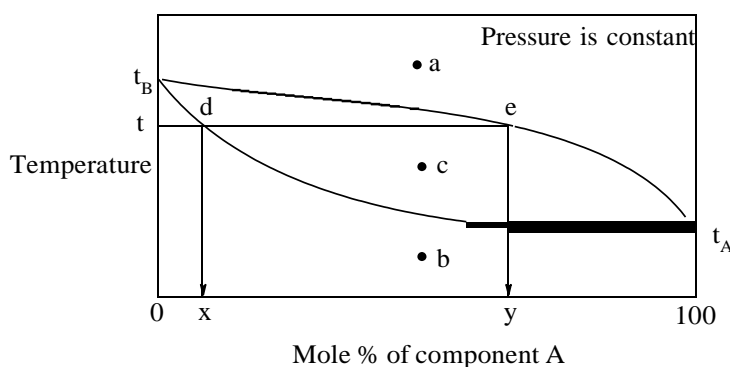
Based on the intention:

- (i) when condensation vapor is required the operation is called distillation
- (ii) when the concentrated liquid residue is required the operation is called evaporation.
- (iii) when the dried solid residue is required as product the process is called drying

### BOILING POINT DIAGRAM OF A BINARY MIXTURE

The figure represents the boiling point and equilibrium-composition relationship, at constant pressure.

Two liquids A (b.p.  $t_A$ ) and B (b.p.  $t_B$ ) are taken in a chamber of constant pressure. Now at any temperature the vapor composition and liquid composition will give two lines when plotted vs. temperature.

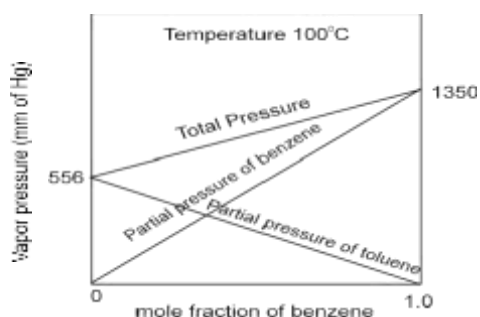


In boiling point diagram, temperatures are plotted as ordinates and compositions as abscissas.

- The diagram consists of two curves, the ends of which coincide with the b.p. of two components ( $t_A$  and  $t_B$ ).
- The upper-curve describes vapor composition and lower-curve liquid composition.
- At any temperature  $t$  the horizontal line cuts the vapor composition curve at 'e' which corresponds to vapor composition of  $y$  (mole% A) and cuts the liquid composition curve at 'd' which corresponds to liquid composition of  $x$  (mole% of A). So any two points on the same horizontal line (such as d and e) represent compositions of liquid and vapor in equilibrium at temperature ' $t$ '.
- For all points above the top line (such as point 'a') the mixture is entirely vapor.
- For all points below the bottom line (such as point 'b') the mixture is completely liquefied.
- For all points between the two curves (such as point 'c') the system consists partly of liquid and partly of vapor.

### RAOULT'S LAW

Raoult's law states that, any particular temperature, the partial pressure of one component of a binary mixture is equal to the mole fraction of that component multiplied by its vapor pressure in the pure state at this temperature.



e.g. to illustrate Raoult's law, let us consider the case of benzene and toluene mixture.

At a temperature of 100°C toluene has a vapor pressure of 556 mm. Consequently, if partial pressure is plotted against composition, the partial pressures of toluene at various compositions will fall along a straight line from 556 mm for pure toluene to zero for pure benzene. At this same temperature benzene has vapor pressure of 1350 mm, and its vapor pressure will change linearly from zero for 0% benzene to 1350 mm for pure benzene.

The total pressure for any composition will be the sum of the two partial pressures at that composition.

If the partial pressures are straight lines i.e. Raoult's law holds then the total pressure will be a straight line between 556 mm for pure toluene and 1350 mm for pure benzene.

#### Derivation

Two liquids A and B are at constant temperature.

Liquid A is more volatile than B.

If Raoult's law holds for this binary mixture then

$$\begin{aligned} \text{from Raoult's law} \quad p_A &= P_A x & (1) \\ \text{and} \quad p_B &= P_B (1 - x) \end{aligned}$$

where  $p_A$  and  $p_B$  = partial pressure of A and B respectively

$P_A$  and  $P_B$  = vapor pressure of pure A and B

$x$  = mole fraction of A in the solution

If  $P$  represents the total pressure, then

$$\begin{aligned} P &= p_A + p_B & (2) \\ &= P_A x + P_B (1 - x) \end{aligned}$$

From Dalton's law

$$\begin{aligned} y &= \frac{p_A}{p_A + p_B} & \text{where } y = \text{mole fraction of A in vapor phase} \\ &= \frac{P_A x}{P} & [\text{from (1) and (2)}] \end{aligned}$$

#### Example

The vapor pressures of benzene and toluene are as given in the table. Assuming that mixtures of benzene and toluene obey Raoult's law, calculate and plot the boiling-point diagram for this pair of liquids at 760mm total pressure.

#### Solution:

Let us take one temperature 180°F

So at 180°F,  $P_A = 811$  Hg

$P_B = 314$  mm Hg

We have to calculate the mole fraction of benzene in liquid ( $x$ ) and in vapor ( $y$ ).

From the eqn.:

$$P = P_A x + P_B (1 - x)$$

$$\text{or, } 760 = 811 x + 314 (1 - x)$$

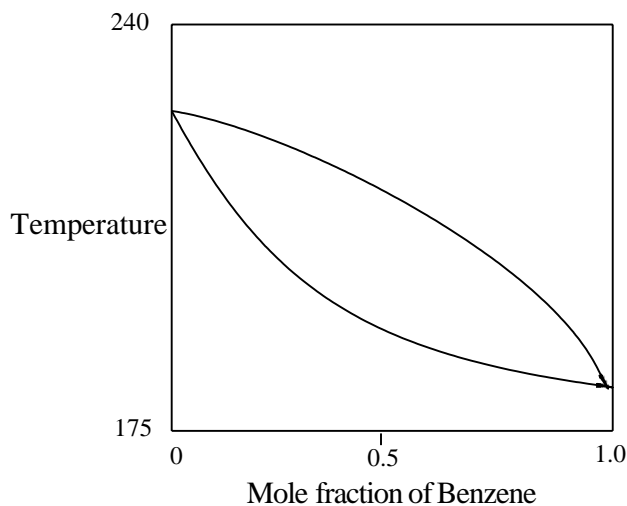
Temp °F	Vapor mm Hg pressure,	
	Benzene $P_A$	Toluene $P_B$
176.2	760	314
180	811	345
185	882	378
190	957	414
195	1037	452
200	1123	494
205	1214	538
210	1310	585
215	1412	635
220	1520	689
225	1625	747
230	1756	760
231.1	—	—

or,  $x = 0.897$

$$\text{From eqn. } y = \frac{P_A x}{P} = \frac{811 \times 0.897}{760} = 0.958$$

Similarly for all temperature values corresponding  $x$  and  $y$  values may be calculated:

Temp. °F	$x$	$y$
185	0.773	0.897
190	0.659	0.831
195	0.555	0.757
200	0.459	0.678
205	0.370	0.591
210	0.288	0.496
215	0.211	0.393
220	0.141	0.281
225	0.075	0.161
230	0.013	0.031



Boiling point diagram for benzene-toluene system at 1 atm pressure

### RELATIVE VOLATILITY

For a more volatile phase in equilibrium with a liquid phase, the relative volatility of component A (the more volatile component) with respect to component B is defined by the equation:

$$\alpha_{AB} = \frac{y_A / x_A}{y_B / x_B}$$

where

$\alpha_{AB}$  = relative volatility of component A with respect to component B

$y$  = mole fraction of component A in vapor phase

$x$  = mole fraction of component in liquid phase

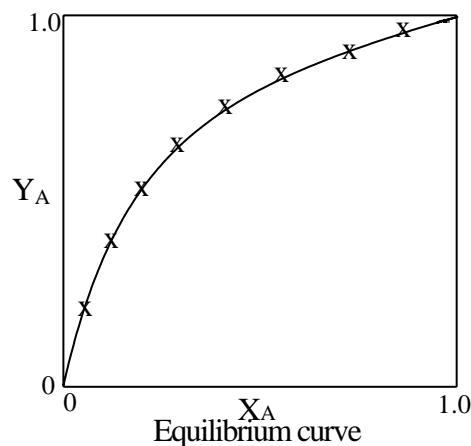
In case of binary system,  $y_B = 1 - y_A$  and  $x_B = 1 - x_A$ .

Substituting,

$$\alpha_{AB} = \frac{y_A}{x_A} \cdot \frac{x_B}{y_B} = \frac{y_A}{x_A} \cdot \frac{1 - x_A}{1 - y_A}$$

Rearranging we get 
$$Y_A = \frac{\alpha_{AB} X_A}{1 + (\alpha_{AB} - 1) X_A}$$

$X_A$						
$Y_A$						



### Equilibrium curve

If  $\alpha_{AB}$  is given then from the above equation a set of  $X_A$  and  $Y_A$  can be calculated. When  $Y_A$  is plotted against  $X_A$  the curve is called *equilibrium curve*.

If the liquid phase obeys Raoult's law and the vapor phase obeys Dalton's law then,

$$y_A = \frac{P_A x_A}{P}$$

$$\text{So } \alpha_{AB} = \frac{P_A x_A / P}{P_B (1 - x_A) / P} \cdot \frac{1 - x_A}{x_A} = \frac{P_A}{P_B}$$

**Example**

Construct an equilibrium curve for binary system of benzene – toluene from the given data.

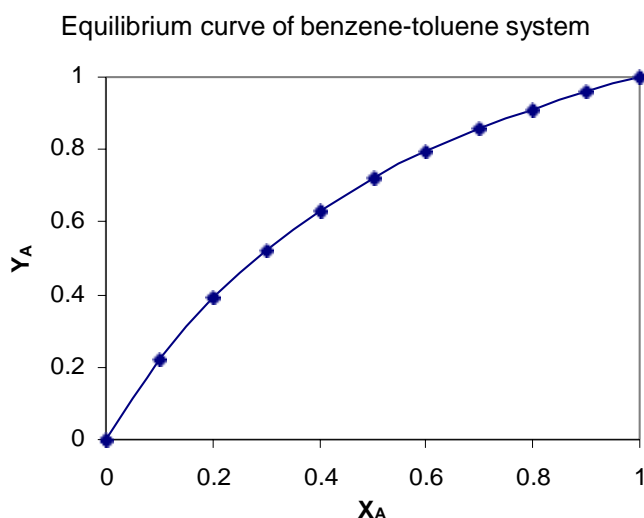
Data	Boiling point at 1 atm	Vapor pressure of benzene ( $P_A$ )	Vapor pressure of toluene ( $P_B$ )	$\alpha_{AB} = \frac{P_A}{P_B}$
Benzene	80.1°C	760 mm	270 mm	2.81
Toluene	110.6°C	1780 mm	760 mm	2.34

Therefore, average relative volatility over the temperature range 80.1 to 110.6°C

$$\alpha_{AB(\text{avg})} = \frac{2.81 + 2.34}{2} = 2.57$$

$$\text{Therefore, } Y_A = \frac{2.57 X_A}{1 + (2.57 - 1) X_A} = \frac{2.57 X_A}{1 + 1.57 X_A}$$

$X_A$	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
$Y_A$	0.222	0.391	0.524	0.631	0.720	0.794	0.857	0.911	0.959

**HENRY'S LAW**

The partial pressure of a component over a solution is proportional to its mole fraction in the liquid. This can be expressed as  $p_A = C x$

where  $p_A$  = partial pressure of component A

$x$  = mole fraction of A in liquid phase

$C$  = Henry's law constant;  $C$  is constant only at constant temperature

N.B. Raoult's law is essentially a special case of Henry's law where the constant  $C$  in equation is the vapor pressure of the pure component.



## DISTILLATION METHODS

### A. Distillation methods for miscible liquid systems

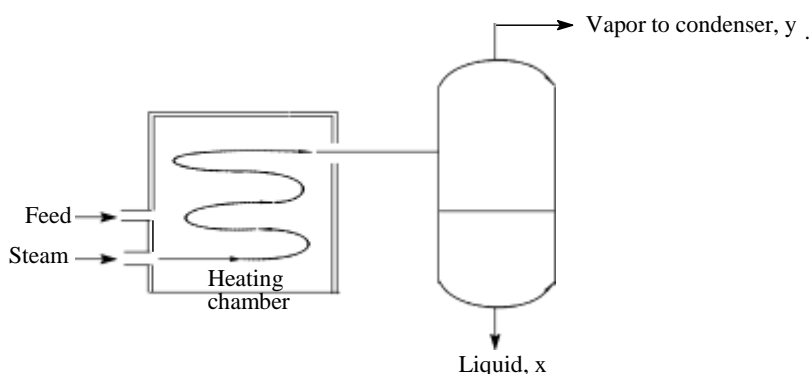
1. Equilibrium or Flash Distillation
2. Simple or Differential Distillation
3. Fractional Distillation
4. Distillation under reduced pressure (e.g. Molecular Distillation)
5. Special Distillation Methods for non-ideal mixtures
  - (a) Distillation of Azeotropic Mixtures
  - (b) Extractive Distillation

### (B) Distillation of immiscible liquids (e.g. Steam Distillation)

## 1. EQUILIBRIUM DISTILLATION / FLASH DISTILLATION

There are two types of distillations that do not involve rectification

- (a) Equilibrium distillation or flash distillation and
- (b) Simple or differential distillation.



(a) Equilibrium distillation or flash distillation

This is a single stage operation where a liquid is partially vaporized, the vapors are allowed to come in equilibrium with the residual liquid and the resulting vapors and liquid are separated. *Use:* This method is used only when the difference between volatilities of two components is very large

Let us consider a binary system whose components are A and B. A is more volatile.

- Feed:  $W_F$  = number of moles of liquid fed  
 $x_F$  = mole fraction of component A in feed
- Suppose  $V$  moles are vaporized in an equilibrium-distillation process.

Now in

Liquid phase      Number of moles left in liquid phase =  $(W_F - V)$  moles  
Let the composition of the residual liquid =  $x$  mole fraction of A

Vapor phase      Composition of vapor phase =  $y$  mole fraction of A  
Number of moles gained =  $V$

Form material balance equation with respect to A

Moles of A at start = Moles of A in vapor phase + Moles of A in liquid phase

$$W_F x_F = V y + (W_F - V) x$$

(i)

In this case all the parameters are known except  $x$  and  $y$ .

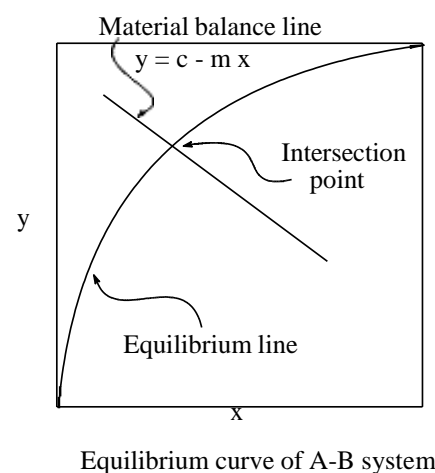
2nd equation required for solving is obtained from equilibrium curve of the A, B system.

Eqn(i) is a straight line,

$$\text{or, } y = \frac{W_F x_F - (W_F - V) x}{V}$$

$$\text{or, } y = c - m x$$

Plotting this equation in the equilibrium curve the point of intersection is obtained. The value of  $x$  and  $y$  can be obtained from the point of intersection.



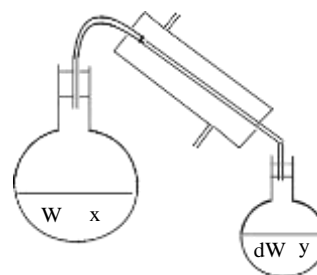
Equilibrium curve of A-B system

## 2. SIMPLE / DIFFERENTIAL DISTILLATION

In this process vapor is removed from the system as soon as it is formed and condensed.

Use:

- This method is commonly used in laboratory
- In industries it is only used for systems having high relative volatilities.



### Derivation of Raleigh's equation

Let us consider a batch of  $W_0$  moles of liquid was taken at the beginning.

Suppose at any given time during distillation there are  $W$  moles of liquid left in the still.

At this time let the mole fraction of A in liquid is  $x$ .

Suppose a very small amount of liquid  $dW$  is vaporized. In the vapor phase the mole fraction of component A is  $y$ .

At a given time

After a moment

$$\ln \frac{W_1}{W_0} = \int_{x_0}^{x_1} \frac{dx}{y}$$

Total moles of liquid present                       $W$                        $W - dW$

Moles of A present in liquid

$Wx$

$(W - dW)(x - dx)$











Therefore a material balance equation with respect to A will be

$$xW = (W - dW) (x - dx) + ydW$$

or,  $xW = xW - x dW - W dx + dW dx + y dW$

$dWdx$  is very small hence ignoring the term the equation will be

$$y dW - x dW = W dx$$

$$\text{or, } (y - x)dW = W dx$$

$$\frac{dW}{W} =$$

or,  $dx$



$y \propto x$

	Time = 0	Time = $t_1$ .
Amount of liquid in the still (moles)	$W_0$ .	$W_1$ .
Moles of component A in liquid	$x_0$ .	$x_1$ .

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$$W_1 dW = X_1 dx$$

or,

$$W = y \cdot x$$

0

0





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The function  $y = \int x$

can be integrated graphically from the equilibrium curve, since the

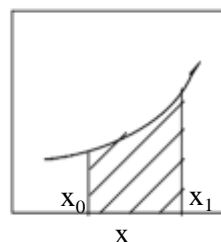
curves gives the relationship between  $x$  and  $y$ .



N.B.

$x$	$y$	$y-x$	$1/(y-x)$
--	--	----	-----
--	--	----	-----

$$\frac{1}{y-x}$$



### Application of Raleigh's Equation

1. By using the Raleigh's equation the effectiveness of simple distillation for a given system can be estimated. [Ref: A.B.Paradkar, *Introduction to Pharmaceutical Engineering*, p. 247]
2. It is used in determination of cut-off point when we can stop distillation as soon as the vapor composition falls below the required purity of the product.

### SIMPLE DISTILLATION

#### Objective

Simple distillation is the process of converting a liquid into its vapors which, are passed through a cooling surface to condense the vapors. The condensed vapors are reformed into liquid which, is collected in a receiver.

#### Apparatus for laboratory scale

It consists of a distillation flask with a side arm sloping downward that is connected to a condenser. The condensed vapors are collected in a flask called 'receiver'. The whole apparatus is made of glass.

A thermometer is fitted in the distillation flask to note down the temperature at which, the vapors are distilled.

Bumping is avoided by adding small pieces of porcelain or porous pot before distillation.

#### Apparatus for preparation of purified water

The boiler may be made of cast iron but the baffles and the condenser tubes that comes into contact with product are made of stainless steel or monel metal.

The cold water from the water tap enters the still through the inlet, which rises in the jacket fitted with a constant level device, the excess of water over flow through the outlet. A portion of hot water at 90 to 95°C enters into the boiler through a narrow opening – the level of water is maintained in the boiler up to overflow level.

The water is boiled in the boiler by means of heating coils. On heating, the dissolved gases in the condenser are allowed to escape through a small opening and only the steam

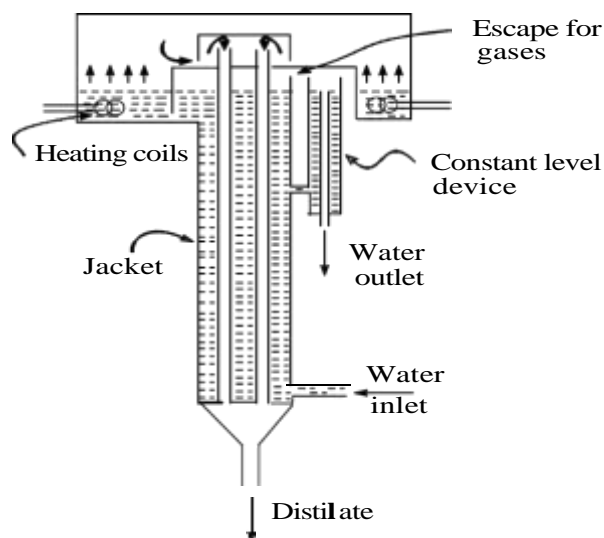


Fig. Distillation unit for purified water

escapes into the condensing tubes.

Since the dissolved gases are more volatile than water they escape in the first portion of the distillate, therefore, must be rejected. Similarly, the last portion may contain volatile portion of the dissolved solid substances in tap water – hence, discarded.

Application of simple distillation in pharmacy

- I. It is used for the preparation of distilled water and water for injection.
- II. Many volatile oils and aromatic waters are prepared by simple distillation e.g. Spirit of nitrous ether and Aromatic Spirit of Ammonia
- III. Concentration of liquid and to separate non-volatile solid from volatile liquids such as alcohol and ether.

### 3. FRACTIONAL DISTILLATION / RECTIFICATION

A rectifying unit consists primarily of

- (a) a still or reboiler, in which vapor is generated,
- (b) a rectifying or fractionating column through which this vapor rises in counter-current contact with a descending stream of liquid, and
- (c) a condenser, which condenses all the vapor leaving the top of the column, sending part of this condensed liquid (the reflux) back to the column to *descend* counter to the rising vapors, and delivering the rest of the condensed liquid as product.

As the liquid stream descends the column, it is progressively enriched with the less volatile constituent.

The top of the column is cooler than the bottom, so that the liquid stream becomes progressively hotter as it descends and the vapor stream becomes progressively cooler as it rises. This heat transfer is accomplished by actual contact of liquid and vapor, and for this purpose effective contact is desirable.

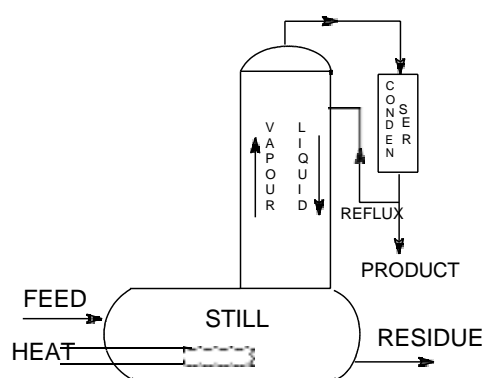


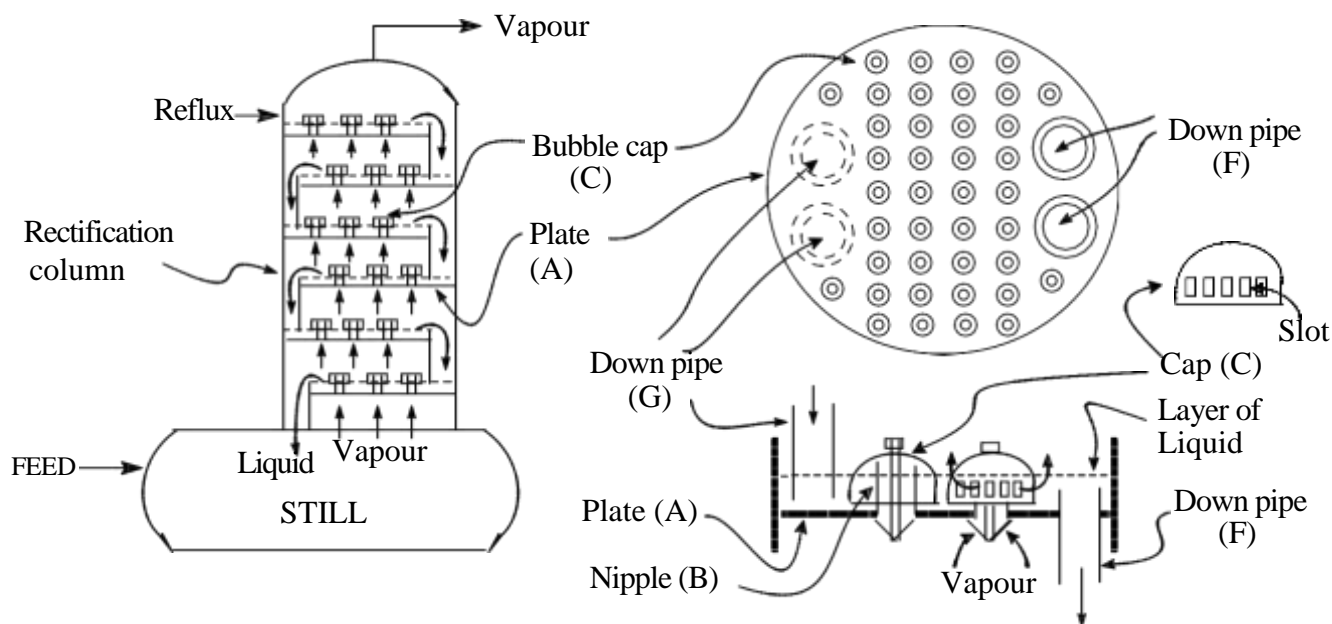
Diagram of still and fractionating column

#### CONSTRUCTION OF RECTIFYING COLUMN

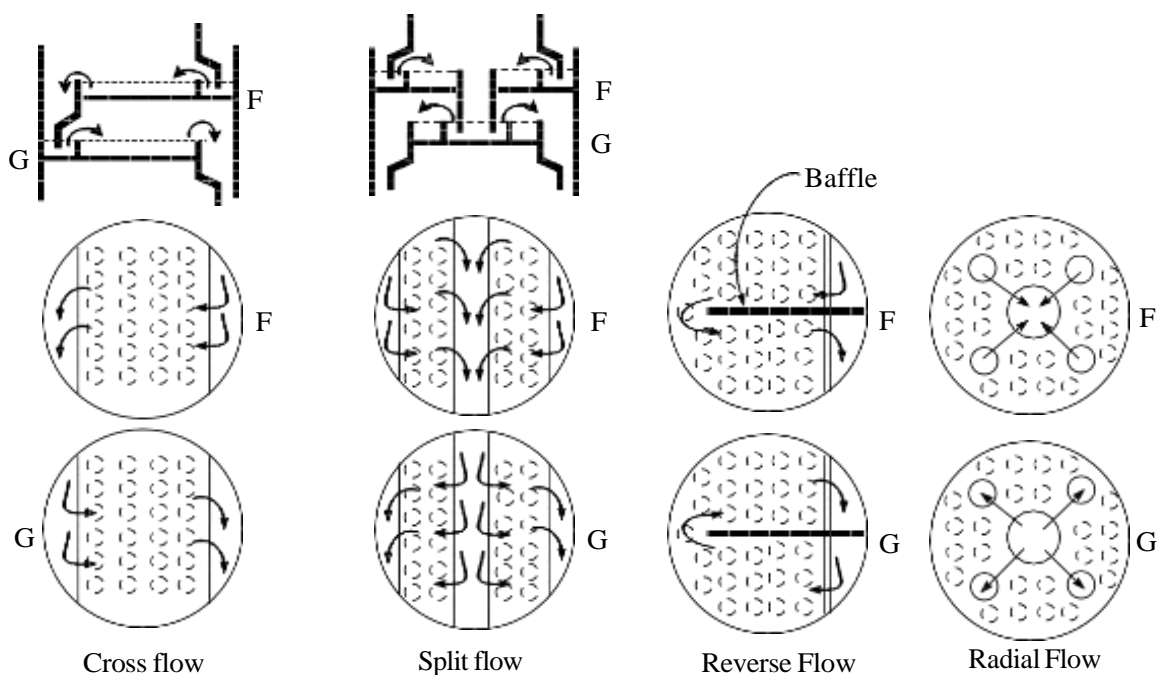
There are different varieties of equipments for rectification

- (a) Plate column                      (i) Bubble cap column
- (ii) Sieve-plate column
- (b) Packed column

## BUBBLE-CAP COLUMN



- The column is divided into sections by means of a series of *horizontal plates A*.
- Each plate carries a number of short *nipples B* (or *riser*). Each nipple is covered by a bell-shaped *cap C* that is secured by a spider and bolt with the plate. The edge of the cap is *serrated* or the sides may be *slotted*.
- Vapor rises from the plate below through the nipple, is diverted downward by the cap, and bubbles out under the serration or through the slots.
- A layer of liquid is maintained on the plate by means of an *overflow* or *down-pipe (F)* and the depth of the liquid is such that the slots are submerged.
- The *down-pipe, (G)* from the plate above, is sealed by the liquid on the plate below, so that the vapor cannot enter the down-pipe.
- Ordinarily, the liquid is delivered at one end of a diameter by the down-pipe from the plate above, flows the other end of the same diameter.

**Types of down-comers****(a) Cross flow**

The liquid flows across the plate from right to left on plate F and left to right on plate H and so on down the column.

**(b) Split flow**

On plate F the liquid flows from the two sides to the center. On plate H it flows from the center to the two sides and so on down the column. This arrangement is commonly known as split flow.

**(c) Reverse flow**

Liquid comes down the space on one side of the baffle and flows across the plate from right to left, around the end of the baffle, from left to right and down the space behind the weir. This arrangement is called reverse flow.

**(d) Radial flow with circular down-take**

One plate will have four or more down-comers around the circumference, and the next plate will have a down-comer at the center so that on the upper plate the flow is from the circumference towards center and on the next plate the flow is from the central down-take to the circumference.

**Specification of bubble cap rectification column**

Column diameter 2 to 15 ft

Height few feet to over 100 ft

Bubble cap diameter 3 to 6 inches

Slots in a 3 inches bubble cap may be  $\frac{1}{8}$  to  $\frac{3}{32}$  inch wide  
 $\frac{1}{2}$  to 1 inch height

**SIEVE PLATE COLUMNS**

All the constructions are same as bubble cap columns. Instead of bubble cap plates, flat plates with a large number of relatively small perforations, drilled in them are used. These perforations are usually  $3/16$  to  $1/4$  inch in diameter.

The velocity of the vapor through these holes is sufficient to produce the liquid running down the holes.

**PACKED COLUMNS**

The column is entirely filled with some sorts of material that offers a large surface area supposedly wetted by the liquid.

A large variety of materials are used among which **Raschig rings** are popular. A Raschig ring is a hollow cylinder whose length is equal to its diameter. This may be made of metal (by sawing sections off a pipe), stone ware, ceramics, carbon, plastics, or other materials. Raschig rings are usually dumped at random in the column.

**Advantages**

- (i) Have a low pressure drop per unit of height than bubble cap
- (ii) For very small diameters of column, where it would be difficult to get in more than two or three bubble caps, a packed column can be used.
- (iii) Since Raschig rings can be made of any material, hence packed columns can be used for corrosive materials.
- (iv) The amount of liquid held up in the column is low so thermolabile liquid remains in contact with high temperature for a short time than bubble cap method.

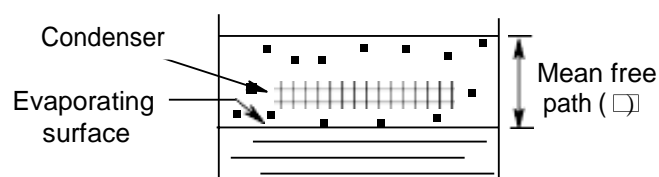
**Disadvantages**

- (i) They are relatively inflexible.
- (ii) Distribution of liquid uniformly in such packed column is difficult. It is found that, as the liquid passes down the tower it tends to concentrate at the walls and leave the center dry.

**4. DISTILLATION UNDER REDUCED PRESSURE / VACUUM DISTILLATION****Theory**

Liquid boils when its vapor pressure is

equal to the atmospheric pressure. Liquids, which are decomposed at their boiling point under atmospheric pressure, can be distilled at a much lower temperature than its boiling point if the pressure is reduced on the surface of the liquid. Boiling under reduced pressure will also increase the rate of distillation.

**Molecular Distillation***Theory / Principle of Molecular Distillation*

In a high vacuum distillation operation, where the material distills from an evaporating surface to a relatively cool condensing-surface. The conditions are such that, the mean free path of the distilling molecules is greater than the distance between the evaporating and condensing surface.

- The vacuum applied in these types of apparatus is about 1 mm Hg pressure or less.

Mean free path is defined as the average distance traveled by the molecules in a straight line without any collision. It can be calculated by Clausius law:

$$\lambda = \frac{1}{\sqrt{2} n d^2 N}$$

where,  $\lambda$  = mean free path (cm)

d = diameter of the molecules (cm)

N = number of molecules in 1 cm<sup>3</sup> volume.

N.B.

Temperature (°C)	Volume (litre)	Pressure (mm Hg)	Number of molecules
0	22.4	760	6.023 x 10 <sup>23</sup> .
0	22.4	1 x 10 <sup>-3</sup> .	7.9 x 10 <sup>17</sup> .

It is clear from the above equation and chart that, mean free path can be increased, by reducing the number of molecules per cm<sup>3</sup> volume. The molecules evaporate from the surface and travel few cm without colliding with the molecules of the residual gas in the space above. If now the condensing surface is placed within distance, a major fraction of the molecules will condense and will not return to the distilland. Thus each molecule distills itself and hence called “*Molecular Distillation*”.

#### Characteristics of molecular distillation

1. Molecules having molecular weight within the range of 300 to 1100 dalton can be distilled by this method. [N.B. Low molecular weight (below 300 dalton) molecules will re-evaporate again from the condenser surface. High molecular weight molecules (greater than 1100 dalton) will not have sufficient volatility.]
2. The molecules to be distilled should reach the surface and evaporate. The molecules at the bottom of the distilland have to overcome the pressure of the layer above, to come to the surface. Hence, the layer should be thin and should be in a state of turbulent motion to facilitate the molecules to reach the surface.
3. The distilland should be degassed before entering in the still, because at very low pressure the dissolved gas will occupy all the space and rate of distillation will be reduced.

#### Falling Film Molecular Still

The vessel has a diameter of the order of 1 m and the walls are heated suitably by a heating jacket. Vacuum pumps are connected by a large diameter

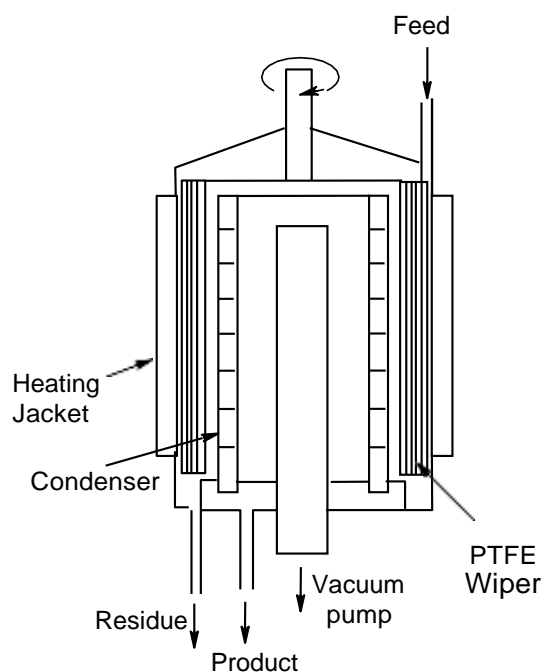


Fig. Falling film Molecular Still

pipe. The feed flows down the walls and is spread to a film by the polytetrafluoroethylene (PTFE) wipers which move about 3 m/s giving a film velocity of about 1.5 m/s. The residue is collected at the bottom of the vessel and it is re-circulated (through the feed line).

The evaporated molecules are then condensed on the condenser surface. The condensate is taken out as product.

### Centrifugal molecular still

The distilland (feed) is introduced on to the center of a bucket-shaped vessel (1 to 1.5 m in diameter) that rotates at high speed. The film of liquid that is formed moves outwards over the surface of the vessel to the residue-collection pipe. The vessel is heated by radiant heaters. Condensers and a collection device are located close to the inner surface of the rotor.

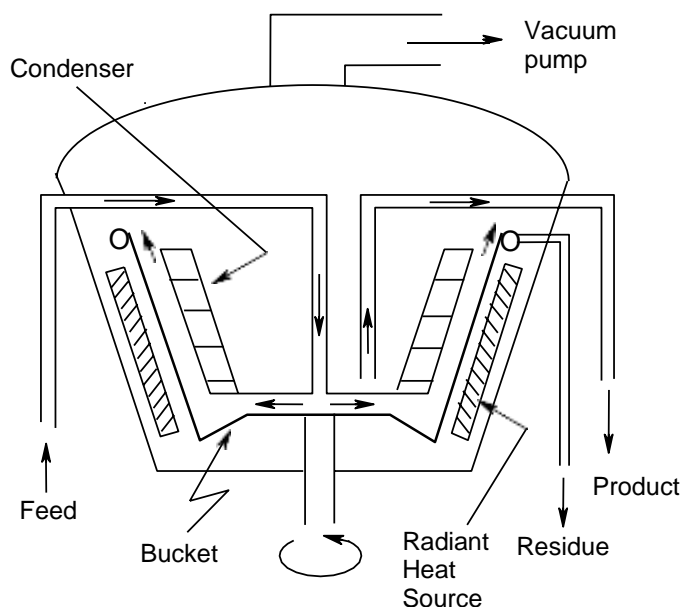


Fig. Centrifugal molecular still

### Application of vacuum distillation in pharmacy

#### 1. Vitamin concentrates

Vitamin A,D,E,K and tocopherols are obtained from vegetable and fish oils. The vitamin-A concentrate produced by molecular distillation is very pure and has good stability. As no chemical is used in this method which could split the ester linkage, the vitamins are retained in the natural ester form which is the most stable form of vitamin A. The stability of the concentrates is further enhanced by natural antioxidants distilling over from the original oil.

#### 2. The fractionation of oil

The fractionation of oils into various components is carried out by molecular distillation.

Components	Molecular Weight	Temperature Range
(a) Fatty acids, unsaponifiable matter of low molecular weight.	150 – 300	50 – 140°C
(b) Unsaponifiable matter like sterols, vitamins, dyes, waxy alcohols, monoglycerides	300 – 600	150 – 190°C
(c) Triglycerides, sterol esters, vitamin esters, resins, waxes	600 – 900	Above 190°C

#### 3. Purification and fractionation of lanolin

It is used to get various fractions from Lanolin like, cetyl alcohol, cholesterol, ceryl alcohol, lanopalmitic acid, ischolesterol etc.

#### 4. Separation of Poly Ethylene Glycol (PEG)

On laboratory scale it is used to separate PEG according to the degree of polymerization.

### 5. SPECIAL DISTILLATION METHODS FOR NON-IDEAL MIXTURES

#### Industrial scale distillation of Azeotropic Mixture

The liquor from fermentation process is a common source of ethanol and contains approximately 8–10% ethanol.

After simple distillation an azeotrope will form containing 95.6% (96E+4W) ethanol and boiling at  $78.15^{\circ}\text{C}$  at atmospheric pressure.

In this type of system a reboiler is used instead of boiler. The feed liquor is introduced into the system and must occur at a point where the equilibrium will not be disturbed. Hence, feed will take place, at a place part of the way up the column, where the equilibrium composition on the plate is similar to the feed composition.

The plate below the *feed plate* form the stripping section where the rising vapor strips the more volatile component (ethanol) from the feed liquor while the upper section is known as the *rectifying section*.

The binary azeotrope produced at this stage is freed from water by making use of ternary azeotrope – ethanol, benzene, and water.

The ethanol/water azeotrope, with sufficient benzene (only required at start-up) is fed to column A and the pure ethanol is obtained as bottom product, since the ternary azeotrope takes off the water.

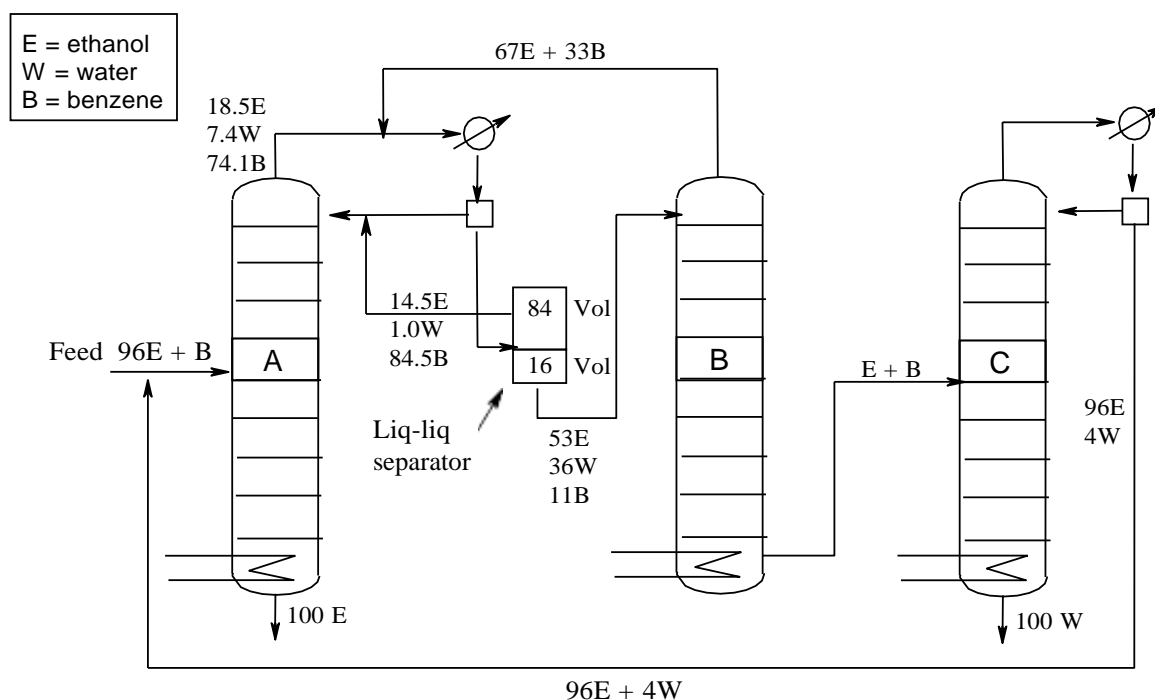


Fig. Plant for manufacture of Absolute ethanol (100% ethanol)

- The azeotrope (E+B+W) is taken from the top of the column A, condensed and separated (in liquid-liquid separator) into two layers, having the compositions given in the diagram.
- The upper layer predominates and, being rich in benzene (14.5E+1.0W+84.5B), is returned to column A. The lower layer (53E+36W+11B) is taken to column B, where the



benzene is recovered as the ethanol/benzene binary azeotrope (67E+33B) and is mixed with the vapor from ethanol.

- The ethanol / water residue passes to column C, where the ethanol is recovered as the ethanol/water binary azeotrope (96E+4W), which can be incorporated with the original feed.
- The final product from column A is 100% ethanol and from column C is 100% water.

## 6. DISTILLATION OF IMMISCIBLE LIQUIDS

### Steam distillation

Steam distillation is used for the distillation of two immiscible liquids one of which is water.

*Application:*

- (i) Separation of volatile oil e.g. eucalyptus oil, rose oil, clove oil etc. and
- (ii) Preparation of some aromatic water e.g. concentrated rose water..

### Theory

Volatile oils are mixtures of high molecular weight compounds having low vapour pressure (i.e. high b.p.). To separate these from the natural sources like petals of flowers, barks etc. it is not possible to take them to their boiling points around 200°C. If these oils are distilled with water (low molecular weight but high vapour pressure i.e. low b.p.) then volatile oil will be distilled out at a temperature below 100°C.

$$\frac{\text{Weight of volatile oil in distillate}}{\text{Weight of water in distillate}} = \frac{M_v P_v}{M_w P_w}$$

Where,  $M_w$  and  $M_v$  are molecular weights of water and volatile oil respectively.

$P_w$  and  $P_v$  are vapor pressure of water and volatile oil respectively.

- The aqueous phase of distillate that is collected is water saturated with volatile oil i.e. called *aromatic water*.

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N.B. When a mixture of two practically immiscible liquids are heated, while being agitated to expose the surfaces of both liquids to the vapor phase, each component independently exerts its own vapor pressure as a function of temperature as if the other constituent was not present.

Boiling begins and distillation may be effected when the sum of the partial pressures of the two immiscible liquids just exceeds the atmospheric pressure.

An immiscible liquid and water independently boils at high temperature but when steam is passed through a mixture of these liquids (agitation) it boils at a much lower temperature than the boiling point of water.

*Example:* Turpentine oil has a boiling point of about 160°C, when mixed with water it can be distilled at about 95.6°C if steam is passed through it.

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**Large scale apparatus**

This consists of a still having a mesh near the bottom. The steam is generated by boiling water below the mesh. The steam passes through the materials (to be extracted) packed over the mesh. The vapor containing volatile oil is then passed to the condenser. The distillate is collected in *Florentine receivers*. Florentine receiver separates the oil and water depending on their densities. The aqueous phase may be re-circulated again to avoid loss of volatile oil in water.

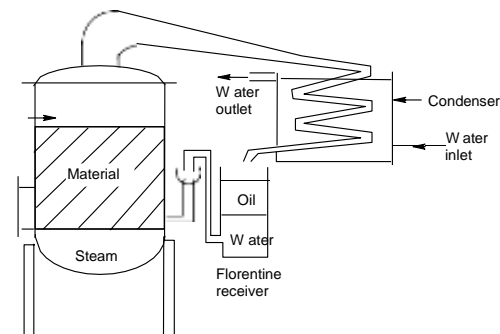


Fig . Steam distillation apparatus

**Florentine Receiver**

It is used for the separation of oil and water.

Florentine receivers are of two types:

**Type-I** Used for separation of oil heavier than water.

**Type-II** Used for separation of oil lighter than water.

**Type-I** receiver has two taps. The tap fitted near the bottom of the vessel is used for collecting oil, whereas the tap fitted near the top of the vessel is used for water to overflow.

**Type-II** receiver is fitted with siphon at the bottom that works when it gets filled with water

whereas the tap fitted near the top is an outlet for the flow of oil.

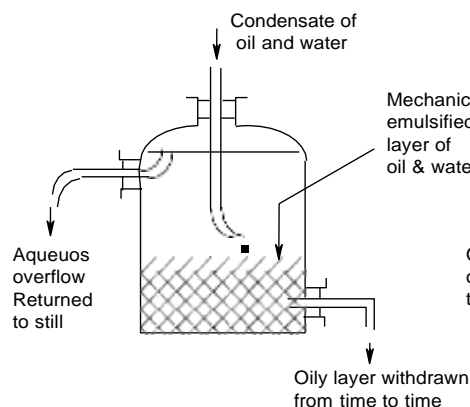


Fig. Florentine receiver for oils heavier than water

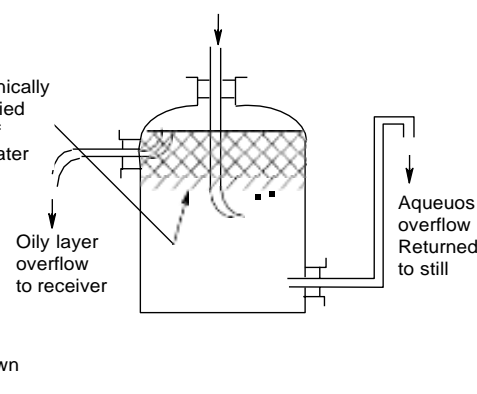


Fig. Florentine receiver for oils lighter than water

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