

Distillation

by

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Lecture 2: Enthalpy Concentration Diagrams

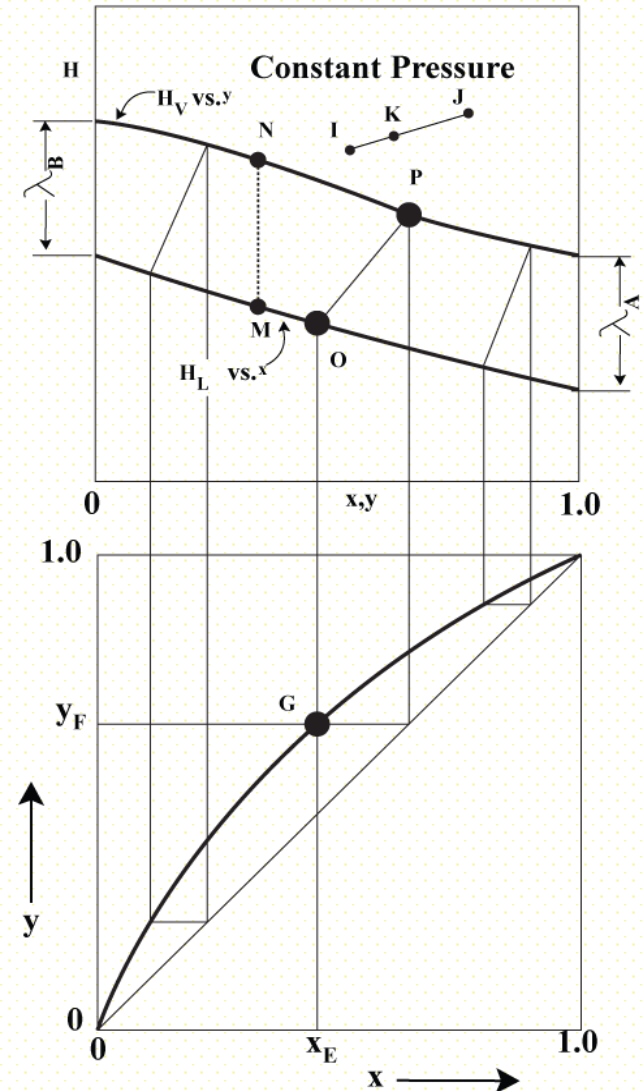
$$H_L = C_L (T_L - T_0) M_{avg} + \Delta H_s$$

$$H_V = y \{ C_{pA} M_A (T_L - T_0) + \lambda_A M_A \} + (1 - y) \{ C_{pB} M_B (T_G - T_0) + \lambda_B M_B \}$$

From the balance equations we can obtain the following

$$\frac{H_K - H_I}{z_K - z_I} = \frac{H_J - H_K}{z_J - z_K}$$

- LHS gives the slope of section IK and RHS gives the slope of section JK.
- So the points I, J and K are collinear.
- Thus, the point K is on the straight line IJ.
- If mixture J is removed adiabatically from mixture K, then the mixture I would result.

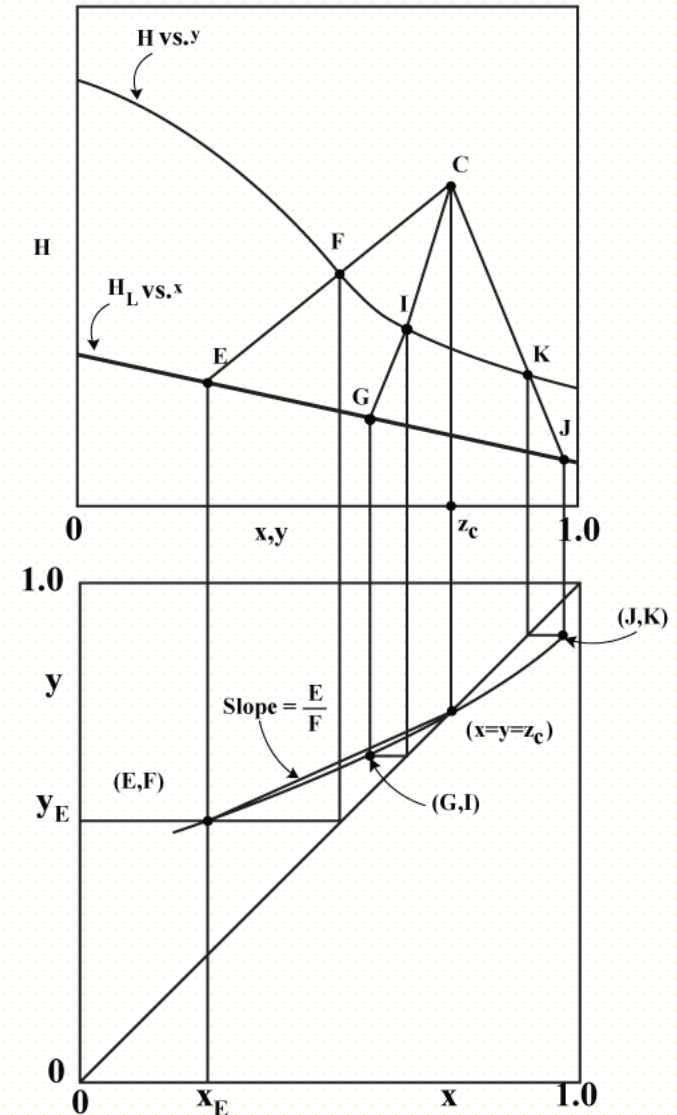


Lecture 2: Enthalpy Concentration Diagrams

- Consider now a mixture C.
- It will be useful such a mixture in terms of saturated vapors and liquids, since distillation is mostly concerned with such mixture.
- C can be considered the result of adiabatically removing saturated liquid E from saturated vapor F (EF is not a tie line). x_E and y_E can be located on the lower diagram as shown.
- But C can equally be considered as having been produced by adiabatically subtracting G from I, or J from K, or indeed by such a combination of saturated liquids and vapors given by any line from C with intersects the saturated enthalpy curves.
- These, when projected to the lower diagram, form the curve shown here.
- Thus any point C on the Hxy diagram can be represented by the difference between the saturated vapors and liquids and in turn also by a curve on the xy plot.

For the combination $F-E = C$, a material balance shows

$$\frac{E}{F} = \frac{z_C - y_E}{z_C - x_E} = \frac{\text{line } CF}{\text{line } CE}$$

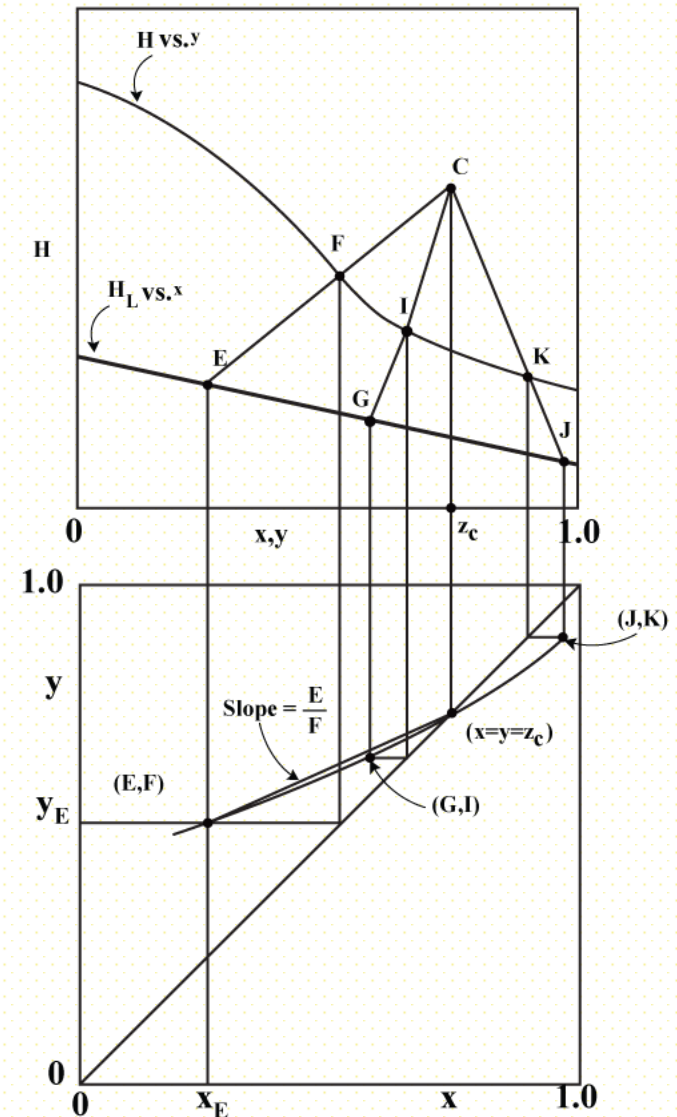


Lecture 2: Enthalpy Concentration Diagrams

- This is the equation on the xy diagram of the chord of slope E/F drawn between point (y_E, x_D) and $y=x=z_C$ on the 45° line.
- Similarly, the ratios F/G and J/K would be shown by the slopes of chords drawn from these points to $y=x=z_C$.

Consideration of the geometry of the diagram will readily show the following:

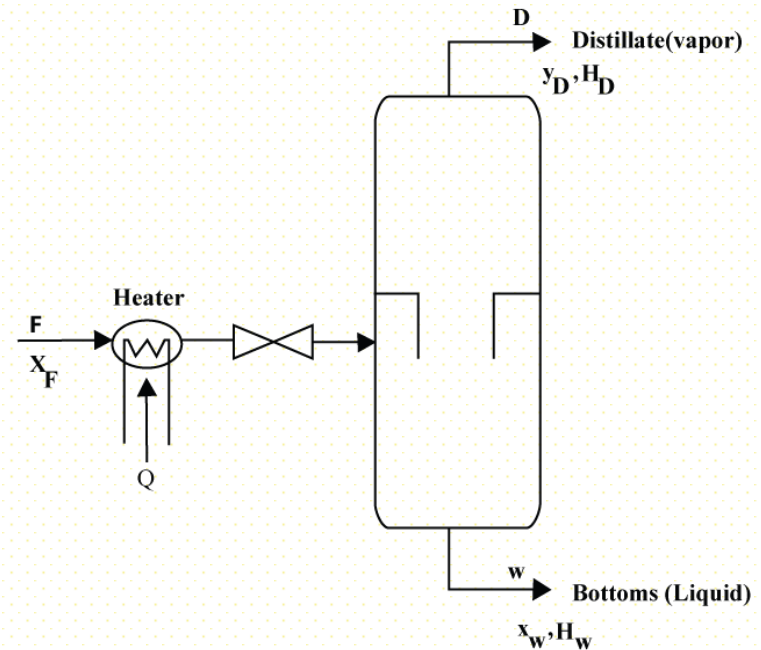
1. If the H_{vy} and H_{Lx} curves are straight parallel lines (which will occur if the molar latent heats of A and B are equal, if the heat capacities are constant over the prevailing temperature range, and if there is no heat of solution), then $E/F = G/I = J/K$ for adiabatic subtraction, since the line segment ratios are then equal, and the curve on xy representing C becomes a straight line.
2. If point C is moved upward the curve on xy becomes steeper, ultimately coinciding with the 45° line when C is at infinite enthalpy.
3. If point C is on the H_{vy} curve, the curve on xy will become a horizontal straight line; If point C is on the H_{Lx} curve, the curve on xy will become a vertical straight line



Lecture 3: Flash Distillation

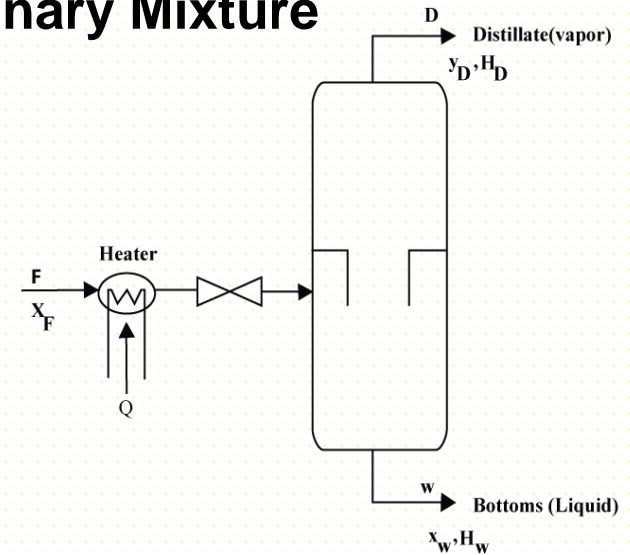
Definition & Purpose:

- Flash distillation (sometimes called “flash vaporization” or "equilibrium distillation") is a single stage separation technique.
 - A liquid mixture feed is pumped at higher pressure through a heater to raise the temperature and enthalpy of the mixture.
 - Mixture flows to the flash drum through a throttling valve under reduced pressure, causing the liquid to partially vaporize.
 - Once the mixture enters the "flash drum", the liquid and vapor separate.
 - The vapor and liquid are allowed to reach equilibrium.
 - Vapor and liquid phases are separated and removed from the system
-
- ✓ Separation by flash vaporization are very common in industry, e.g., petroleum refining.
 - ✓ Even when some other method of separation is to be used, it is not uncommon to use a "pre-flash" to reduce the load on the separation itself.
 - ✓ When designing a flash drum it is important to provide enough space in the drum for disengagement of liquid and vapor. Drums can be designed as cyclone type.



Lecture 3: Flash Distillation of a Binary Mixture

- Consider a binary mixture of components A and B.
- Flow rates, composition and enthalpy of feed are: F , z_F , H_F
- Flow rates, composition and enthalpy of distillate (top product) are: D , z_D , H_D
- Flow rates, composition and enthalpy of bottom product are: W , z_W , H_W
- Let Q be the rate of supply of heat to the heat exchanger.



Following are the assumptions:

1. No heat losses to surroundings, 2. Ideal gas behavior for vapor, 3. Perfect mixing

Materials and energy balance equations for a steady-state flash vaporization unit:

Total material balance: $F = D + W$ (1)

Component balance: $Fz_F = Dy_D + Wx_W$ (2)

Energy balance: $FH_F + Q = DH_D + WH_W$ (3)

Lecture 3: Flash Distillation of a Binary Mixture

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Energy balance: $FH_F + Q = DH_D + WH_W$ (3)

Using equations (2) and (1),
we have

$$\begin{aligned} (D + W)z_F &= Dy_D + Wx_W \\ \Rightarrow D(z_F - y_D) &= W(x_W - z_F) \\ \Rightarrow -\frac{W}{D} &= \frac{y_D - z_F}{x_W - z_F} \end{aligned} \quad (4)$$

Using equations (3) and (1),
we have

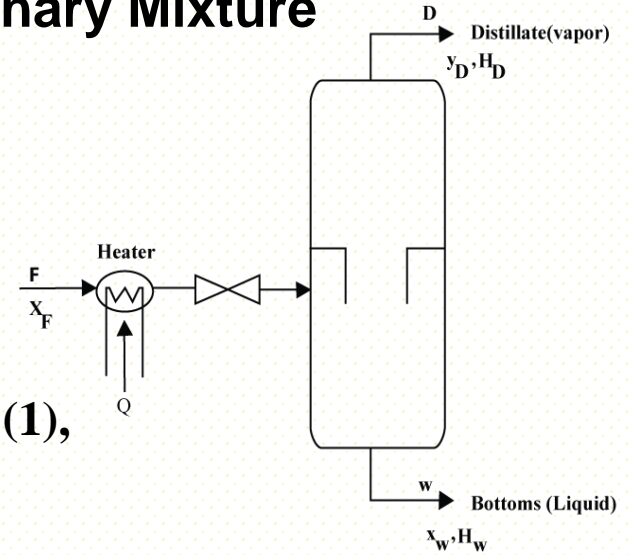
$$\begin{aligned} FH_F + Q &= (F - W)H_D + WH_W \\ \Rightarrow \frac{W}{F}(H_D - H_W) &= H_D - \left(H_F + \frac{Q}{F}\right) \end{aligned} \quad (5)$$

Similarly, using equations
(3) and (1) again, we have

$$\begin{aligned} FH_F + Q &= DH_D + (F - D)H_W \\ \Rightarrow \frac{D}{F}(H_W - H_D) &= H_W - \left(H_F + \frac{Q}{F}\right) \end{aligned} \quad (6)$$

Dividing eq (5) by eq (6) we have,

$$-\frac{W}{D} = \frac{H_D - (H_F + Q/F)}{H_W - (H_F + Q/F)} \quad (7)$$



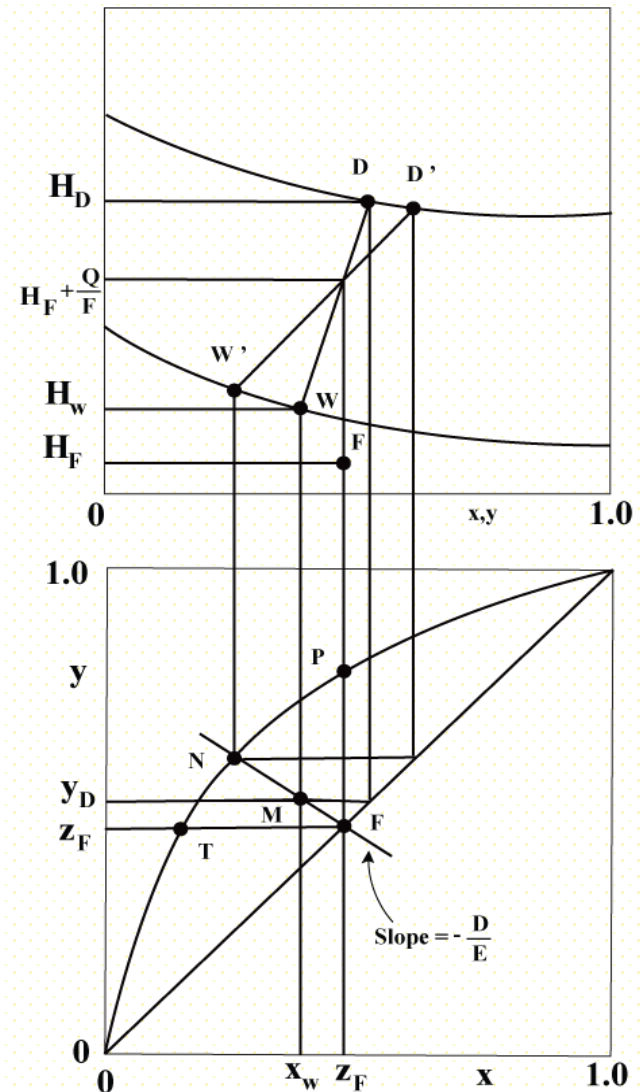
Lecture 3: Flash Distillation of a Binary Mixture

$$-\frac{W}{D} = \frac{H_D - (H_F + Q/F)}{H_W - (H_F + Q/F)} \quad (7)$$

On the Hxy diagram, eq 7 represent equation of straight line through points D (H_D, y_D), W (H_W, x_W) and ($H_F + Q/F, z_F$) feed mixture after it leaves the heat exchanger.

$-\frac{W}{D}$ and $\frac{y_D - z_F}{x_W - z_F}$ are the slopes of operating line

If the effluent streams are in equilibrium, the product D' and W' would be the tie line.



Lecture 3: Flash Distillation of a Binary Mixture

We'll consider a different rearrangement of the steady state model. These will be useful in some of the solution methods.

Let the *fraction vaporized* of the feed as $f=D/F$.

Material balance can be rewritten as: $1 = \frac{D}{F} + \frac{W}{F} = f + \frac{W}{F} \Rightarrow \frac{W}{F} = 1 - f$ (8)

Component balance can then be written as: $z_F = \frac{D}{F} y_D + \frac{W}{F} x_W = f y_D + (1 - f) x_W$ (9)

There are two unknown in eq (9). A second equation between the unknown must be available. This is provided by equilibrium curve or equation based on relative volatility, α . For binary system:

$$\alpha_{AB} = \frac{y_A / x_A}{y_B / x_B} \quad \text{where } x_A = 1 - x_B \text{ and } y_B = 1 - y_A$$

The above equation can be rearranged to get:

$$y = \frac{\alpha x}{1 + (\alpha - 1)x}$$

Lecture 3: Flash Distillation of a Binary Mixture

Calculation Techniques

- To solve a flash distillation problem, one simultaneously solves the operating and equilibrium equations.
- Flash calculations can be solved directly, but usually require an iterative solution.
- Graphical techniques are also common.
- Often, the choice of technique depends on the available form of the equilibrium relationship.

EXAMPLE: A mixture of liquid containing 50 mole % n-hexane and 50% n-heptane is subjected to flash distillation at 1 atm total pressure and 40°C to vaporize 50 mol% of feed. The relative volatility of n-hexane in the mixture is 2.36. Calculate the composition of vapor and liquid leaving the flash chamber considering an equilibrium stage.

Lecture 3: Flash Distillation of a Binary Mixture

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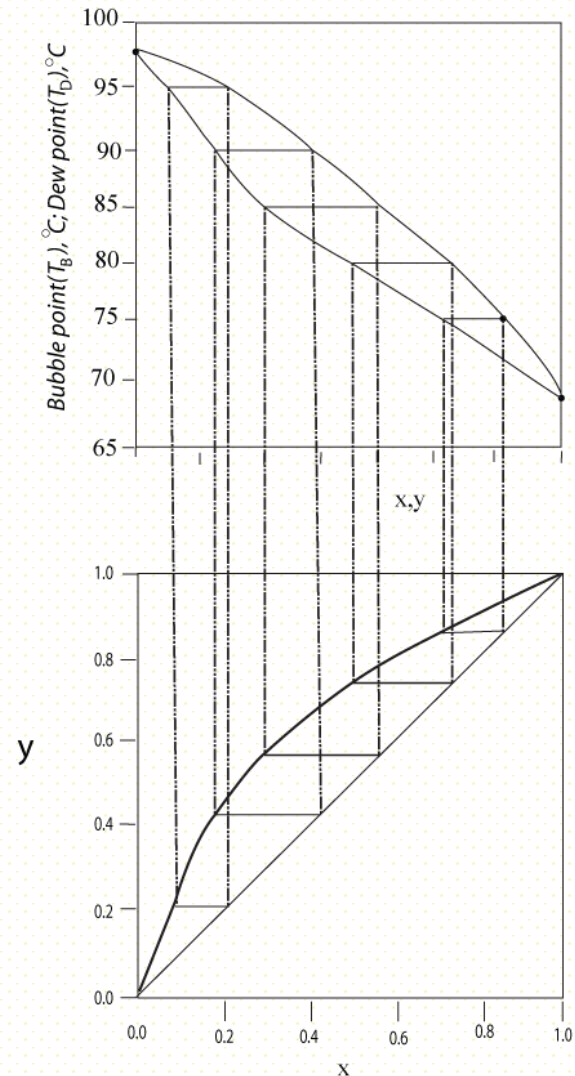
Solution:

Given: $x_F=0.5$, $D=50$, $W = 50$, $-W/F = -1.0$, $\alpha=2.36$

$$y = \frac{\alpha x}{1 + (\alpha - 1)x}$$

x	alpha	y
0	2.36	0
0.076	2.36	0.162558
0.199	2.36	0.369609
0.341	2.36	0.54979
0.508	2.36	0.709027
0.705	2.36	0.849398
1	2.36	1

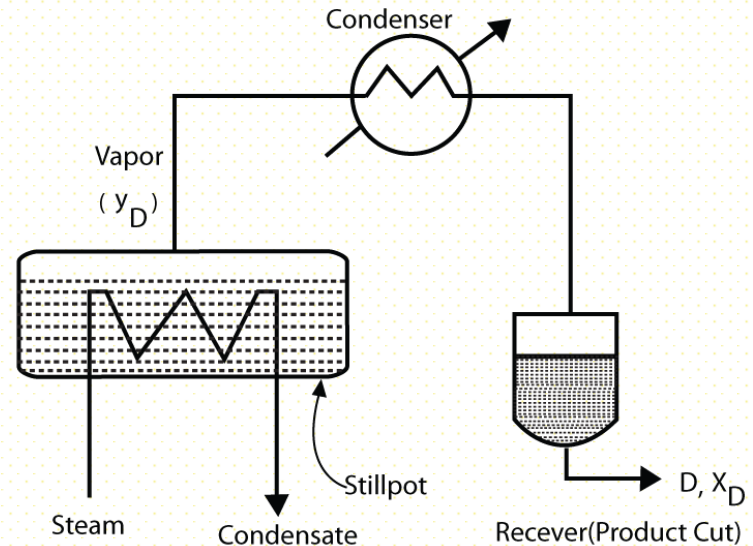
1. Draw the xy diagram
2. Then from xy diagram draw bubble and dew point diagram (Txy)



$y_D = 0.65$ mol fraction of hexane
 $x_W = 0.35$ mol fraction, $T = 82^\circ\text{C}$

Lecture 4: Batch Distillation

- In batch distillation, a tank is charged with feed and then heated using steam jacket or steam coil.
- Vapor flows overhead, is condensed and collected in a receiver.
- The liquid remaining in the tank is generally called the *residue*. T
- The composition of the material collected in the receiver varies with time, so the composition of the product is an average of all the material collected.
- Often, the receiver will be emptied or switched several times during a distillation to collect separate *cuts* of product.



A batch process is inherently dynamic -- it cannot be modeled steady state.

Batch distillation can be conducted with or without reflux. When reflux is used, any of several different operating *policies* may be used -- you might use a constant reflux rate, you might vary it, etc.

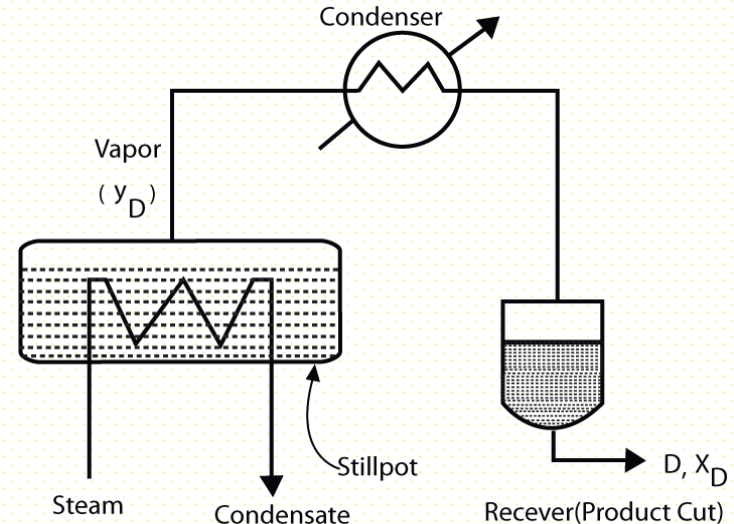
Lecture 4: "Differential" Distillation of a Binary Mixture

Batch distillation is most common:

- in small capacity plants
- when feed or products vary widely and frequently (as in specialty chemical production)
- for test runs on new products
- when the feed is the result of batch processing
- when the process requires frequent cleaning which would interrupt continuous processing

We want to consider one main variant -- without reflux. Other variant --with reflux will be considered later.

Batch distillation without reflux is often called *differential distillation*. Because there is no reflux, the vapor product is assumed in equilibrium with the liquid residue in the tank at any given time.



Lecture 4: " Differential" Distillation of a Binary Mixture

Let L = number of moles of liquid in still pot at any time t

x = mol fraction of more volatile component A

D = moles of condensate accumulated

y^* = concentration of equilibrium vapor

Differential mass balance is as follows:

Total material balance: $-dL = dD$ (1)

Component A balance: $-d(Lx) = y^* dD$

$$\Rightarrow -Ldx - x dL = y^* dD$$

Using eq.1:

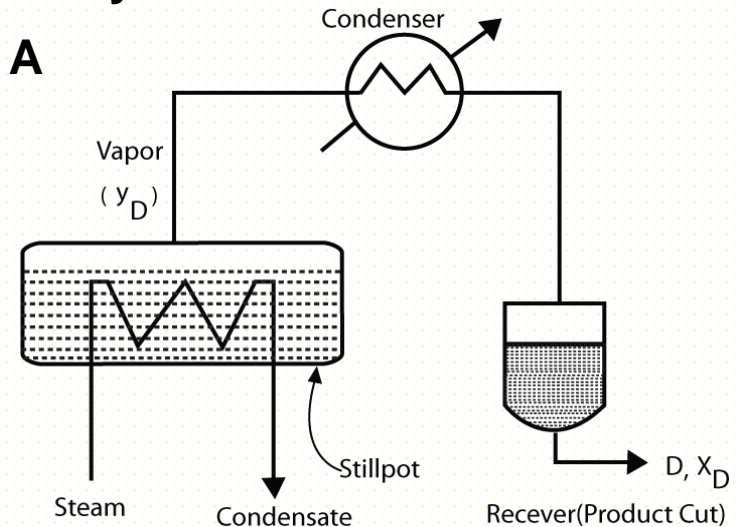
$$-Ldx + x dD = y^* dD$$

$$\Rightarrow -Ldx = (y^* - x) dD$$

Now again using eq.1

$$-Ldx = (y^* - x) dL$$

$$\Rightarrow \frac{dL}{L} = \frac{dx}{(y^* - x)}$$



If F = amount of feed, moles

z_F = concentration of feed

W = moles of residue

x_W = concentration of more volatiles in the residue

Then,

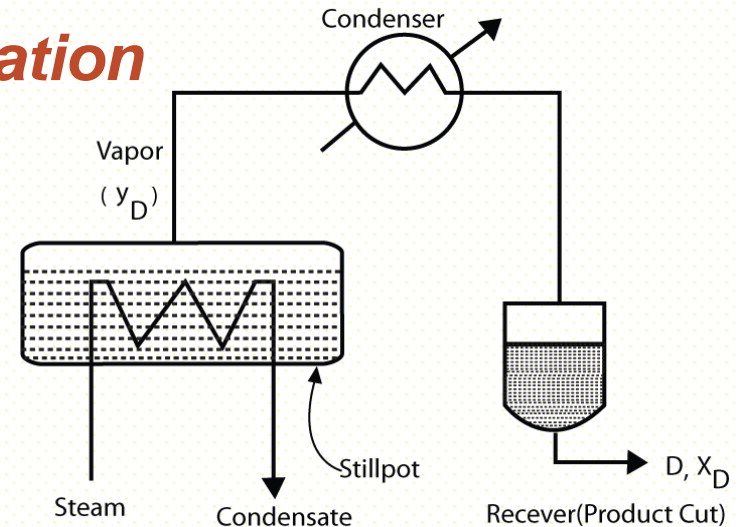
$$\int_F^W \frac{dL}{L} = \int_{z_F}^{x_W} \frac{dx}{(y^* - x)} \quad \Rightarrow \ln \frac{F}{W} = \int_{x_W}^{z_F} \frac{dx}{(y^* - x)}$$

This is the **Rayleigh Equation** which relates the amount of residue to the composition.

Lecture 4: " Differential" Distillation of a Binary Mixture

$$\ln \frac{F}{W} = \int_{x_W}^{z_F} \frac{dx}{(y^* - x)} \quad \text{Rayleigh Equation}$$

- If equilibrium data (x - y^*) are available in tabular form, graphical integration of the RHS is possible
- If algebraic relation between x and y is available then analytical integration is possible.



Example $y^* = \frac{\alpha x}{1 + (\alpha - 1)x}$

$$\ln \frac{F}{W} = \int_{x_W}^{z_F} \frac{dx}{\left(\frac{\alpha x}{1 + (\alpha - 1)x} - x \right)} = \frac{1}{\alpha} \ln \frac{z_F}{x_W} \left(\frac{1 - x_W}{1 - z_F} \right) + \ln \left(\frac{1 - x_W}{1 - z_F} \right)$$

More convenient form

$$\ln \frac{F z_F}{W x_W} = \alpha \ln \frac{F}{W} \left(\frac{1 - z_F}{1 - x_W} \right)$$

Average conc. ($y_{D,avg}$) of the accumulated distillate can be obtained by material balance:

$$F z_F = D y_{D,avg} + W x_W \quad \text{and} \quad F = D + W$$

Lecture 4: " Differential" Distillation of a Binary Mixture

EXAMPLE: A liquid mixture of 150 mol containing 40 mole% n-hexane and 60 mole% n-heptane is to be batch-distilled at 1 atm total pressure.

- (a) If 50 mol is distilled, what is the average composition of the distillate and the composition of the liquid left in the still?
- (b) If the accumulated vapor is 90% n-hexane, calculate the amount of distillate. The relative volatility of the n-hexane in the mixture is 2.36.

SOLUTION: (a) Given, $F = 150$ mol, $Z_F = 0.4$, Distillate, $D = 50$, $W = 100$ mol, $y_{D, avg} = ?$

$$\ln \frac{FZ_F}{Wx_W} = \alpha \ln \frac{F}{W} \left(\frac{1 - Z_F}{1 - x_W} \right)$$

\Rightarrow

$$FZ_F = Dy_{D, avg} + Wx_W$$

\Rightarrow

\Rightarrow

After solving

\rightarrow Composition of the Distillate

Lecture 4: " Differential" Distillation of a Binary Mixture

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- (a) If 50 mol is distilled, what is the average composition of the distillate and the composition of the liquid left in the still?
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SOLUTION: (b) Given, $y_{D,avg} = x_D = 0.9$, **D=?**

We know, $150 = D + W$

$$\begin{aligned}\Rightarrow 150 * 0.4 &= Wx_w + D * 0.9 \\ &= Wx_w + (150 - W) * 0.9\end{aligned}$$

$$\Rightarrow 60 = Wx_w + (150 - 0.9W)$$

$$\Rightarrow W(0.9 - x_w) = 75$$

$$\Rightarrow W = \frac{75}{(0.9 - x_w)}$$

$$\ln \frac{FZ_F}{Wx_w} = \alpha \ln \frac{F}{W} \left(\frac{1 - Z_F}{1 - x_w} \right)$$

$$\Rightarrow \ln \left[\left(\frac{150}{\left(\frac{75}{(0.9 - x_w)} \right)} \right) * \left(\frac{0.45}{x_w} \right) \right] = 2.36 \ln \left[\left(\frac{150}{\left(\frac{75}{(0.9 - x_w)} \right)} \right) * \left(\frac{1 - 0.4}{1 - x_w} \right) \right]$$

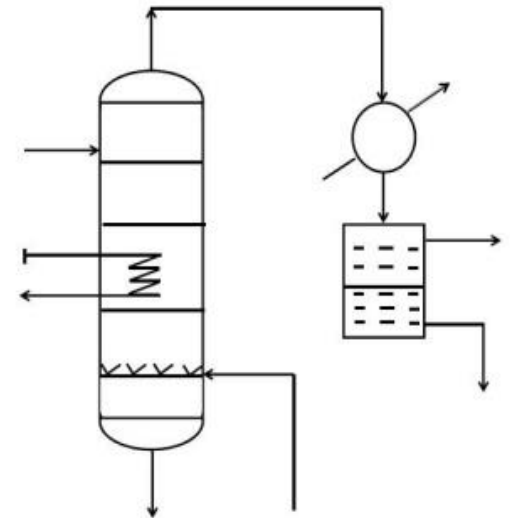
\Rightarrow

Solving,

\therefore Amount of Distillate,

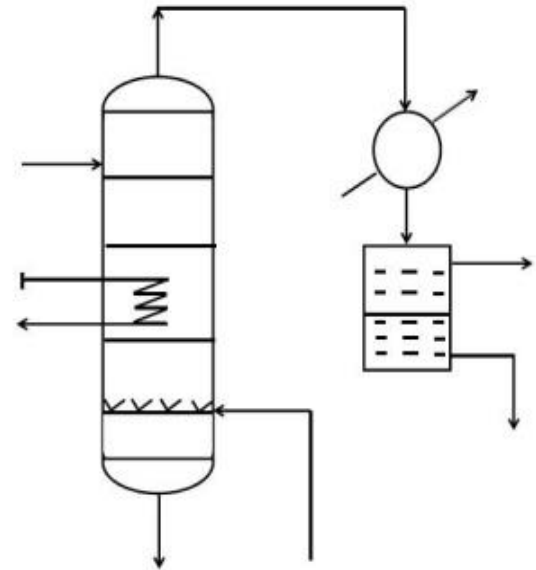
Lecture 4: Steam Distillation

- High boiling liquid cannot be purified by distillation at atmospheric pressure, since the materials may decompose at high temperature.
- Often the high boiling substances are essentially insoluble in water. Hence, a separation at lower temperature can be obtained by steam distillation.
- Steam distillation is a separation process in which live steam is blown through a component 'A' (having high boiling substance and low solubility in water). 'A' vaporizes slowly and leaves with the steam.
- Component 'A' is the target component which we want to recover at a relatively pure state.
- The mixture to be separated may contain traces of non-volatile impurity i.e. a mixture of A and another compound C (non-volatile).



Lecture 4: Steam Distillation

- 1) Feed is taken in a vessel or still.
- 2) Live steam is sparged at the bottom of the still.
- 3) Vapor of A along with steam is condensed in the condenser at the top.
- 4) Since A has less miscibility, two layers forms (a layer of target material and water layer) and can be easily separated.
- 5) Energy is required to heating the feed to its bubble point and for vaporization of A.
- 6) A still coil may be provided to make up such energy requirement and to avoid condensation of live steam.



Use of Steam Distillation:

- Food industries: Removal of volatile taints and flavors from edible fats & oils.
- Separation of thermally unstable substance from a mixture.
- Separation of NH_3 and VOCS from waste water.

Lecture 4: Steam Distillation

Now: If a layer of immiscible high boiling liquid (A) and water (B) are boiled, then the phase rule for three phases and two components is

$$F = C - P + 2 \quad \Rightarrow F = 2 - 3 + 2 = 1 \quad \text{Degree of freedom}$$

Hence, if the total pressure is fixed then the system is fixed.

- The two liquid phases each will exert its own vapor pressure at the prevailing temperature and cannot be influenced by the presence of others.
- When sum of the separate vapor pressures equals to the total pressure, the mixture boils:

$$P_A^v + P_B^v = P_t \quad \therefore y_A = \frac{P_A^v}{P_t} \quad y_B = \frac{P_B^v}{P_t} \quad \rightarrow \text{Vapor composition}$$

- Note that by steam distillation as long as liquid water is present, the high boiling component (A) vaporizes at a temperature well below its normal boiling point without using a vapor pressure.
- Disadvantage of this method is that large amounts of heat must be used to evaporate the water simultaneously with the high boiling mixture.

Lecture 4: Steam Distillation

If m_A moles of substance are to be volatilized by putting

m_B moles of steam and if the system operates at equilibrium, we may write:

$$\frac{m_A}{m_B} = \frac{P_A^v}{P_B^v} = \frac{P_A^v}{P_t - P_A^v} \qquad m_A = m_B \frac{P_A^v}{P_t - P_A^v}$$

- However, if the system does not operate at equilibrium, the partial pressure of A will be less than its vapor pressure.
- To take into account such as deviation, a factor E, vaporizing efficiency is used which

$$p_A = EP_A^v \qquad \therefore m_A = m_B \frac{EP_A^v}{P_t - EP_A^v}$$

Example: A 2 kg mixture of A and C (non-volatile) is to be steam distilled. Live steam at 110°C is used at a flow rate of 10 kg/h. Assume A is immiscible with water. The vaporization efficiency is 0.9. Vapor pressure of water at 110°C is 1.4 bar and for component A is 0.1 bar. Molecular weight of A is 200. Calculate the distillation time required.

Fractional Distillation

- *Fractional distillation* normally used to separate liquid mixtures into two or more vapor or liquid products with different compositions.
- *Equilibrium stage operation---* in each stage vapor phase is contacted with a liquid phase.
- Less volatile components --- concentrate in the liquid phase
- More volatile components---concentrate in the vapor.
- Using multiple stages in series with recycle, separation can be accomplished.

Fractional Distillation

- **Feed Condition:** may be liquid, vapor, or a liquid-vapor mixture.
- **Feed Entry:** may enter at any point in the column
- **Multiple Feed and Product cuts:** more than one stream may be fed to the system, and more than one product may be drawn.
- **Trays:** columns built from a set of distinct "trays" or "plates".
- **Trays numbering:** may be numbered from top down or bottom up. Flows and compositions take the number of the tray they leave. The top tray of the column "Tray 1" and numbers downward - - this is the convention.

Operating Principle

Point 1. Liquid stream flows down from one tray to the next lower tray; vapor stream flows up bubbling through the liquid on the trays.

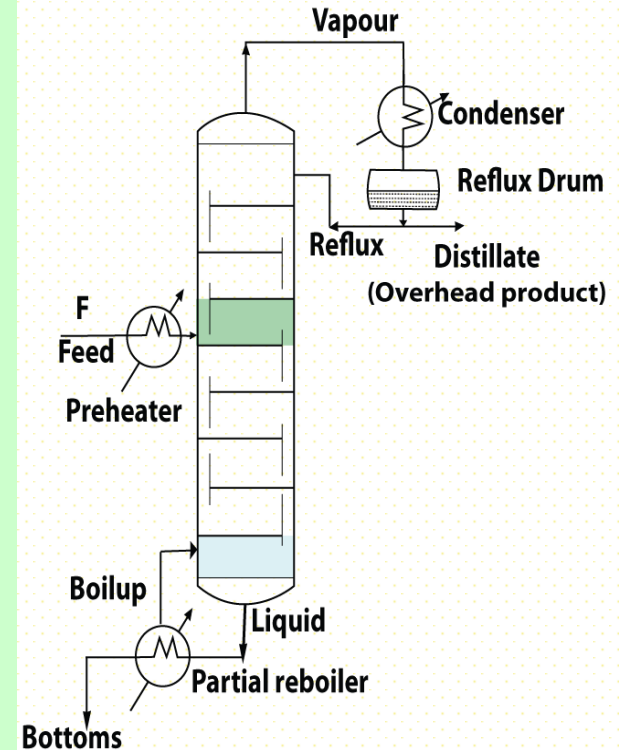
Point 2. Vapor from top tray is condensed and condensate is collected in a reflux drum.

Point 3. A part of condensate liquid is drawn as the top product and the other part is fed back to the top tray as reflux.

Point 4. Top product contains the more volatile component A and a little of the less volatile B.

Point 5. Liquid from bottom tray goes to a reboiler where it is partly vaporized, vapor is fed back to the tower and liquid part is continuously withdrawn as the bottom product.

Point 6. Bottom product is rich in less volatile component B and has only a small amount of A in it.



Operating Principle

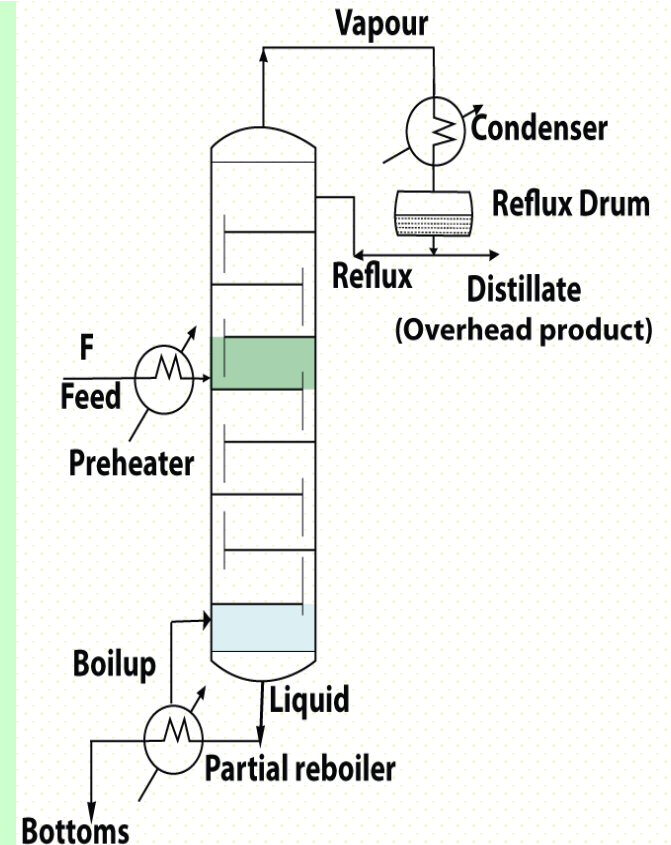
Point 7: Transport of more volatile component A occurs from liquid to vapor phase, while transport of less volatile B occurs from vapor to liquid phase.

Point 8: As the vapour flows up, it becomes progressively richer in A.

Point 9: Liquid becomes richer in less volatile B as it flows down the column.

Point 10: Product leaving the top of the column is called the distillate. Distillate product may be liquid or vapor (or occasionally both)

Point 11: Product leaving the bottom of the column is called the *bottom product* or "bottoms"



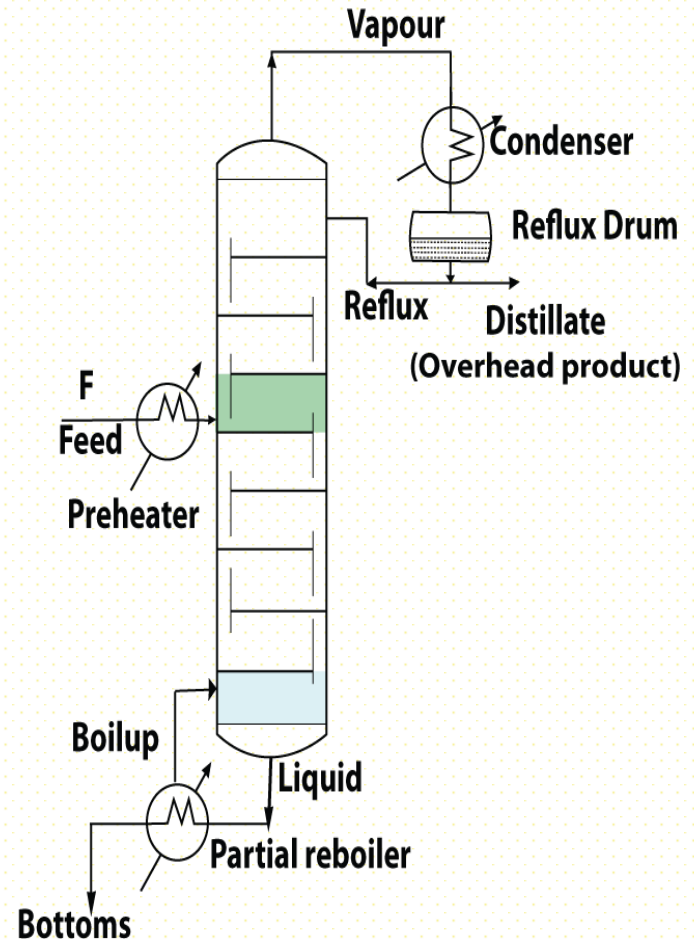
Operating Principle

Point 12: One or more intermediate or "sidedraw" products may be removed from the column.

Point 13: Portion of column above feed tray is called *rectification section*. In this section, vapor is enriched by contact with reflux.

Point 14: Portion of column below feed tray is called *stripping section*. Liquid portion of feed serves as reflux for this section.

Point 15: Operating pressure of column is typically controlled by adjusting heat removal in the condenser.



Operating Principle

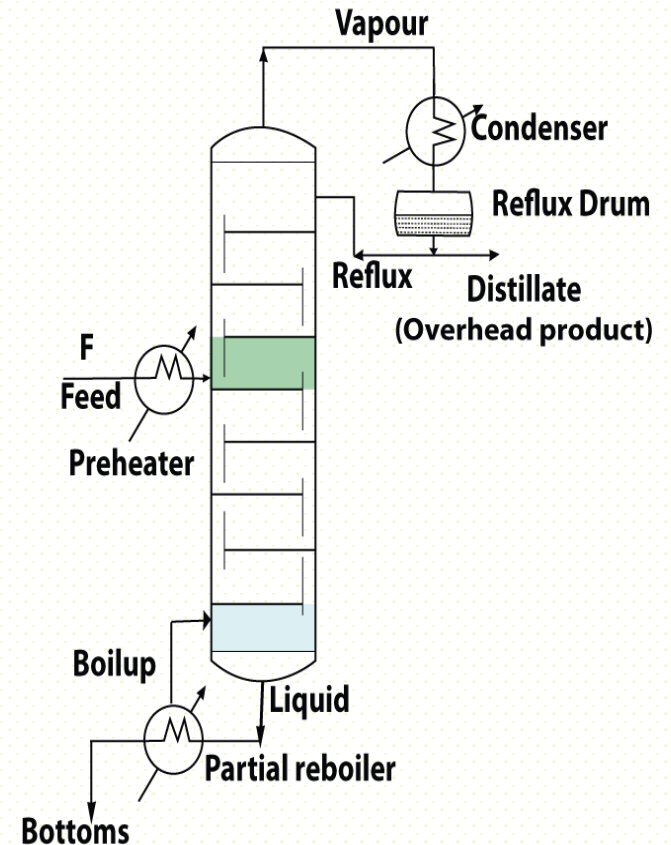
Point 16: A normal column has a temperature gradient and a pressure gradient from bottom to top.

Point 17: Inside the tower, liquids at bubble points and vapors at dew points, so that highest temperature is at bottom and lowest at top.

Point 18: Entire device is called fractionators.

Point 19: There are five "handles" that can be adjusted to manipulate the behavior of a distillation column ----

feed flow, two product flows, reflux flow, and boilup flow/or reboiler heat input



Ideal Stage

- **Ideal stage:** Where vapor and liquid leave the stage in equilibrium.
- **A key result of ideal stage assumption** is that liquid streams leaving an ideal stage are assumed to be at their bubble point. Vapor streams leave at their dew point.
- **Top and bottom products may be obtained in any desired purity** if enough stages are provided and enough reflux is available.
- There are **limits to the number of stages and to the amount of reflux**. Theoretical limits on performance are imposed by total reflux and minimum reflux.

Condenser

- **Total condenser:** All of the vapor leaving the top of the column is condensed. Composition of the vapor leaving the top tray is the same as that of the liquid distillate product and reflux.
- **Partial condenser:** Vapor is only partially liquefied. Liquid produced is returned to the column as liquid, and a vapor product stream is removed. The compositions of these three streams (G_1 , D, and R) are different.
- A partial condenser functions as an equilibrium separation stage, so columns with a partial condenser effectively have an extra ideal stage.

Reboiler

- Mostly *partial reboilers* and only vaporize part of the liquid in the column base. Partial reboilers also provide an ideal separation stage.
- Reboilers:
 - Thermosiphon types--- rely on the thermal effects on density to draw liquid through the heat exchanger.
 - Forced circulation types-- use a pump to force liquid through.
- In large, complex columns, sidestream reboilers can be used. These draw liquid off a tray, heat it, and then return the vapor liquid mixture to the same or a similar trays.

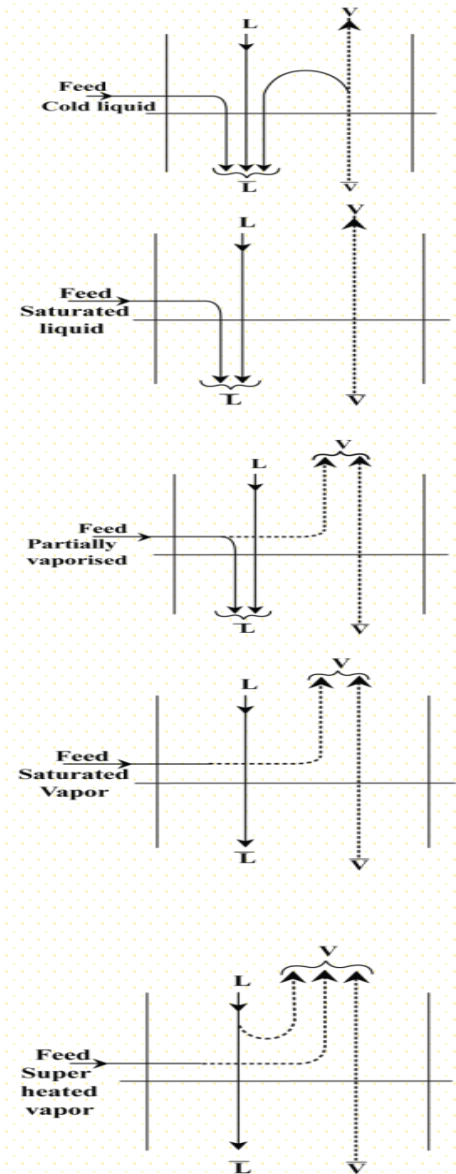
Feed Condition

Thermal condition of feed determines column internal flows.

Case 1: If the feed is below its bubble point, heat is needed to raise it to where it can be vaporized. This heat is obtained by condensing vapor rising through column. Hence, liquid flow moving down the column increases by entire amount of feed plus condensed material and vapor flow upward is decreased.

Case 2: If the feed enters as superheated vapor, it will vaporize some of the liquid to equalize the enthalpy. Liquid flow down the column drops and vapor flow up is increased by the entire amount of feed plus the vaporized material.

Case 3: If the feed is saturated (liquid or vapor), no additional heat must be added or subtracted, and feed adds directly to liquid or vapor flow.



Slope and Position of Feed Line

Case 1: Cold Feed--Positive slope and lie to the right of the vertical

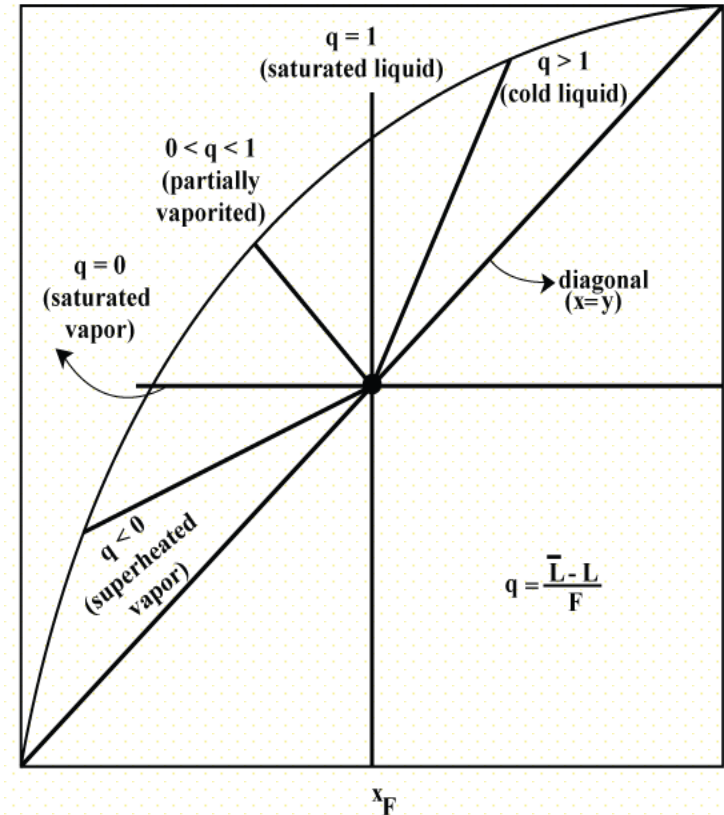
Case 2: Saturated liquid-- Vertical

Case 3: Saturated vapor--Horizontal

Case 4: For a mixed vapor-liquid feed-- it will lie between the horizontal and vertical (negative slope).

Case 5: Superheated feed-- will produce a line below the horizontal.

- Rectifying and stripping lines intersect on the feed line.
- If the column has an intermediate feed or product, the same rules apply.



Number of Trays by McCabe Thiele Method

Assumption 1:

- Molar flow rate of liquid from one tray to another is constant over any section of the column.
- Molar flow rate of vapor in respective sections remain constant.
- Assumption is true when molar heat of vaporization of mixture does not depend upon composition or temperature in the column.
- If constant molar overflow occurs, mass exchange between phases occurs in equimolar counter-diffusion mode.

Assumption 2:

- Heat loss from the column is negligible

Major steps of the graphical construction in the McCabe-Thiele method:

- Draw equilibrium curve using the available data,
- Draw operating lines for the rectifying and the stripping sections
- Draw steps between the equilibrium and operating lines to find out number of ideal trays.

Operating Line Equations

Rectifying Section (Total Condenser – reflux at the bubble points)

A total materials balance over the envelope: $G = L + D$

Let us define reflux ratio: $R = L/D$

$$\therefore G = RD + D = D(R + 1)$$

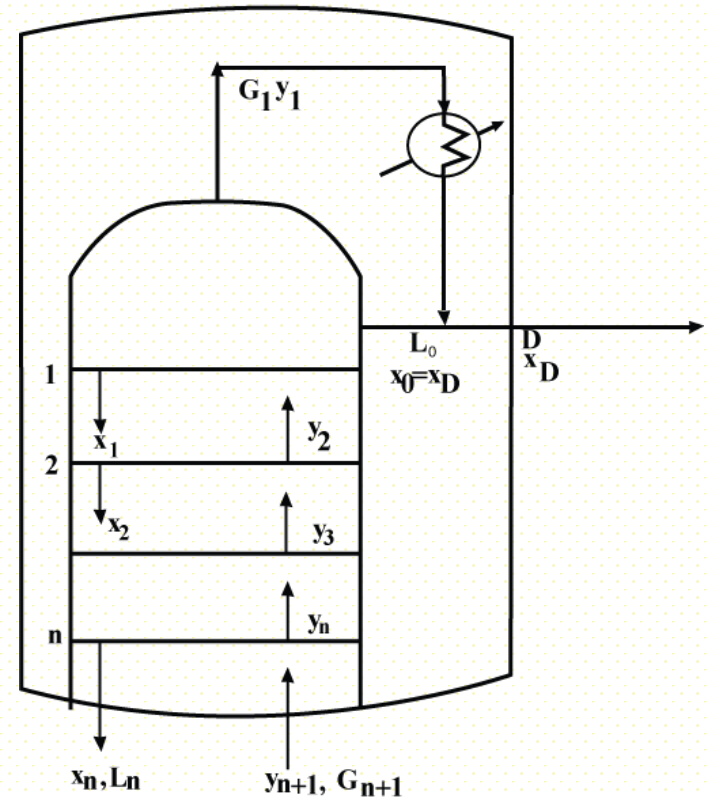
$$\Rightarrow \frac{G}{D} = R + 1$$

Equation represents material balance of component A over an envelope shown:

$$Gy_{n+1} = Lx_n + Dx_D$$

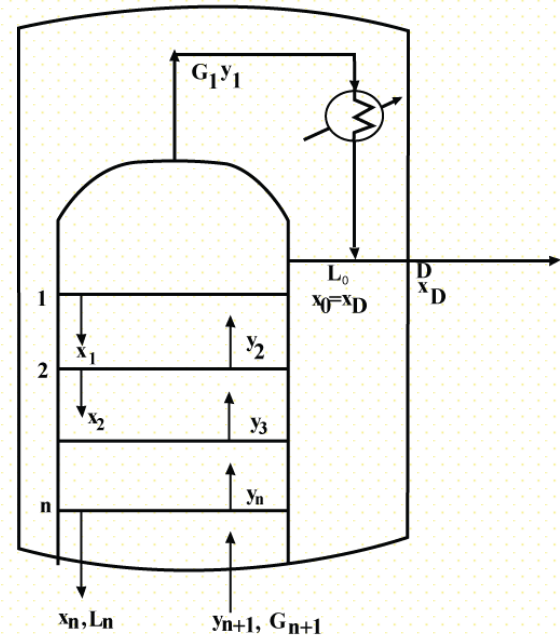
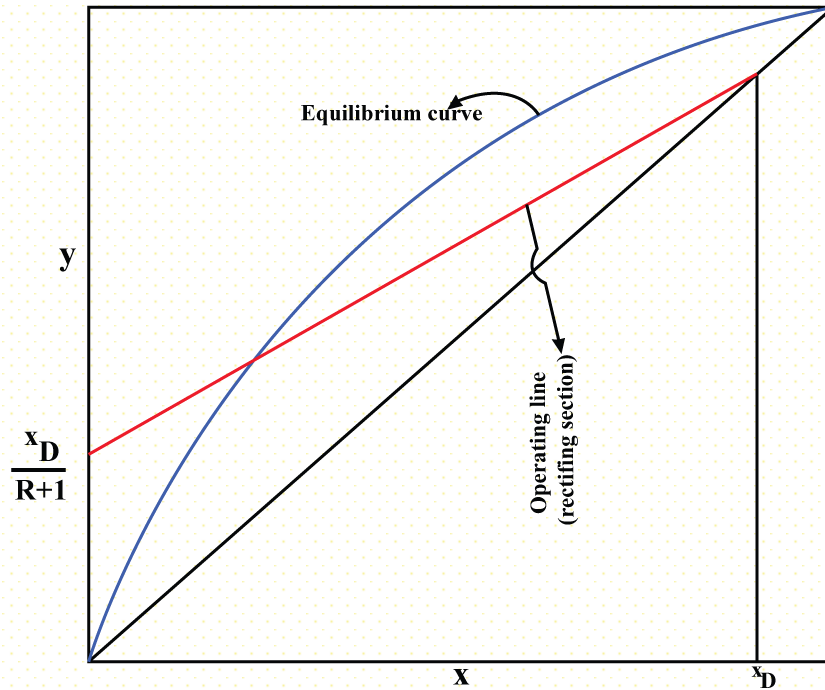
$$\Rightarrow y_{n+1} = \frac{L}{G}x_n + \frac{D}{G}x_D = \frac{L/D}{G/D}x_n + \frac{x_D}{G/D}$$

$$\Rightarrow y_{n+1} = \frac{R}{R+1}x_n + \frac{x_D}{R+1}$$



Operating Line Equations

Rectifying Section (Total Condenser – **reflux at the bubble points**)



Operating Line Equations

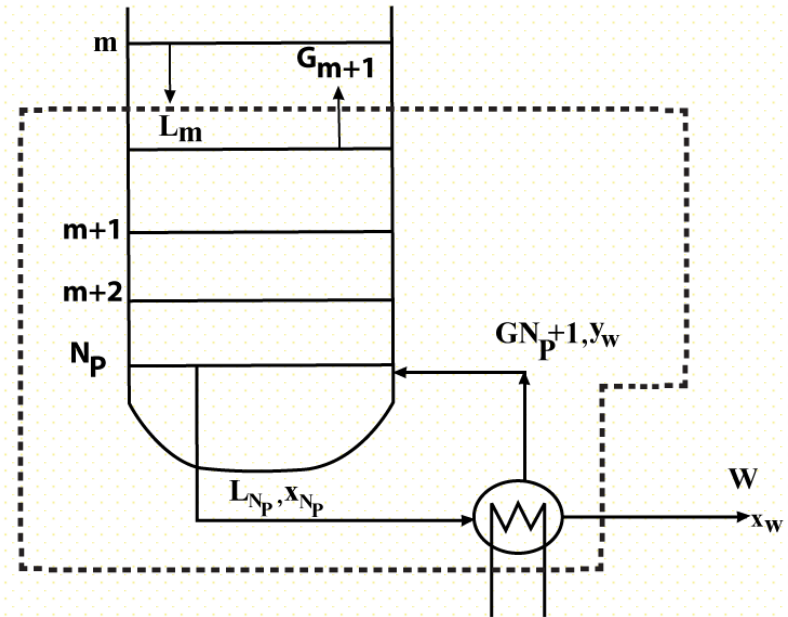
Stripping Section

With the constant molar overflow assumption, the material balance for component A over an envelope shown:

$$\bar{L}x_m = \bar{G}y_{m+1} + Wx_w$$

Putting $\bar{G} = \bar{L} - W$

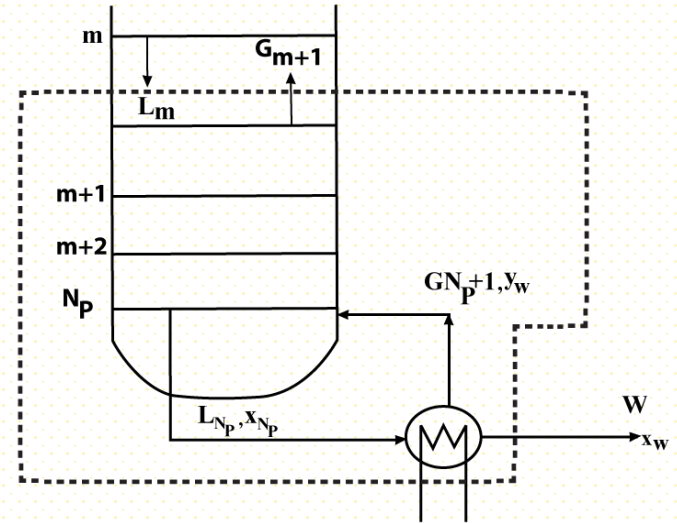
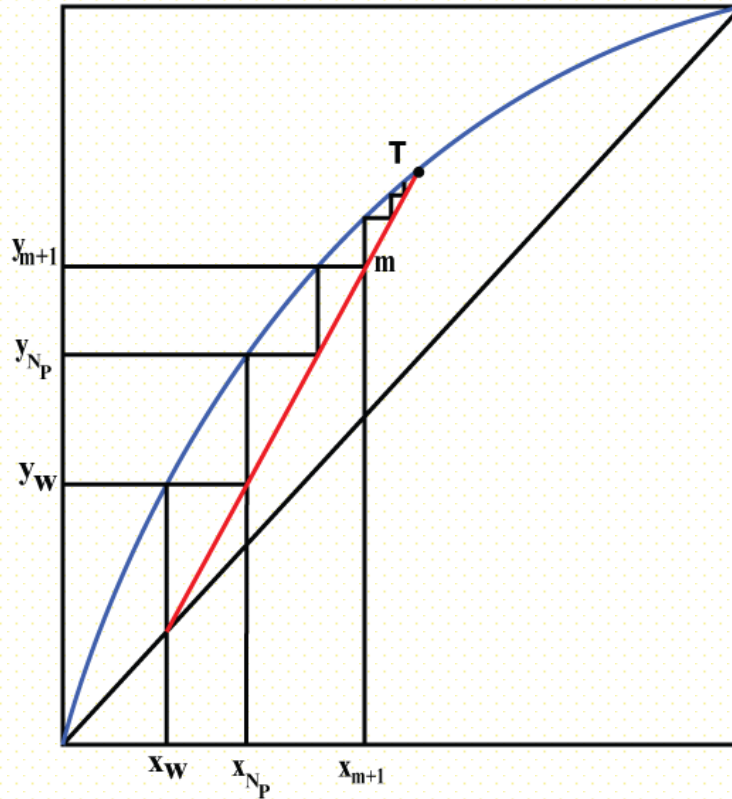
$$y_{m+1} = \frac{\bar{L}}{\bar{G}} x_m - \frac{W}{\bar{G}} x_w = \frac{\bar{L}}{\bar{L} - W} x_m - \frac{W}{\bar{L} - W} x_w$$



Similar to the reflux ratio defined for the rectified section, we may define a quantity called the boil-up ratio, R_G , for the stripping section

$$R_G = \frac{\bar{G}}{W} = \frac{\text{moles of vapour leaving the reboiler per hour}}{\text{moles of liquid drawn as the bottom product per hour}}$$

Stripping Section



Example

A mixture of 45 mol% n-hexane and 55 mol% n-heptane is subjected to continuous fractionation in a tray column. The feed rate is 100 kmol/hr. The distillate contains 90% n-hexane and the residue contains 5% n-hexane. The reflux is saturated liquid and the reflux ratio is 2.5. The feed is saturated liquid. The relative volatility of n-hexane in the mixture is 2.36. Plot the operating line for rectifying and stripping section.

Solution

$$F = 100 \frac{\text{kmol}}{\text{hr}}, \quad Z_F = 0.45$$

$$x_D = 0.9, x_W = 0.05, R = 2.5$$

$$F = D + W \quad \Rightarrow \quad 100 = D + W$$

$$F z_F = D x_D + W x_W$$

$$\Rightarrow 100 \times 0.45 = D \times 0.9 + W \times 0.05$$

$$\Rightarrow 45 = 0.9D + 0.05W$$

$$= 0.9(100 - W) + 0.05W$$

$$= 90 - 0.9W + 0.05W$$

$$= 0.85W$$

$$\Rightarrow W = \frac{45}{0.85} \Rightarrow W = 53 \frac{\text{kmol}}{\text{hr}}, \quad \text{solving } D = 47 \frac{\text{kmol}}{\text{hr}}$$

$$R = \frac{L_O}{D} \Rightarrow 2.5 = \frac{L_O}{47} \Rightarrow L_O = 117.5$$

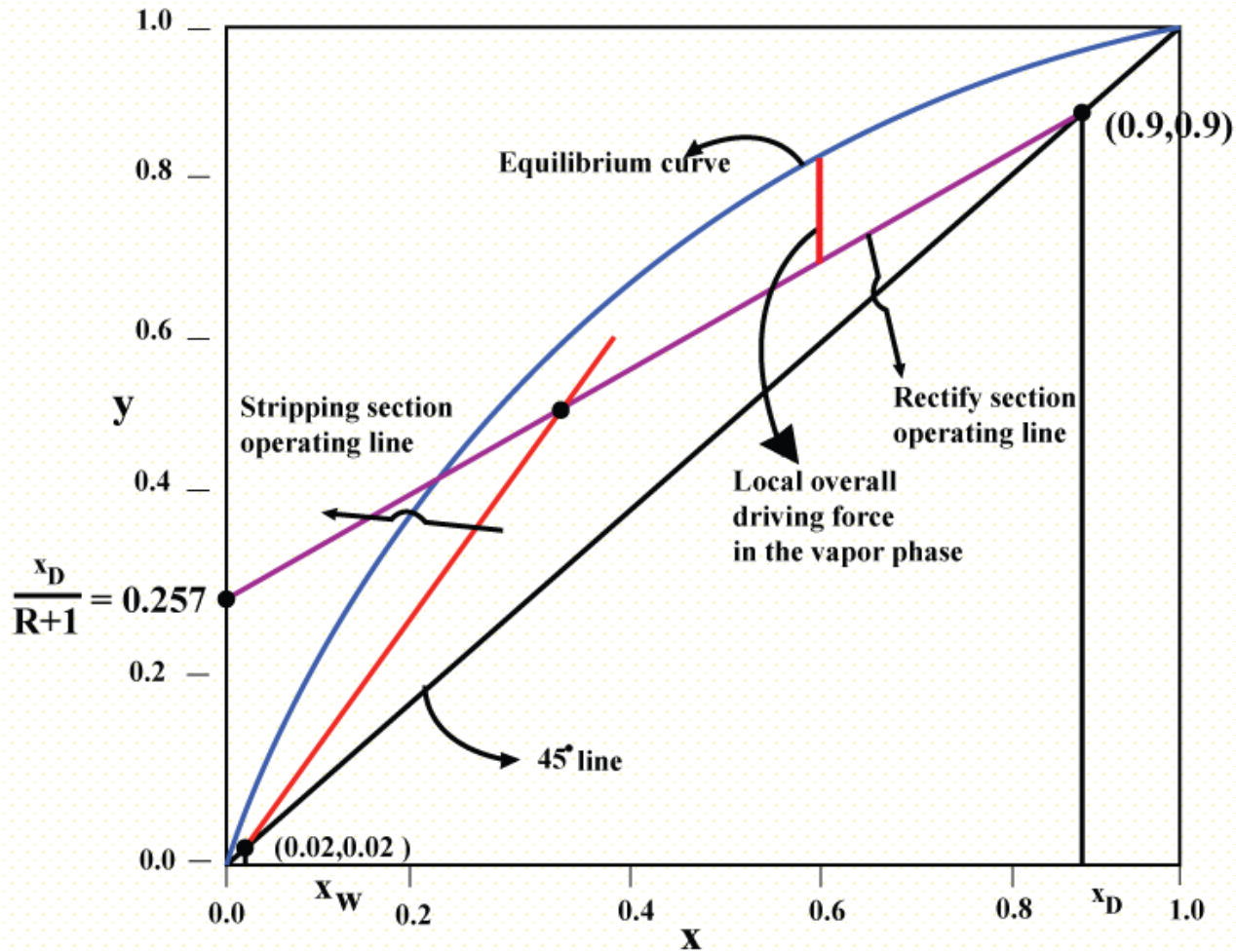
$$G_1 = D(R + 1) = 47 \times (2.5 + 1) = 47 \times 3.5 = 164.5$$

$$G = G_1 = 164.5$$

$$\bar{L} = L + 100 = 117.5 + 100 = 217.5$$

$$\bar{G} = G = 164.5$$

Solution



Equimolal Overflow

Calculations using OLs are much more convenient if they are straight lines. This is true only if the liquid and vapor flows do not change in a given section of the column.

What is required for them to be constant?

- **Equimolal overflow or *Constant Molal Overflow* is required.**
- This occurs when the molar heat of vaporization of the liquid phase is essentially equal to that of the vapor phase.
- Quickest way to check the validity of this assumption is to compare the heats of vaporization of the components. If their ratio is roughly 1:1, the assumption is probably acceptable.
- When $x=x_D$ that $y=x_D$ as well . This means that the point (x_D, x_D) lies on the rectifying line.
- If we assume this rectifying OL can be drawn using only this point and the slope.
- Stripping OL line can be drawn with point (x_W, x_W) and the slope.

Feed Tray

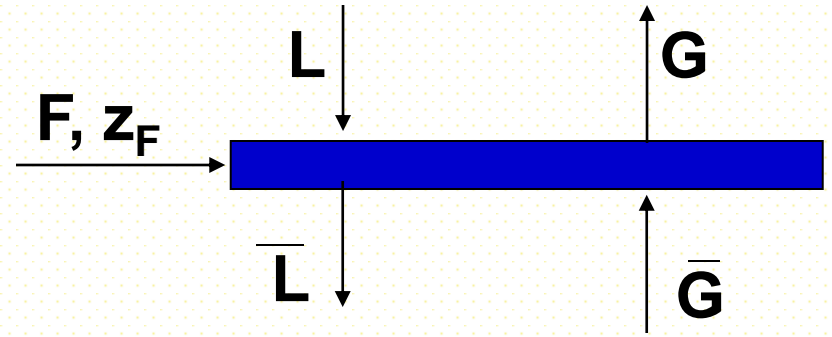
Total material balance on feed tray

$$F + L + \bar{G} = \bar{L} + G$$

Energy Balance:

Assume the change in enthalpy of a phase as it passes through the feed plate is small

$$FH_F + LH_L + \bar{G}H_G = \bar{L}H_L + GH_G \Rightarrow (\bar{L} - L)H_L = (\bar{G} - G)H_G + FH_F$$



Now using the total material balance equation

$$\bar{G} - G = \bar{L} - L - F$$

$$(\bar{L} - L)H_L = (\bar{L} - L - F)H_G + FH_F = (\bar{L} - L)H_G + F(H_F - H_G)$$

$$\Rightarrow (\bar{L} - L)(H_L - H_G) = F(H_F - H_G) \Rightarrow \frac{(\bar{L} - L)}{F} = \frac{H_F - H_G}{H_L - H_G} = \frac{H_G - H_F}{H_G - H_L} = q$$

But $\bar{L} - L$ = increase in the liquid flow rate across the feed tray as a result of introduction of feed
= rate of input of liquid with the feed

So, q is the fraction of liquid in the feed.

Another significance: $q = \frac{\text{heat required to convert 1 mol feed to saturated vapor}}{\text{molar heat of vaporization of the saturated liquid}}$

Feed Line

Where the rectifying line and the stripping line intersect?

- Point of intersection (x, y) must be satisfied by the material balance equations of both rectifying and stripping section.

Rectifying balance: $G y = L x + D x_D$

Stripping balance: $\bar{G} y = \bar{L} x - W x_W$

Subtracting the stripping balance from rectifying balance yields

$$(G - \bar{G}) y = (L - \bar{L}) x + (D x_D + W x_W)$$

Now using overall material balance equation, the above equation reduces to:

$$(G - \bar{G}) y = (L - \bar{L}) x + F z_F$$

Now if we divide by F to the total material balance equation and use the definition of q then:

$$\frac{\bar{G} - G}{F} + 1 = \frac{\bar{L} - L}{F} = q$$

$$\frac{-(\bar{G} - G)}{F} y = \frac{-(\bar{L} - L)}{F} x + z_F \Rightarrow -(q - 1) y = -q x + z_F \Rightarrow y = \frac{q}{q - 1} x - \frac{z_F}{q - 1}$$

Slope and Position of Feed Line

Case 1: Cold Feed--Positive slope and lie to the right of the vertical

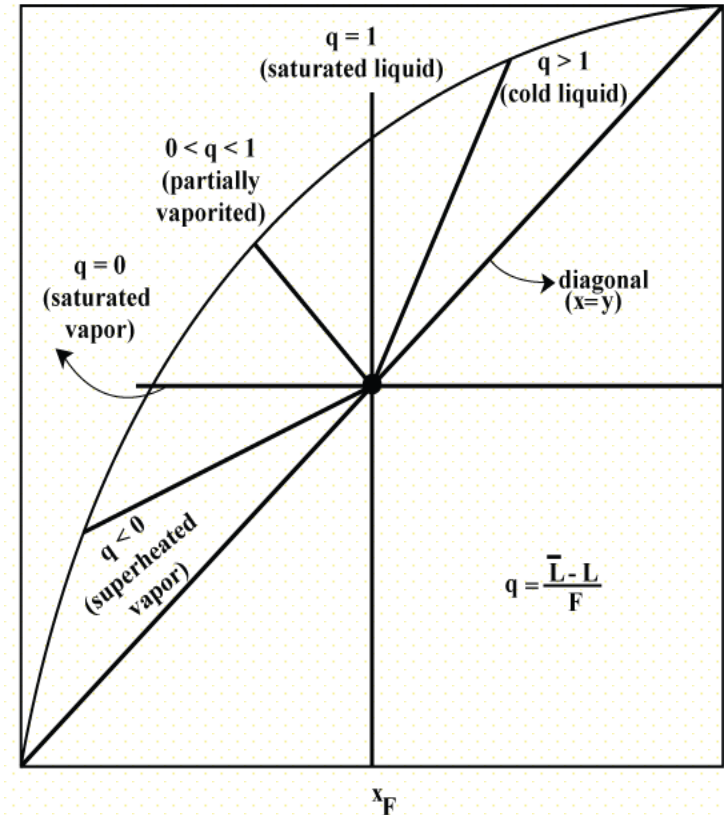
Case 2: Saturated liquid-- Vertical

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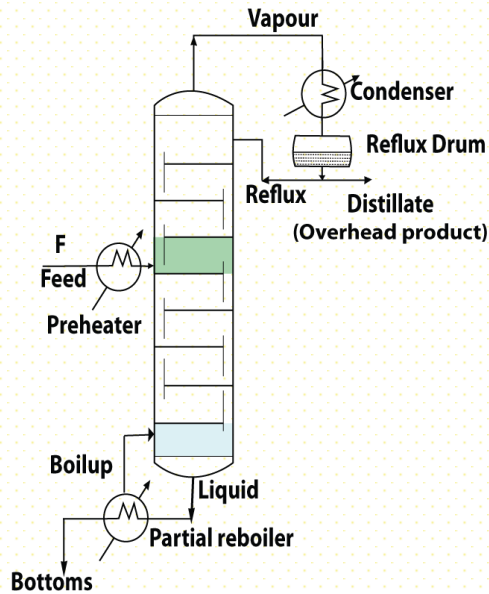


McCabe Thiele Method

Topic of discussion

- McCabe-Thiele graphical construction
- Determination of N and X_B
- Total reflux
- Example

McCabe Thiele Method

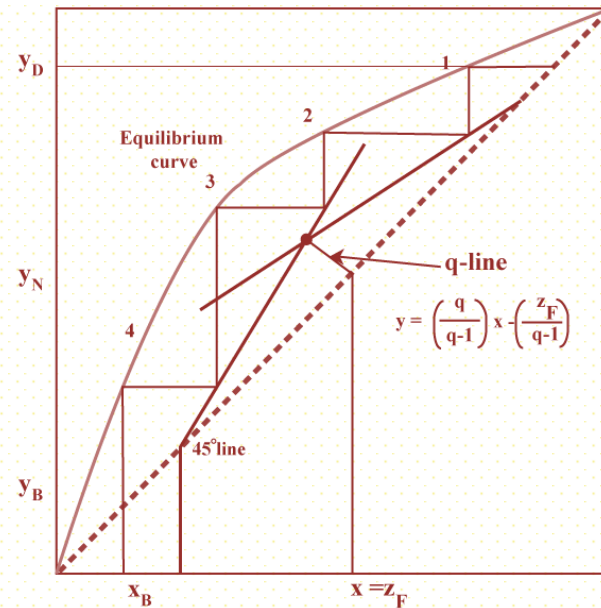
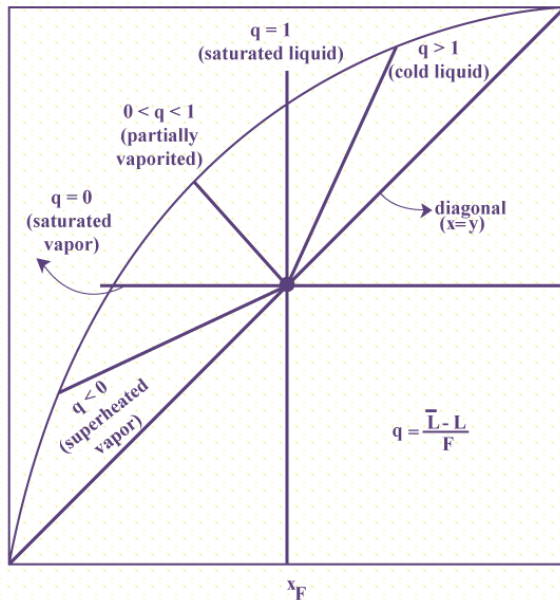


Rectifying Section:
Operating line
 Slope = $L/G = R/(R+1) < 1$

$$y_{n+1} = \frac{L}{G} x_n + \frac{D}{G} x_D$$

Stripping Section:
Operating line
 Slope = $\underline{L}/\underline{V} = (V_B + 1)/V_B$

$$y_{m+1} = \frac{\bar{L}}{G} x_m - \frac{W}{G} x_W$$



McCabe Thiele Method

Step 1: Plot **equilibrium curve** and 45° line.

Step 2: Locate and plot given compositions (x_F , x_W , and x_D)

Step 3: Calculate **slope** $[q/(q-1)]$ of q-line and draw **q-line** using $x=x_F$ and the slope

Step 4: Calculate y-intercept $[x_D/(R+1)]$ of the rectifying line and draw operating line (OL) for **Rectifying section**

Step 5: Draw OL for **Stripping section** using point (x_W, x_W) and intersection between rectifying OL and q-line

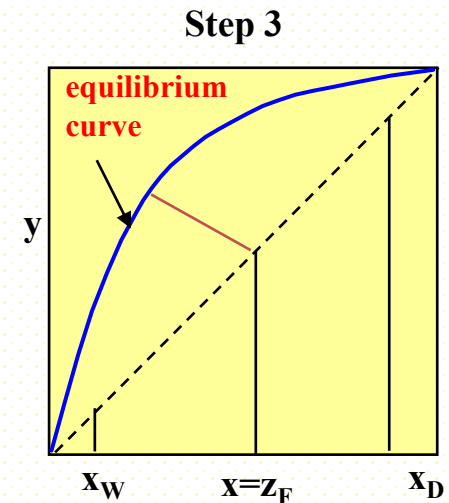
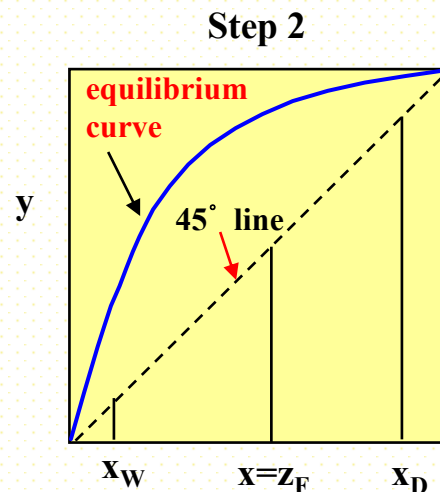
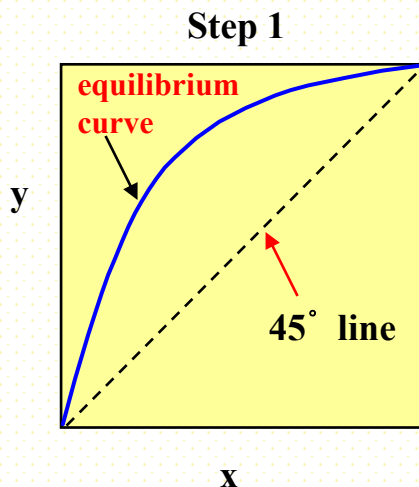
Step 6: From x_D locate x_1 and y_1 drawing a **horizontal line** to the **equilibrium line** for stage 1.

Step 7: Find y_2 drawing a **vertical line** to the **rectifying OL** locate the mass balance condition between x_1 and y_2 .

Step 8: From y_2 draw a **horizontal line** to the **equilibrium line** for stage 2 to locate x_2 .

Step 9: Return to step 7 and cycle through steps 7 and 8 until $x_i < x_F$. Draw subsequent **vertical lines** to the **stripping OL**.

Step 10: End after predetermined number of stages, or when x_i is less than x_W .



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Step 1: Plot **equilibrium curve** and 45° line.

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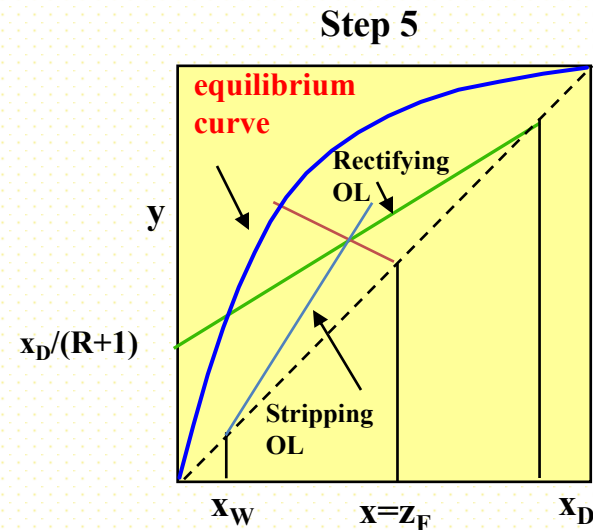
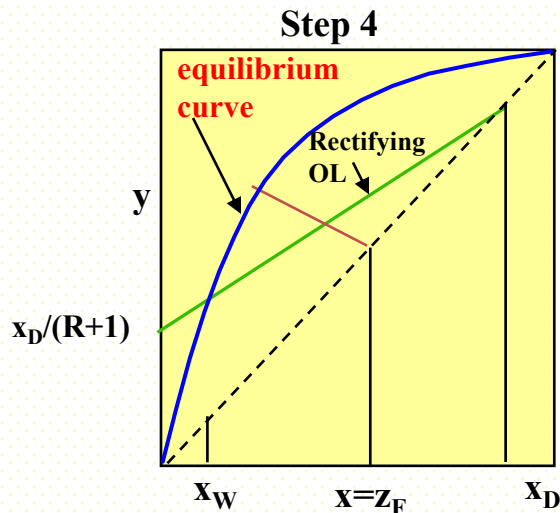
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Step 9: Return to step 7 and cycle through steps 7 and 8 until $x_i < z_F$. Draw subsequent **vertical lines** to the **stripping OL**.

Step 10: End after predetermined number of stages, or when x_i is less than x_W .



McCabe Thiele Method

Step 1: Plot **equilibrium curve** and 45° line.

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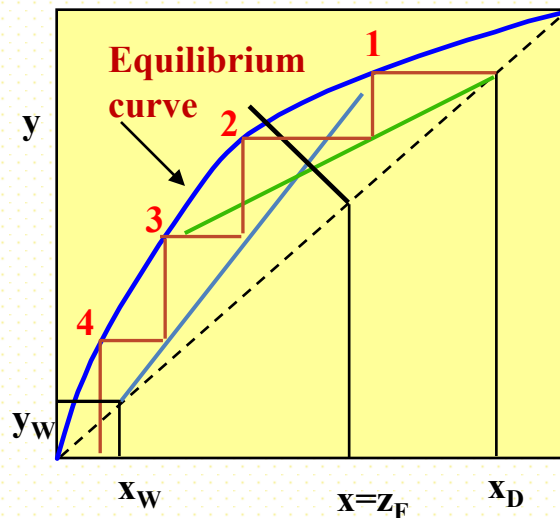
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Step 9: Return to step 7 and cycle through steps 7 and 8 until $x_i < z_F$. Draw subsequent **vertical lines** to the **stripping OL**.

Step 10: End after predetermined number of stages, or when x_i is less than x_W .



Example

A mixture of 45 mole % n-hexane and 55 mole % n-heptane is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% n-hexane and the residue contains 5% n-hexane. The feed is saturated liquid. A reflux ratio is 2.5 is used. The relative volatility of n-hexane in mixture is 2.36. Determine the number of ideal trays required.

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Solution

Distillate contains 95% n-hexane

Distillate (x_D , x_D) is (0.95, 0.95)

Feed mixture contains 45 mole % n- hexane, Feed (x_F , x_F) is (0.45, 0.45)

Residue contains 5% mole of n- hexane (x_W , x_W) is (0.05, 0.05)

Relative volatility of n- hexane is 2.36.

Reflux ratio is 1.5

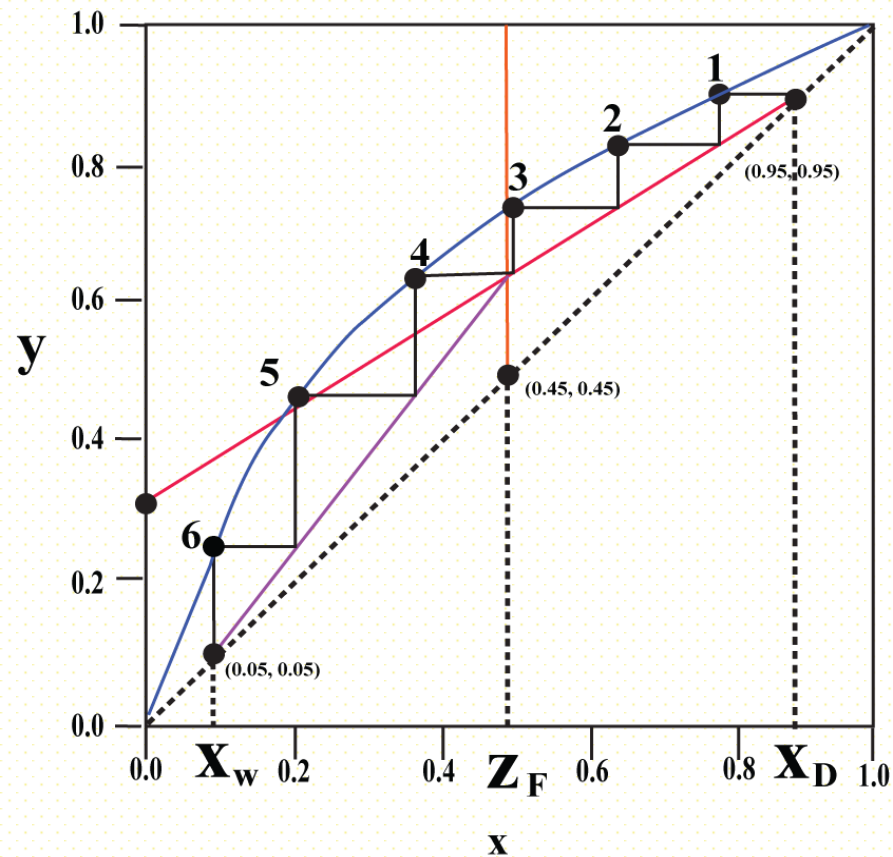
Intersect $\frac{x_D}{R+1} = \frac{0.95}{1.5+1} = 0.38$ $q = 1, \therefore slope = \frac{q}{q-1} = \frac{1}{1-1} = \infty$

Solution

$$y = \frac{\alpha x}{1 + (\alpha - 1)x}$$

Given $\alpha = 2.36$

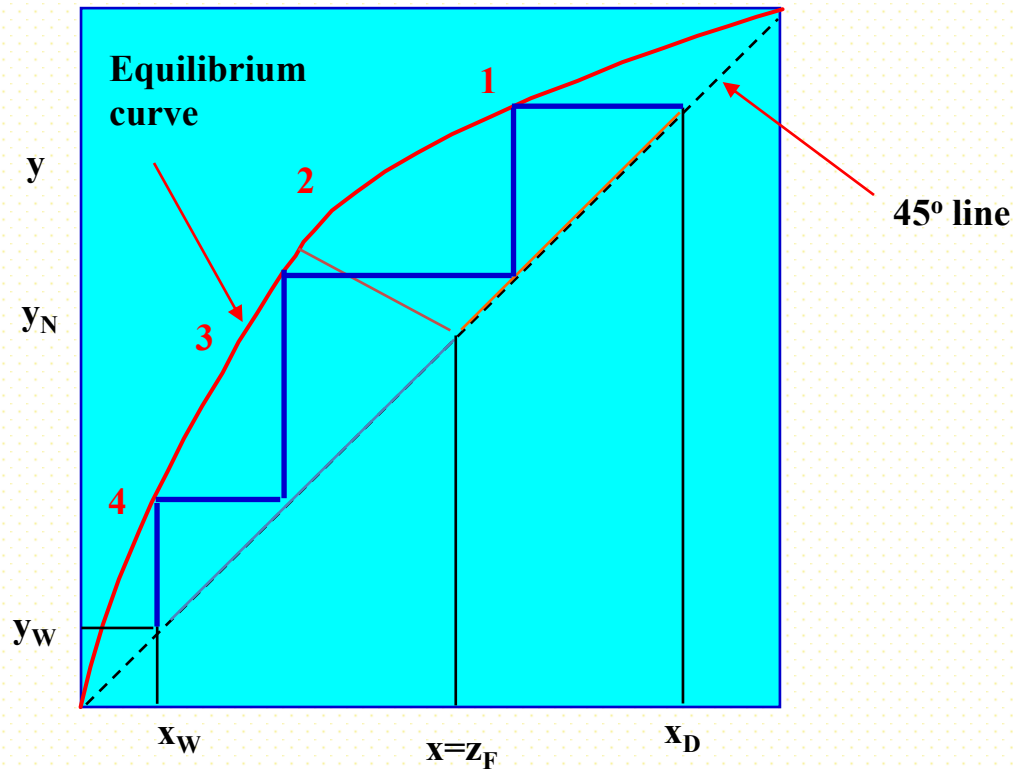
x	0	0.076	0.199	0.341	0.505	0.705	1
y	0	0.163	0.37	0.55	0.71	0.85	1



Total Reflux: Minimum number of Stages

- At total reflux, no distillate is removed from the reflux drum ($D=0$).
- Reflux ratio: $R=L_o/D = L_o/0 = \text{infinity}$.
- No product is drawn from the reboiler either. All the liquid flowing to the reboiler is vaporized and fed back to the column.
- Column operating at total reflux under steady state conditions, there should not be any flow of feed into it.
- At total reflux: slope of rectifying OL [$R/(R+1)$] is unity and it passes through the point (x_D, x_D) on the diagonal. The OL coincides with the diagonal.
- So does the stripping section operating line.
- Gives *theoretical minimum number* of stages to achieve a given separation.
- Total reflux is very during startup of column till steady state condition is reached and product is not withdrawn until a separation close to that desired is achieved. After this, continuous feed flow and product withdrawal are started.

Total Reflux: Minimum number of Stages



Recycling all exiting vapor as reflux and all exiting liquid as boilup: **operating lines have slope of one.**

No product is produced: **feed must then go to zero**

Total Reflux: Fenske Equation

Fenske Equation is another method for determining the minimum number of trays required for a given separation

- ***This equation can be used to theoretically calculate minimum number of trays if the relative volatility remains reasonably constant.***

Let N_m be the minimum number of trays in the column. Besides, there is a ***total reboiler***.

If α_w is relative volatility of A at the reboiler temp. and pressure, and x_w and y_w are the equilibrium liquid and vapour concentration in the reboiler, then by definition:

$$\frac{y_w}{1 - y_w} = \alpha_w \frac{x_w}{1 - x_w}$$

Vapour leaving the reboiler and entering the lowest tray (tray number N_m in this case) has a mole fraction y_w of the component A.

Liquid leaving this tray has a composition x_{N_m}

Total Reflux: Fenske Equation

Vapour leaving the reboiler and entering the lowest tray (tray number N_m in this case) has a mole fraction y_w of the component A.

Liquid leaving this tray has a composition x_{N_m}

So the point (x_{N_m}, y_w) lies on the operating line. Because operating line coincides with diagonal at total reflux, $x_{N_m} = y_w$

Putting this result in the above equation:
$$\frac{x_{N_m}}{1 - x_{N_m}} = \alpha_w \frac{x_w}{1 - x_w}$$

Applying the same procedure to the case of tray number N_m

$$\frac{y_{N_m}}{1 - y_{N_m}} = \alpha_{N_m} \frac{x_{N_m}}{1 - x_{N_m}} = \alpha_{N_m} \cdot \alpha_w \cdot \frac{x_w}{1 - x_w}$$

Similarly for tray number $N_m - 1$

$$\frac{y_{(N_m-1)}}{1 - y_{(N_m-1)}} = \alpha_{(N_m-1)} \frac{x_{(N_m-1)}}{1 - x_{(N_m-1)}} = \alpha_{(N_m-1)} \cdot \alpha_{N_m} \cdot \alpha_w \cdot \frac{x_w}{1 - x_w}$$

Total Reflux: Fenske Equation

Similarly for tray number $N_m - 1$

$$\frac{y_{(N_m-1)}}{1 - y_{(N_m-1)}} = \alpha_{(N_m-1)} \frac{x_{(N_m-1)}}{1 - x_{(N_m-1)}} = \alpha_{(N_m-1)} \cdot \alpha_{N_m} \cdot \alpha_w \cdot \frac{x_w}{1 - x_w}$$

Note that the point (x_{N_m-1}, y_{N_m}) lies on the operating line which coincides with the diagonal; therefore, $x_{N_m-1} = y_{N_m}$

Continuing the procedure up to the top tray (where $y_1 = x_D$),

$$\frac{x_D}{1 - x_D} = \frac{y_1}{1 - y_1} = \alpha_1 \alpha_2 \dots \alpha_{N_m} \cdot \alpha_w \cdot \frac{x_w}{1 - x_w} \Rightarrow \frac{x_D}{1 - x_D} = \left(\alpha_{av}^{N_m+1} \right) \cdot \frac{x_w}{1 - x_w}$$

$$i.e. \quad N_m + 1 = \frac{\log \frac{x_D (1 - x_w)}{x_w (1 - x_D)}}{\log \alpha_{av}}$$

Here α_{av} is the average relative volatility of the more volatile component. The above equation is called *Fenske's equation*, which is useful for the calculation on the minimum number of trays.

Example

A mixture of 45 mole % n-hexane and 55 mole % n-heptane is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% n-hexane and the residue contains 5% n-hexane. The feed is saturated liquid. A reflux ratio of 2.5 is used. The average relative volatility is 2.36. Determine the number of ideal trays using Fenske equation.

Example

A mixture of 45 mole % n-hexane and 55 mole % n-heptane is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% n-hexane and the residue contains 5% n-hexane. The feed is saturated liquid. A reflux ratio of 2.5 is used. The average relative volatility is 2.36. Determine the number of ideal trays using Fenske equation.

Solution

Distillate contains 95% n-hexane

Distillate (x_D , x_D) is (0.95, 0.95)

Feed mixture contains 45 mole % n- hexane, Feed (x_F , x_F) is (0.45, 0.45)

Residue contains 5% mole of n- hexane (x_W , x_W) is (0.05, 0.05)

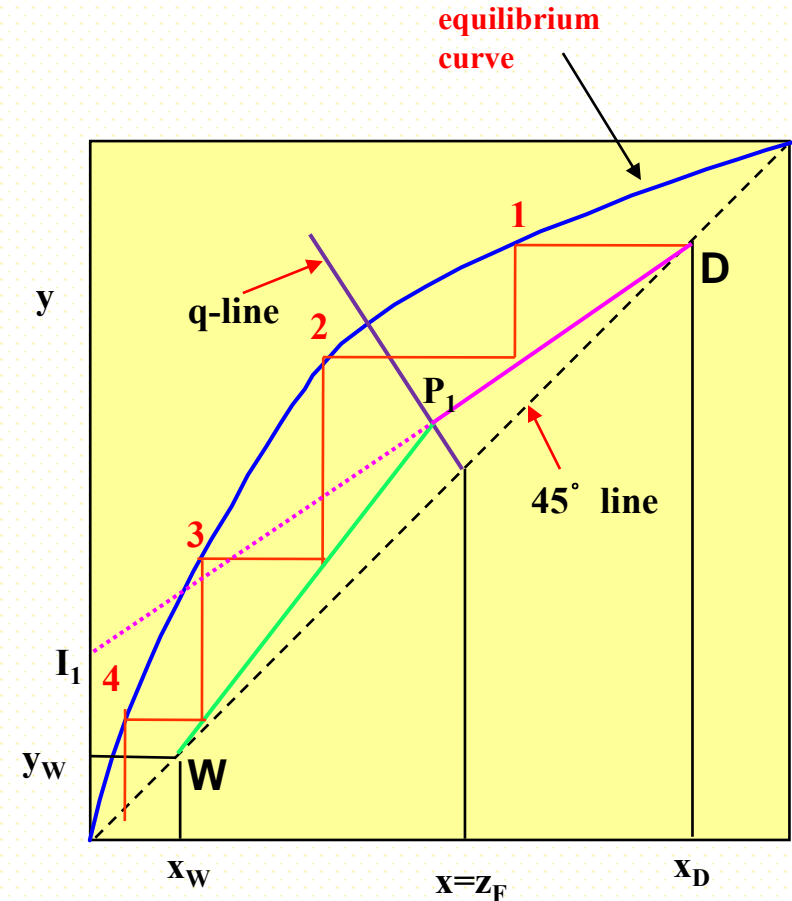
Relative volatility of n- hexane is 2.36.

Fenske equation

$$N_m + 1 = \frac{\log \frac{x_D (1 - x_W)}{x_W (1 - x_D)}}{\log \alpha_{av}} = \frac{\log \frac{0.95(1 - 0.05)}{0.05(1 - 0.95)}}{\log 2.36} = 6.8$$

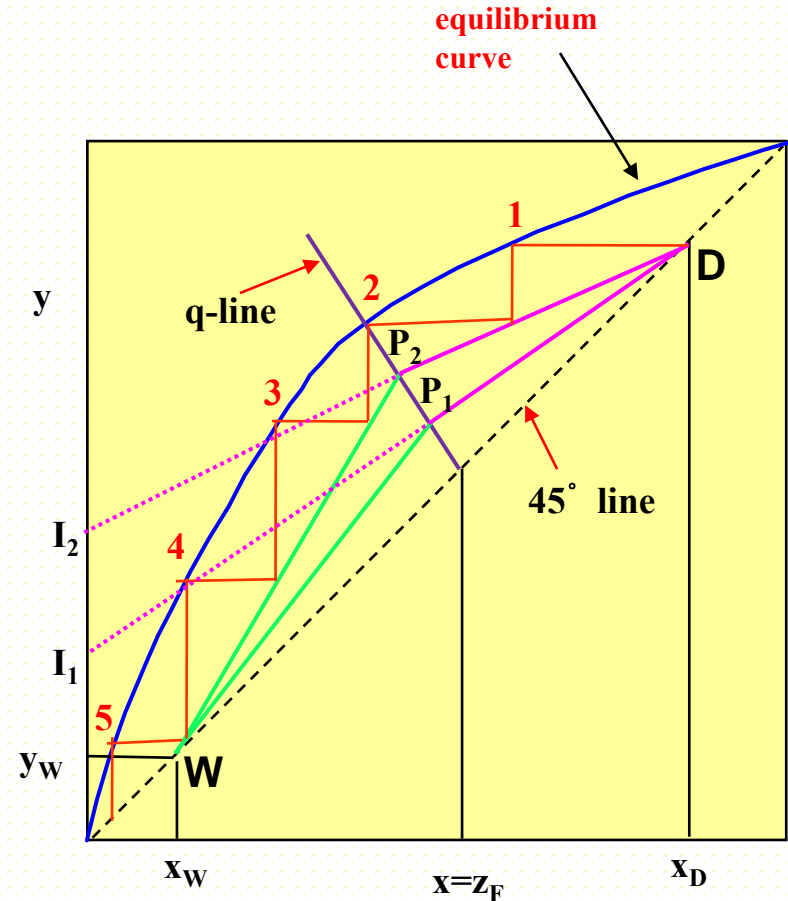
Minimum Reflux: Pinch Point

- For a particular reflux ratio (R_1 , say), DI_1 is the enriching section operating line having a slope $R_1/(R_1+1)$. It intersects the feed line point P_1
- WP_1 is stripping section operating line.
- The number of theoretical trays required is obviously finite.



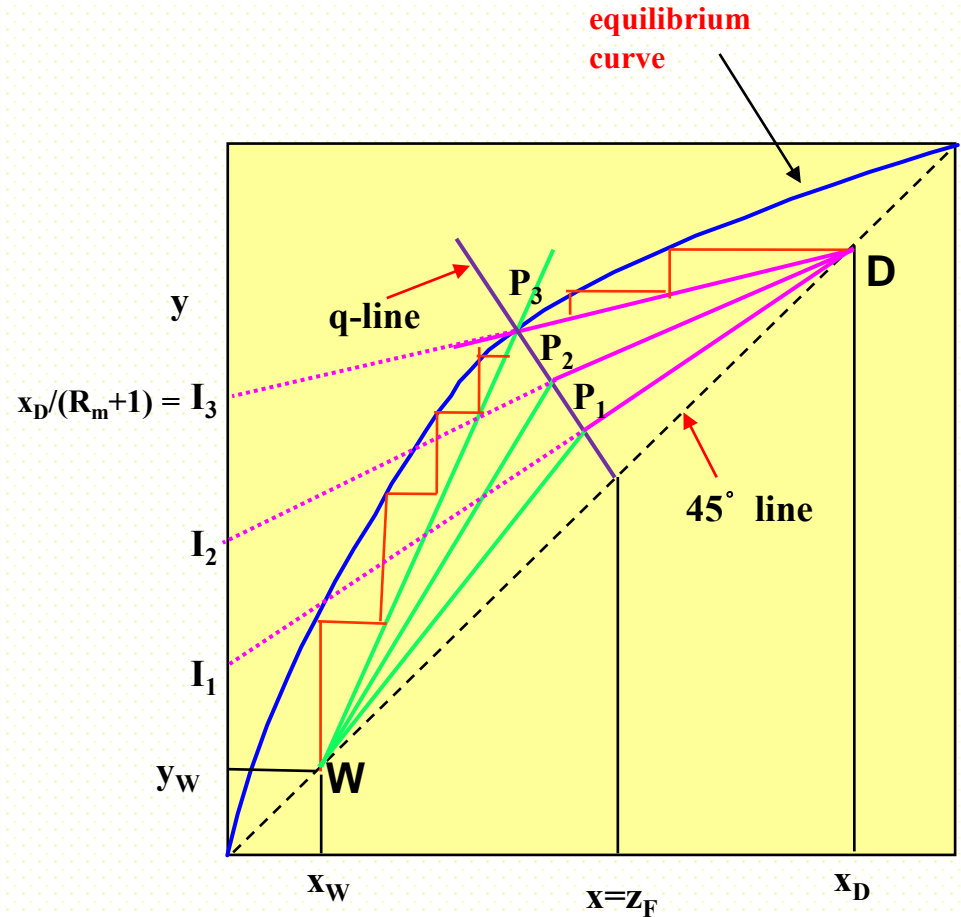
Minimum Reflux: Pinch Point

- As the reflux ratio R_1 decreases to R_2 , the slope of the upper operating line decreases, but the intercept increases, i.e. the point I_1 moves to I_2 (say). The upper operating line is DP_2
- The stripping section line is WP_2 ; and they intersect at P_2 on the feed line.
- Now the driving force is less at all the points and the number of theoretical trays will be more.



Minimum Reflux: Pinch Point

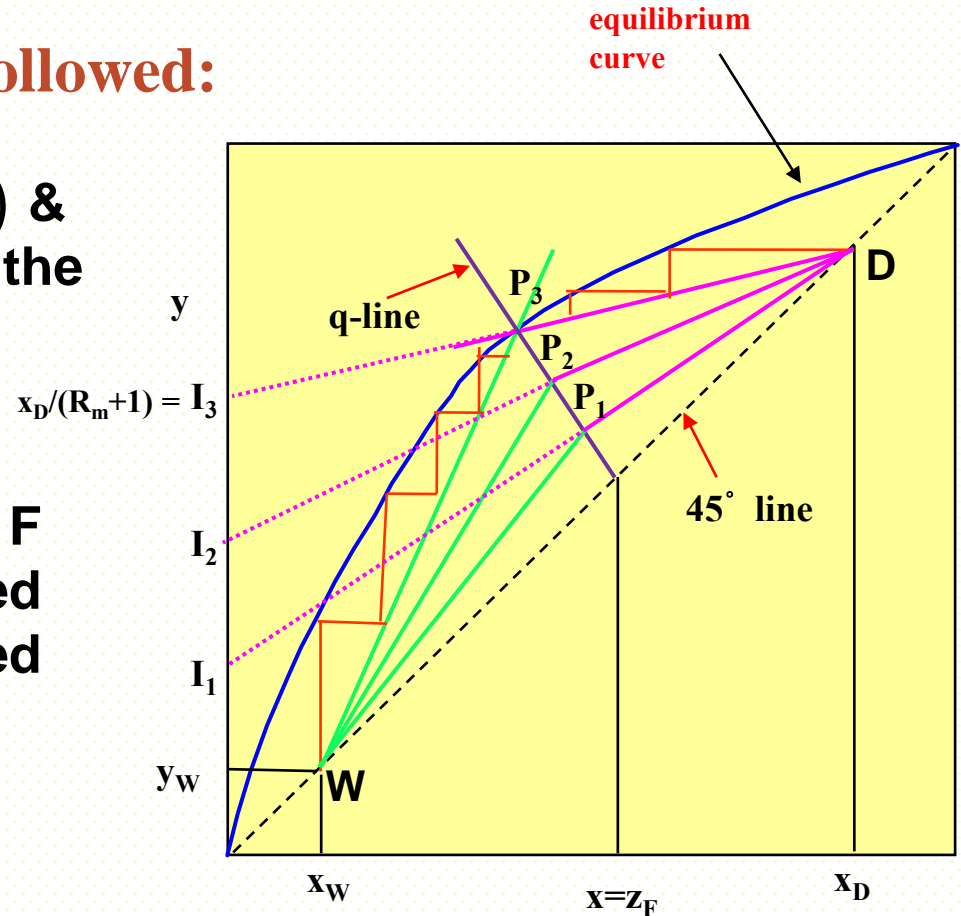
- If the reflux ratio is gradually reduced, a situation will appear when the upper operating line DP_3 intersects the feed line at the point P_3 that lies on the equilibrium curve.
- The driving force is zero at the point P_3 it is *pinch point*.
- The number of theoretical trays required to achieve the given separation becomes infinite. This operating line DP_3 corresponds to the minimum reflux ratio



Minimum Reflux: Pinch Point

Following steps should be followed:

- Locate the points $D(x_D, x_D)$ & $F(Z_F, Z_F)$ on the diagonal on the $x-y$ plane
- Draw the feed line through F from the known feed composition and the feed quality (given by q)
- Locate the point P_3 .
- Join DP_3 and extend to intersect the y -axis at I_3 . Find out the ordinate of I_3 and equate it to $x_D/(R_m+1)$. Calculate the minimum reflux ratio, R_m .



Example

A mixture of 45 mole % n-hexane and 55 mole % n-heptane is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% n-hexane and the residue contains 5% n-hexane. The feed is saturated vapor. The relative volatility of n-hexane in mixture is 2.36. Determine the minimum reflux ratio for this separation.

Solution

A mixture of 45 mole % n-hexane and 55 mole % n-heptane is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% n-hexane and the residue contains 5% n-hexane. The feed is saturated vapor. The relative volatility of n-hexane in mixture is 2.36. Determine the minimum reflux ratio for this separation.

$$y = \frac{\alpha x}{1 + (\alpha - 1)x} = \frac{2.36x}{1 + (2.36 - 1)x} = \frac{2.36x}{1 + 1.36x}$$

x	0	0.076	0.199	0.341	0.505	0.705	1
y	0	0.163	0.37	0.55	0.71	0.85	1

$$\frac{x_D}{R_m + 1} = 0.26$$

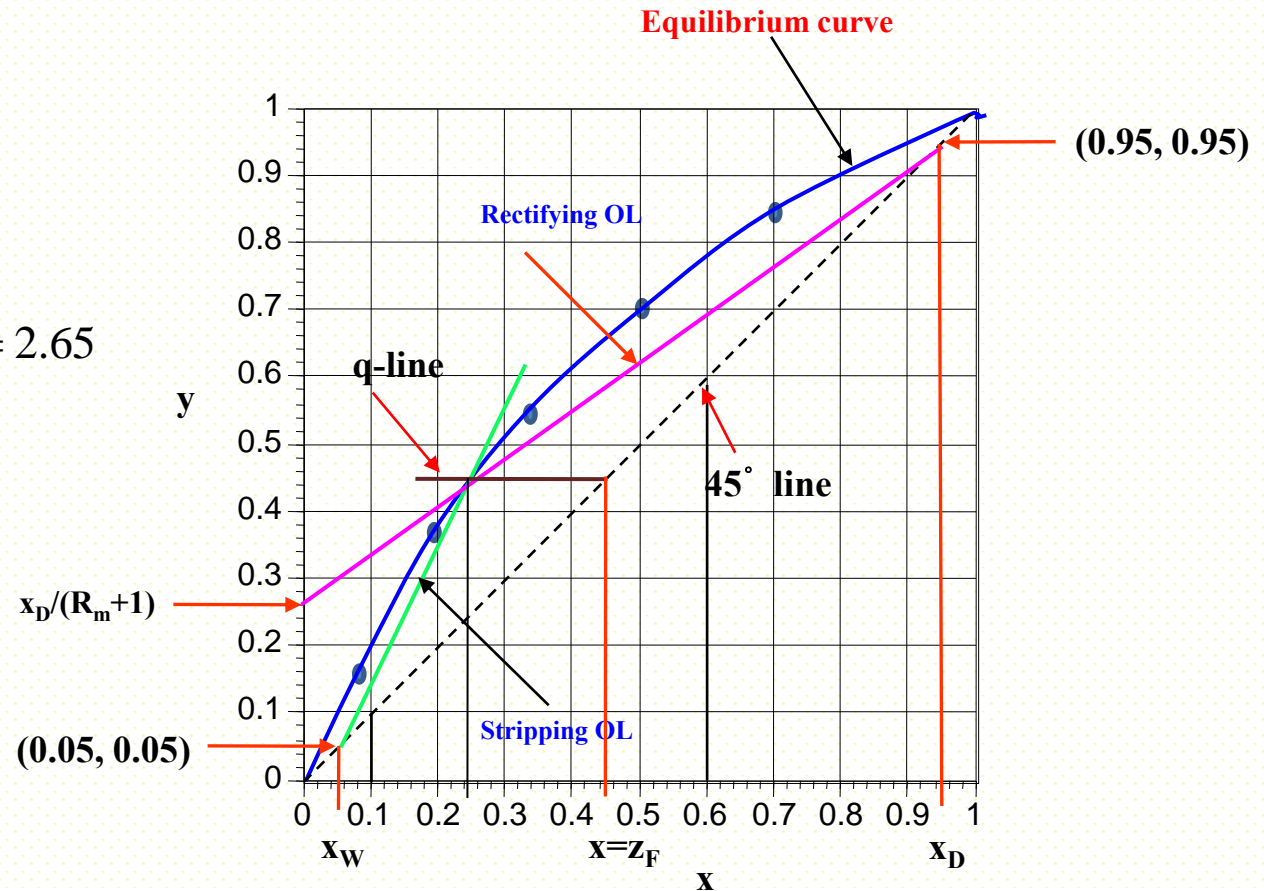
$$\Rightarrow R_m = \frac{0.95}{0.26} - 1 = 3.65 - 1 = 2.65$$

Solution

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$$\frac{x_D}{R_m + 1} = 0.26$$

$$\Rightarrow R_m = \frac{0.95}{0.26} - 1 = 3.65 - 1 = 2.65$$



Example

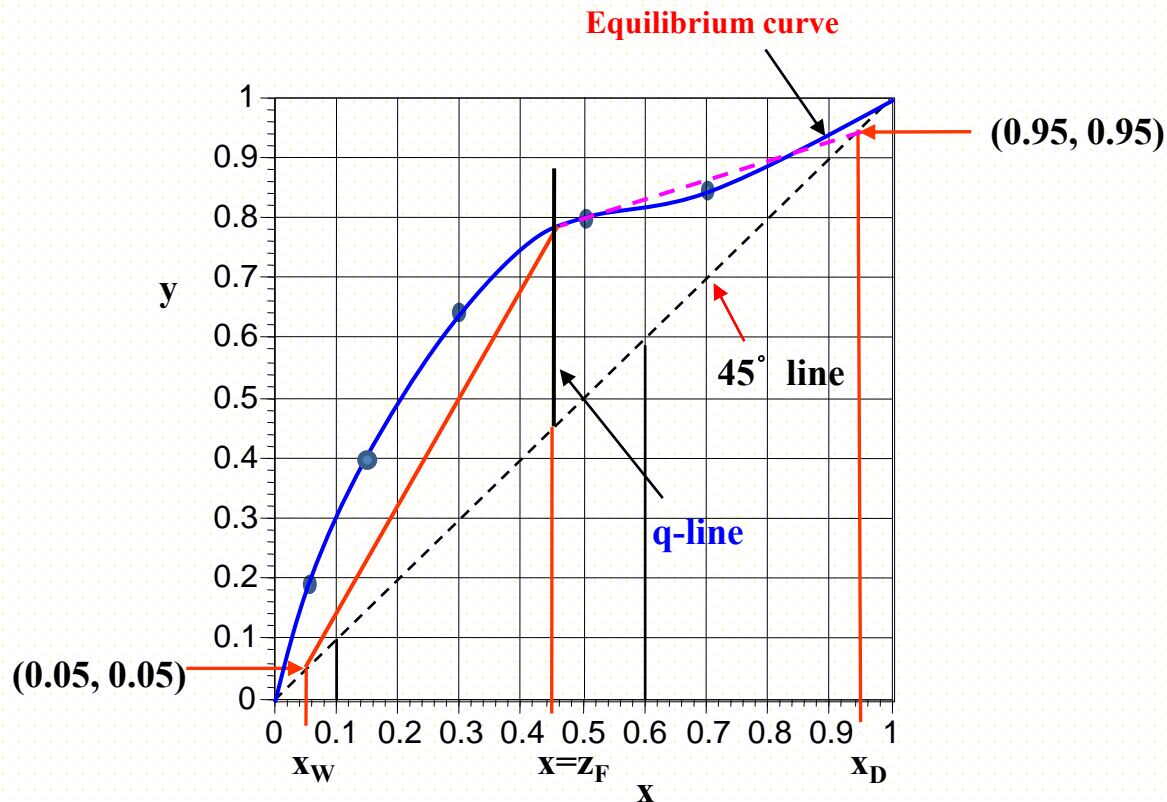
A mixture of 45 mole % A and 55 mole % B is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% A and the residue contains 5% A. The feed is saturated liquid. Determine the minimum reflux ratio for this separation. The equilibrium data for the system is given below:

x	0	0.05	0.15	0.3	0.5	0.7	1
y	0	0.2	0.4	0.65	0.8	0.85	1

Solution

A mixture of 45 mole % A and 55 mole % B is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% A and the residue contains 5% A. The feed is saturated liquid. Determine the minimum reflux ratio for this separation. The equilibrium data for the system is given below:

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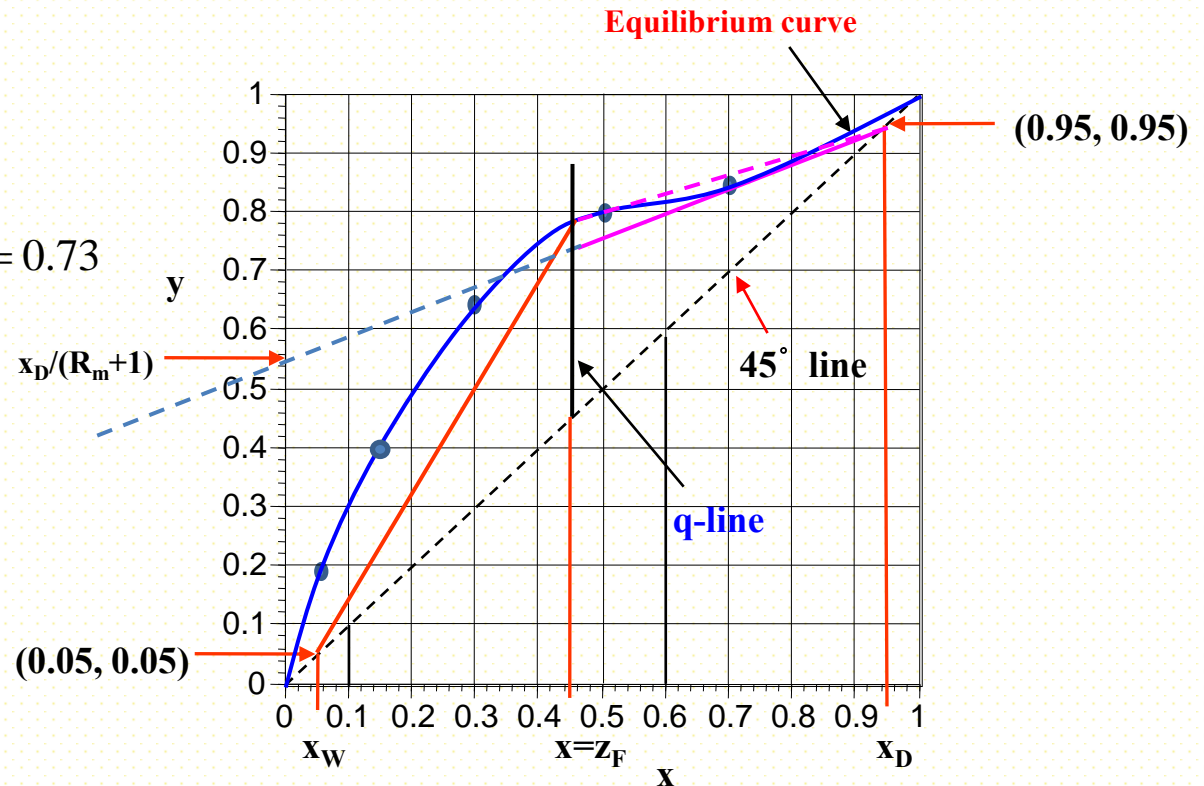
Solution

A mixture of 45 mole % A and 55 mole % B is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% A and the residue contains 5% A. The feed is saturated liquid. Determine the minimum reflux ratio for this separation. The equilibrium data for the system is given below:

x	0	0.05	0.15	0.3	0.5	0.7	1
y	0	0.2	0.4	0.65	0.8	0.85	1

$$\frac{x_D}{R_m + 1} = 0.55$$

$$\Rightarrow R_m = \frac{0.95}{0.55} - 1 = 1.73 - 1 = 0.73$$



THANK YOU