Distillation

PROBLEM 11.1

A liquid containing four components, **A**, **B**, **C** and **D**, with 0.3 mole fraction each of **A**, **B** and **C**, is to be continuously fractionated to give a top product of 0.9 mole fraction **A** and 0.1 mole fraction **B**. The bottoms are to contain not more than 0.5 mole fraction **A**. Estimate the minimum reflux ratio required for this separation, if the relative volatility of **A** to **B** is 2.0.

Solution

The given data may be tabulated as follows:

	Feed	Top	Bottoms
A	0.3	0.9	0.05
B	0.3	0.1	
C	0.3	—	
D	0.1	—	

The Underwood and Fenske equations may be used to find the minimum number of plates and the minimum reflux ratio for a binary system. For a multicomponent system n_m may be found by using the two key components in place of the binary system and the relative volatility between those components in equation 11.56 enables the minimum reflux ratio R_m to be found. Using the feed and top compositions of component **A**:

$$R_m = \frac{1}{\alpha - 1}\left[\left(\frac{x_d}{x_f}\right) - \alpha\frac{(1 - x_d)}{(1 - x_f)}\right] \qquad \text{(equation 11.50)}$$

Thus:
$$R_m = \frac{1}{2 - 1}\left[\left(\frac{0.9}{0.3}\right) - 2\frac{(1 - 0.9)}{(1 - 0.3)}\right] = \underline{\underline{2.71}}$$

PROBLEM 11.2

During the batch distillation of a binary mixture in a packed column the product contained 0.60 mole fraction of the more volatile component when the concentration in the still was 0.40 mole fraction. If the reflux ratio used was 20:1, and the vapour composition y is related to the liquor composition x by the equation $y = 1.035x$ over the range of

concentration concerned, determine the number of ideal plates represented by the column. x and y are in mole fractions.

Solution

It is seen in equation 11.48, the equation of the operating line, that the slope is given by $R/(R+1)(=L/V)$ and the intercept on the y-axis by:

$$x_d/(R+1) = (D/V_n)$$

$$y_n = \frac{R}{R+1}x_{n+1} + \frac{x_d}{R+1} \qquad \text{(equation 11.41)}$$

In this problem, the equilibrium curve over the range $x = 0.40$ to $x = 0.60$ is given by $y = 1.035x$ and it may be drawn as shown in Figure 11a. The intercept of the operating line on the y-axis is equal to $x_d/(R+1) = 0.60/(20+1) = 0.029$ and the operating line is drawn through the points (0.60, 0.60) and (0, 0.029) as shown.
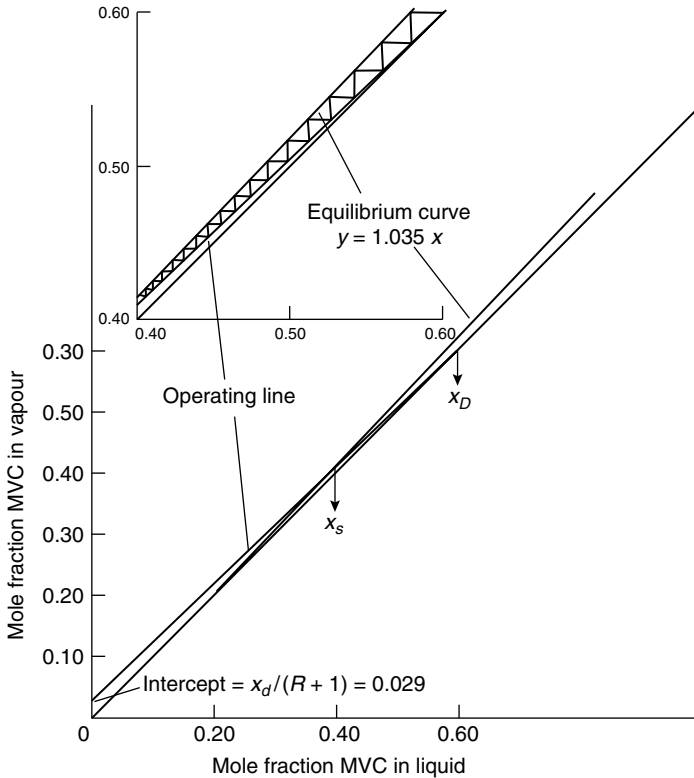

Figure 11a.   Construction for Problem 11.2

In this particular example, all these lines are closely spaced and the relevant section is enlarged in the inset of Figure 11a. By stepping off the theoretical plates as in the McCabe–Thiele method, it is seen that 18 theoretical plates are represented by the column.

PROBLEM 11.3

A mixture of water and ethyl alcohol containing 0.16 mole fraction alcohol is continuously distilled in a plate fractionating column to give a product containing 0.77 mole fraction alcohol and a waste of 0.02 mole fraction alcohol. It is proposed to withdraw 25 per cent of the alcohol in the entering stream as a side stream containing 0.50 mole fraction of alcohol.

Determine the number of theoretical plates required and the plate from which the side stream should be withdrawn if the feed is liquor at its boiling point and a reflux ratio of 2 is used.

Solution

Taking 100 kmol of feed to the column as a basis, 16 kmol of alcohol enter, and 25 per cent, that is 4 kmol, are to be removed in the side stream. As the side-stream composition is to be 0.5, that stream contains 8 kmol.

An overall mass balance gives:

$$F = D + W + S$$

That is:
$$100 = D + W + 8 \quad \text{or} \quad 92 = D + W$$

A mass balance on the alcohol gives:

$$(100 \times 0.16) = 0.77D + 0.02W + 4$$

or:
$$12 = 0.77D + 0.02W.$$

from which: distillate, $D = 13.55$ kmol and bottoms, $W = 78.45$ kmol.

In the top section between the side-stream and the top of the column:

$$R = L_n/D = 2, \text{ and hence } L_n = (2 \times 13.55) = 27.10 \text{ kmol}$$

$$V_n = L_n + D \quad \text{and} \quad V_n = (27.10 + 13.55) = 40.65 \text{ kmol}$$

For the section between the feed and the side stream:

$$V_s = V_n = 40.65, \quad L_n = S + L_s$$

and:
$$L_s = (27.10 - 8) = 19.10 \text{ kmol}$$

At the bottom of the column:

$$L_m = L_s + F = (19.10 + 100) = 119.10, \text{ if the feed is at its boiling-point.}$$

$$V_m = L_m - W = (119.10 - 78.45) = 40.65 \text{ kmol.}$$

The slope of the operating line is always L/V and thus the slope in each part of the column can now be calculated. The top operating line passes through the point (x_d, x_d) and has a slope of $(27.10/40.65) = 0.67$. This is shown in Figure 11b and it applies until $x_s = 0.50$ where the slope becomes $(19.10/40.65) = 0.47$. The operating line in the bottom of the column applies from $x_f = 0.16$ and passes through the point (x_w, x_w) with a slope of $(119.10/40.65) = 2.92$.
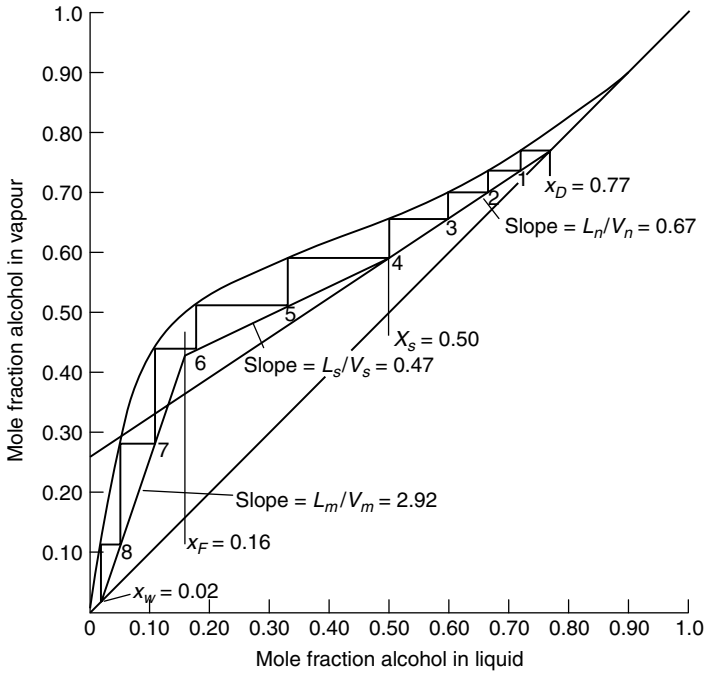
Figure 11b. Graphical construction for Problem 11.3

The steps corresponding to the theoretical plates may be drawn in as shown, and 8 plates are required with the side stream being withdrawn from the fourth plate from the top.

PROBLEM 11.4

In a mixture to be fed to a continuous distillation column, the mole fraction of phenol is 0.35, *o*-cresol is 0.15, *m*-cresol is 0.30 and xylenols is 0.20. A product is required with a mole fraction of phenol of 0.952, *o*-cresol 0.0474 and *m*-cresol 0.0006. If the volatility to *o*-cresol of phenol is 1.26 and of *m*-cresol is 0.70, estimate how many theoretical plates would be required at total reflux.

Solution

The data may be tabulated in terms of mole fractions as follows.

Component	Feed	Top	Bottoms	α
P	0.35	0.952		1.26
O	0.15	0.0474		1.0
M	0.30	0.0006		0.7
X	0.20	—		
		1.0000		

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Fenske's equation may be used to find the minimum number of plates.

Thus the number of plates at total reflux is given by:

$$n + 1 = \frac{\log[(x_A/x_B)_d(x_B/x_A)_s]}{\log \alpha_{AB}}$$ (equation 11.58)

For multicomponent systems, components **A** and **B** refer to the light and heavy keys respectively. In this problem, o-cresol is the light key and m-cresol is the heavy key. A mass balance may be carried out in order to determine the bottom composition. Taking as a basis, 100 kmol of feed, then:

$$100 = D + W$$

For phenol: $(100 \times 0.35) = 0.952D + x_{wp}W$

If x_{wp} is zero then: $D = 36.8 \quad W = 63.2$

For o-cresol: $(100 \times 0.15) = (0.0474 \times 36.8) + (x_{wo} \times 63.2)$ and $x_{wo} = 0.21$

For m-cresol: $(100 \times 0.30) = (0.0006 \times 36.8) + (x_{wm} \times 63.2)$ and $x_{wm} = 0.474$

By difference: $x_{wx} = 0.316 \quad \alpha_{om} = (1/0.7) = 1.43$

Hence, substituting into Fenske's equation gives:

$$n + 1 = \frac{\log[(0.0474/0.0006)(0.474/0.21)]}{\log 1.43}$$

and: $\underline{\underline{n = 13.5}}$

PROBLEM 11.5

A continuous fractionating column, operating at atmospheric pressure, is to be designed to separate a mixture containing 15.67 per cent CS_2 and 84.33 per cent CCl_4 into an overhead product containing 91 per cent CS_2 and a waste of 97.3 per cent CCl_4 all by mass. A plate efficiency of 70 per cent and a reflux of 3.16 kmol/kmol of product may be assumed. Using the following data, determine the number of plates required.

The feed enters at 290 K with a specific heat capacity of 1.7 kJ/kg K and a boiling point of 336 K. The latent heats of CS_2 and CCl_4 are 25.9 kJ/kmol.

CS_2 in the vapour (Mole per cent)	0	8.23	15.55	26.6	33.2	49.5	63.4	74.7	82.9	87.8	93.2
CS_2 in the liquid (Mole per cent)	0	2.36	6.15	11.06	14.35	25.85	33.0	53.18	66.30	75.75	86.04

Solution

The equilibrium data are shown in Figure 11c and the problem may be solved using the method of McCabe and Thiele. All compositions are in terms of mole fractions so that:
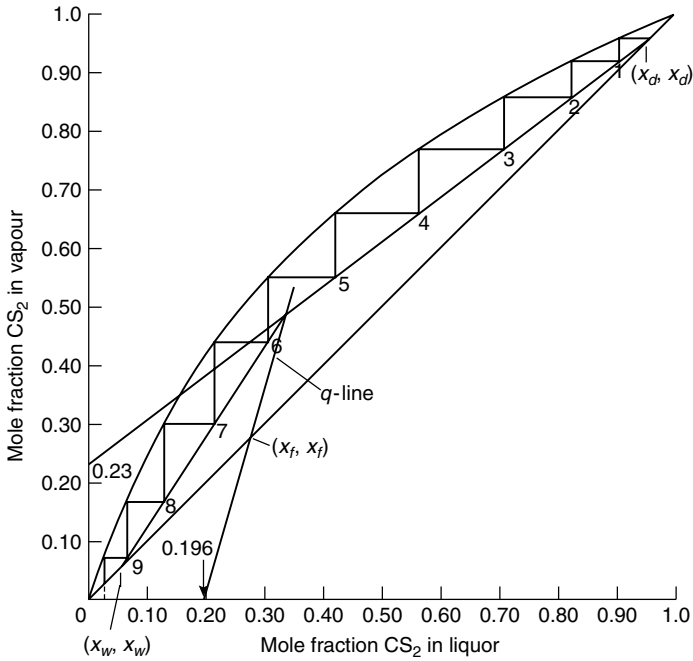
Figure 11c. Equilibrium data for Problem 11.5

Top product:
$$x_d = \frac{(91/76)}{(91/76) + (9/154)} = 0.953$$

Feed:
$$x_f = \frac{(15.67/76)}{(15.67/76) + (84.33/154)} = 0.274$$

Bottom product:
$$x_w = \frac{(2.7/76)}{(2.7/76) + (97.3/154)} = 0.053$$

In this problem, the feed is not at its boiling-point so the slope of the q-line must be determined in order to locate the intersection of the operating lines.

q is defined as the heat required to vaporise 1 kmol of feed/molar latent heat of feed, or

$$q = (\lambda + H_{fs} - H_f)/\lambda$$

where λ is the molar latent heat. H_{fs} is the enthalpy of 1 kmol of feed at its boiling-point, and H_f is the enthalpy of 1 kmol of feed.

The feed composition is 27.4 per cent CS_2 and 72.6 per cent CCl_4 so that the mean molecular mass of the feed is given by:

$$(0.274 \times 76) + (0.726 \times 154) = 132.6 \text{ kg/kmol}$$

Taking a datum of 273 K:

$$H_f = 1.7 \times 132.6(290 - 273) = 3832 \text{ kJ/kmol}$$

$$H_{fs} = 1.7 \times 132.6(336 - 273) = 14,200 \text{ kJ/kmol}$$

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$$\lambda = 25{,}900 \text{ kJ/kmol}$$

Thus: $\qquad\qquad q = (25{,}900 + 14{,}200 - 3832)/25{,}900 = 1.4$

The intercept of the q-line on the x-axis is shown from equation 11.46 to be x_f/q or:

$$y_q = \left(\frac{q}{q-1}\right)x_q - \left(\frac{x_f}{q-1}\right) \qquad \text{(equation 11.46)}$$

$$x_f/q = (0.274/1.4) = 0.196$$

Thus the q-line is drawn through (x_f, x_f) and $(0.196, 0)$ as shown in Figure 11c. As the reflux ratio is given as 3.16, the top operating line may be drawn through (x_d, x_d) and $(0, x_d/4.16)$. The lower operating line is drawn by joining the intersection of the top operating line and the q-line with the point (x_w, x_w).

The theoretical plates may be stepped off as shown and 9 theoretical plates are shown. If the plate efficiency is 70 per cent, the number of actual plates $= (9/0.7) = 12.85$,

Thus: $\qquad\qquad\qquad\qquad$ <u>13 plates are required</u>

PROBLEM 11.6

A batch fractionation is carried out in a small column which has the separating power of 6 theoretical plates. The mixture consists of benzene and toluene containing 0.60 mole fraction of benzene. A distillate is required, of constant composition, of 0.98 mole fraction benzene, and the operation is discontinued when 83 per cent of the benzene charged has been removed as distillate. Estimate the reflux ratio needed at the start and finish of the distillation, if the relative volatility of benzene to toluene is 2.46.

Solution

The equilibrium data are calculated from the relative volatility by the equation:

$$y_A = \frac{\alpha x_A}{1 + (\alpha - 1)x_A} \qquad \text{(equation 11.16)}$$

to give:

x_A	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
y_A	0	0.215	0.380	0.513	0.621	0.711	0.787	0.852	0.908	0.956	1.0

If a constant product is to be obtained from a batch still, the reflux ratio must be constantly increased. Initially S_1 kmol of liquor are in the still with a composition x_{s1} of the MVC and a reflux ratio of R_1 is required to give the desired product composition x_d. When S_2 kmol remain in the still of composition x_{s2}, the reflux ratio has increased to R_2 when the amount of product is D kmol.

From an overall mass balance: $\quad S_1 - S_2 = D$

For the MVC: $\qquad\qquad\qquad\qquad S_1 x_{s1} - S_2 x_{s2} = D x_d$

from which: $\qquad\qquad\qquad\qquad\qquad D = S_1 \dfrac{(x_{s1} - x_{s2})}{(x_d - x_{s2})} \qquad\qquad$ (equation 11.98)

In this problem, $x_{s1} = 0.6$ and $x_d = 0.98$ and there are 6 theoretical plates in the column. It remains, by using the equilibrium data, to determine values of x_{s2} for selected reflux ratios. This is done graphically by choosing an intercept on the y-axis, calculating R, drawing in the resulting operating line, and stepping off in the normal way 6 theoretical plates and finding the still composition x_{s2}.

This is shown in Figure 11d for two very different reflux ratios and the procedure is repeated to give the following table.

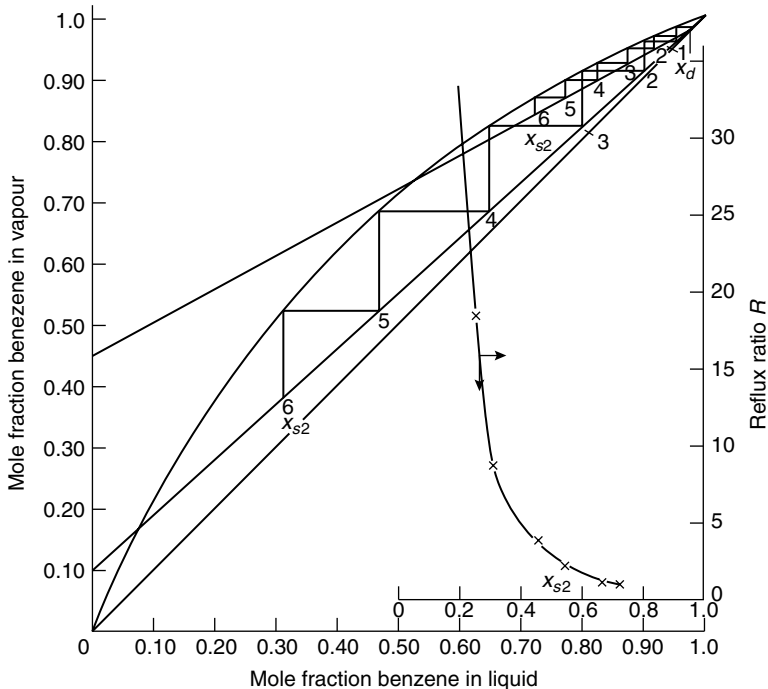| Intercept on y-axis (ϕ)	Reflux ratio ($\phi = x_d/R + 1$)	x_{s2}
0.45	1.18	0.725
0.40	1.20	0.665
0.30	2.27	0.545
0.20	3.90	0.46
0.10	8.8	0.31
0.05	18.6	0.26

Figure 11d. Equilibrium data for Problem 11.6

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From the inset plot of x_{s2} against R in Figure 11d:

At the start: $x_{s2} = 0.6$ and $\underline{\underline{R = 1.7}}$.

At the end: x_{s2} is calculated using equation 11.98 as follows.
If $S_1 = 100$ kmol,

$$\text{kmol of benzene initially} = (100 \times 0.60) = 60 \text{ kmol.}$$

$$\text{kmol of benzene removed} = (0.83 \times 60) = 49.8 \text{ kmol.}$$

Thus: $$D = (49.8/0.98) = 50.8$$

and: $$50.8 = 100\frac{(0.6 - x_{s2})}{(0.98 - x_{s2})}$$

from which: $$x_{s2} = 0.207 \quad \text{and} \quad \underline{\underline{R = 32}}$$

PROBLEM 11.7

A continuous fractionating column is required to separate a mixture containing 0.695 mole fraction n-heptane (C_7H_{16}) and 0.305 mole fraction n-octane (C_8H_{18}) into products of 99 mole per cent purity. The column is to operate at 101.3 kN/m^2 with a vapour velocity of 0.6 m/s. The feed is all liquid at its boiling-point, and this is supplied to the column at 1.25 kg/s. The boiling-point at the top of the column may be taken as 372 K, and the equilibrium data are:

mole fraction of								
heptane in vapour	0.96	0.91	0.83	0.74	0.65	0.50	0.37	0.24
mole fraction of								
heptane in liquid	0.92	0.82	0.69	0.57	0.46	0.32	0.22	0.13

Determine the minimum reflux ratio required. What diameter column would be required if the reflux used were twice the minimum possible?

Solution

The equilibrium curve is plotted in Figure 11e. As the feed is at its boiling-point, the q-line is vertical and the minimum reflux ratio may be found by joining the point (x_d, x_d) with the intersection of the q-line and the equilibrium curve. This line when produced to the y-axis gives an intercept of 0.475.

Thus: $$0.475 = x_D/(R_m + 1) \quad \text{and} \quad R_m = \underline{\underline{1.08}}$$

If $2R_m$ is used, then: $$R = 2.16 \quad \text{and} \quad L_n/D = 2.16$$

Taking 100 kmol of feed, as a basis, an overall mass balance and a balance for the n-heptane give:

$$100 = (D + W)$$

and: $$100 \times 0.695 = 0.99D + 0.01W$$

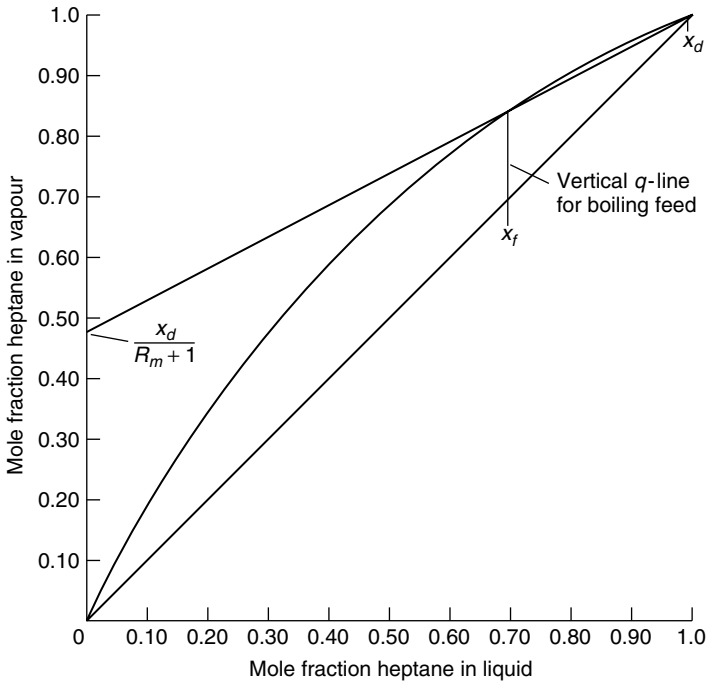since 99 per cent n-octane is required.

Figure 11e. Geometrical construction for Problem 11.7

Hence: $\qquad\qquad\qquad D = 69.9 \quad \text{and} \quad W = 30.1$

and: $\qquad\qquad\qquad L_n = 2.16D = 151 \quad \text{and} \quad V_n = L_n + D = 221$

The mean molecular mass of the feed $= (0.695 \times 100) + (0.305 \times 114) = 104.3 \text{ kg/kmol}$.

Thus: $\qquad\qquad\qquad$ feed rate $= (1.25/104.3) = 0.0120 \text{ kmol/s}$

The vapour flow at the top of the column $= (221/100) \times 0.0120 = 0.0265 \text{ kmol/s}$.

The vapour density at the top of the column $= (1/22.4)(273/372) = 0.0328 \text{ kmol/m}^3$.

Hence the volumetric vapour flow $= (0.0265/0.0328) = 0.808 \text{ m}^3/\text{s}$.

If the vapour velocity $= 0.6 \text{ m/s}$, the area required $= (0.808/0.6) = 1.35 \text{ m}^2$ equivalent to a column diameter of $[(4 \times 1.35)/\pi]^{0.5} = \underline{\underline{1.31 \text{ m}}}$.

PROBLEM 11.8

The vapour pressures of chlorobenzene and water are:

Vapour pressure (kN/m²)	13.3	6.7	4.0	2.7
(mm Hg)	100	50	30	20
Temperatures, (K)				
Chlorobenzene	343.6	326.9	315.9	307.7
Water	324.9	311.7	303.1	295.7

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A still is operated at 18 kN/m^2 and steam is blown continuously into it. Estimate the temperature of the boiling liquid and the composition of the distillate if liquid water is present in the still.

Solution

For steam distillation, assuming the gas laws to apply, the composition of the vapour produced may be obtained from:

$$\left(\frac{m_A}{M_A}\right) \bigg/ \left(\frac{m_B}{M_B}\right) = \frac{P_A}{P_B} = \frac{y_A}{y_B} = \frac{P_A}{(P - P_A)} \qquad \text{(equation 11.120)}$$

where the subscript **A** refers to the component being recovered and **B** to steam, and m is the mass, M is the molecular mass, P_A and P_B are the partial pressures of **A** and **B** and P is the total pressure.

If there is no liquid phase present, then from the phase rule there will be two degrees of freedom. Thus both the total pressure and the operating temperature can be fixed independently, and $P_B = P - P_A$ (which must not exceed the vapour pressure of pure water if no liquid phase is to appear).

With a liquid water phase present, there will only be one degree of freedom, and setting the temperature or pressure fixes the system and the water and the other component each exert a partial pressure equal to its vapour pressure at the boiling-point of the mixture. In this case, the distillation temperature will always be less than that of boiling water at the total pressure in question. Consequently, a high-boiling organic material may be steam-distilled at temperatures below 373 K at atmospheric pressure. By using reduced operating pressures, the distillation temperature may be reduced still further, with a consequent economy of steam.

A convenient method of calculating the temperature and composition of the vapour, for the case where the liquid water phase is present, is by using Figure 11.47 in Volume 2 where the parameter $(P - P_B)$ is plotted for total pressures of 101, 40 and 9.3 kN/m^2 and the vapour pressures of a number of other materials are plotted directly against temperature. The intersection of the two appropriate curves gives the temperature of distillation and the molar ratio of water to organic material is given by $(P - P_A)/P_A$.

The relevance of the method to this problem is illustrated in Figure 11f where the vapour pressure of chlorobenzene is plotted as a function of temperature. On the same graph $(P - P_B)$ is plotted where $P = 18$ kN/m^2 (130 mm Hg) and P_B is the vapour pressure of water at the particular temperature. These curves are seen to intersect at the distillation temperature of 323 K.

The composition of the distillate is found by substitution in equation 11.120 since $P_A = 5.5$ kN/m^2 (41 mmHg) at 323 K.

Hence:
$$\frac{y_A}{y_B} = \frac{P_A}{(P - P_A)} = \frac{5.5}{(18 - 5.5)} = \underline{\underline{0.44}}$$
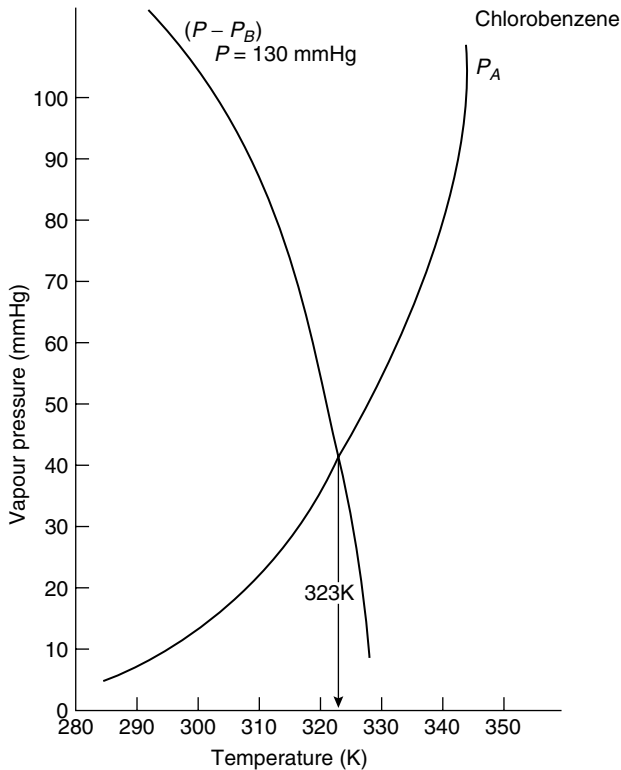
Figure 11f. Vapour pressure as a function of temperature, Problem 11.8

PROBLEM 11.9

The following values represent the equilibrium conditions in terms of mole fraction of benzene in benzene–toluene mixtures at their boiling-point:

Liquid	0.521	0.38	0.26	0.15
Vapour	0.72	0.60	0.45	0.30

If the liquid compositions on four adjacent plates in a column were 0.18, 0.28, 0.41 and 0.57 under conditions of total reflux, determine the plate efficiencies.

Solution

The equilibrium data are plotted in Figure 11g over the range given and a graphical representation of the plate efficiency is shown in the inset. The efficiency E_{Ml} in terms of the liquid compositions is defined by:

$$E_{Ml} = \frac{(x_{n+1} - x_n)}{(x_{n+1} - x_e)}$$
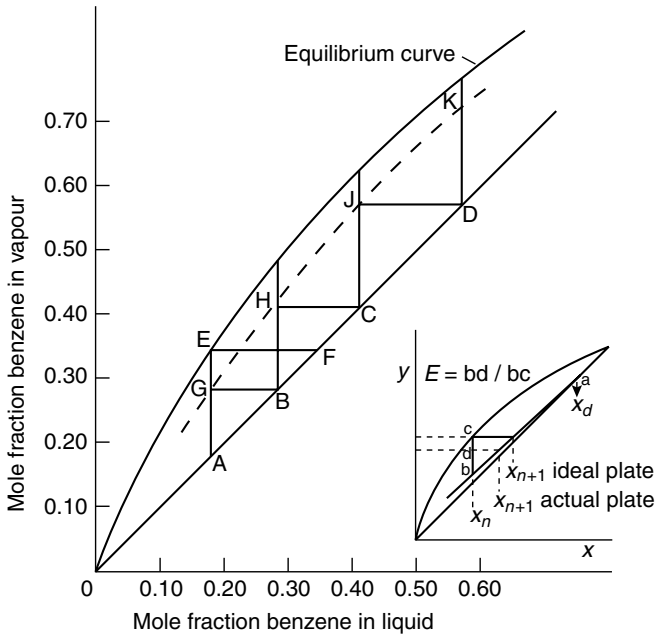
(equation 11.125)

Figure 11g. Graphical construction for Problem 11.9

In the inset, the line ab represents an operating line and bc is the enrichment achieved on a theoretical plate. bd is the enrichment achieved on an actual plate so that the efficiency is then the ratio ba/bc.

Referring to the data given, at total reflux, the conditions on actual plates in the column are shown as points A, B, C, and D. Considering point A, if equilibrium were achieved on that plate, point E would represent the vapour composition and point F the liquid composition on the next plate. The liquid on the next plate is determined by B however so that the line AGE may be located and the efficiency is given by AG/AE = 0.59 or 59 per cent

In an exactly similar way, points H, J, and K are located to give efficiencies of 66 per cent, 74 per cent, and 77 per cent.

PROBLEM 11.10

A continuous rectifying column handles a mixture consisting of 40 per cent of benzene by mass and 60 per cent of toluene at the rate of 4 kg/s, and separates it into a product containing 97 per cent of benzene and a liquid containing 98 per cent toluene. The feed is liquid at its boiling-point.

(a) Calculate the mass flows of distillate and waste liquor.
(b) If a reflux ratio of 3.5 is employed, how many plates are required in the rectifying part of the column?

(c) What is the actual number of plates if the plate-efficiency is 60 per cent?

Mole fraction of benzene in liquid	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
Mole fraction of benzene in vapour	0.22	0.38	0.51	0.63	0.7	0.78	0.85	0.91	0.96

Solution

The equilibrium data are plotted in Figure 11h. As the compositions are given as mass per cent, these must first be converted to mole fractions before the McCabe–Thiele method may be used.

Mole fraction of benzene in feed, $\quad x_f = \dfrac{(40/78)}{(40/78) + (60/92)} = 0.440$

Similarly: $\qquad\qquad\qquad\qquad x_d = 0.974 \quad \text{and} \quad x_w = 0.024$

As the feed is a liquid at its boiling-point, the q-line is vertical and may be drawn at $x_f = 0.44$.
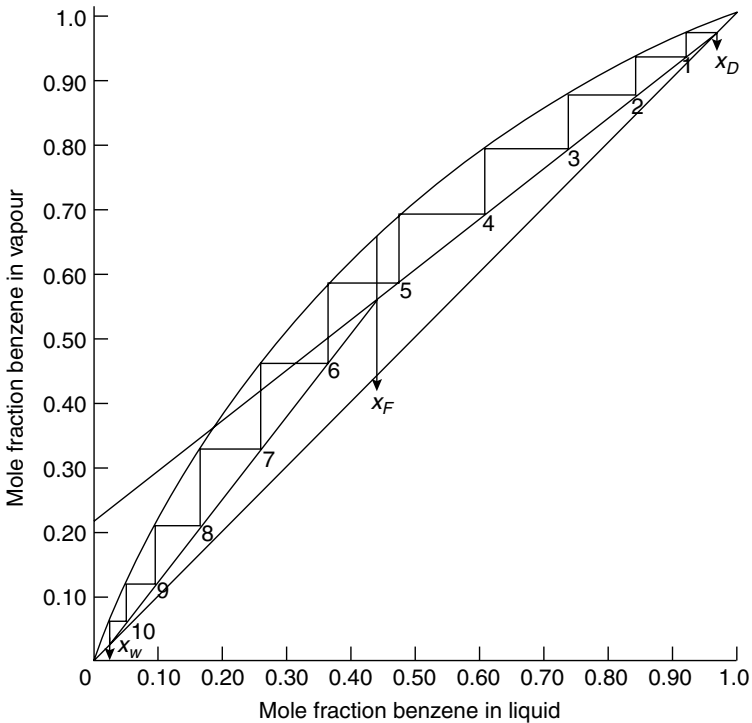

Figure 11h. Graphical construction for Problem 11.10

(a) A mass balance over the column and on the more volatile component in terms of the mass flow rates gives:

$$4.0 = W' + D'$$

$$(4 \times 0.4) = 0.02W' + 0.97D'$$

from which: bottoms flowrate, $W' = \underline{\underline{2.4 \text{ kg/s}}}$

and: top product rate, $D' = \underline{\underline{1.6 \text{ kg/s}}}$

(b) If $R = 3.5$, the intercept of the top operating line on the y-axis is given by $x_d/(R + 1) = (0.974/4.5) = 0.216$, and thus the operating lines may be drawn as shown in Figure 11h. The plates are stepped off as shown and $\underline{\underline{10 \text{ theoretical plates}}}$ are required.

(c) If the efficiency is 60 per cent, the number of actual plates $= (10/0.6)$

$$= 16.7 \text{ or } \underline{\underline{17 \text{ actual plates}}}$$

PROBLEM 11.11

A distillation column is fed with a mixture of benzene and toluene, in which the mole fraction of benzene is 0.35. The column is to yield a product in which the mole fraction of benzene is 0.95, when working with a reflux ratio of 3.2, and the waste from the column is not to exceed 0.05 mole fraction of benzene. If the plate efficiency is 60 per cent, estimate the number of plates required and the position of the feed point. The relation between the mole fraction of benzene in liquid and in vapour is given by:

Mole fraction of benzene in liquid	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
Mole fraction of benzene in vapour	0.20	0.38	0.51	0.63	0.71	0.78	0.85	0.91	0.96

Solution

The solution to this problem is very similar to that of Problem 11.10 except that the data are presented here in terms of mole fractions. Following a similar approach, the theoretical plates are stepped off and it is seen from Figure 11i that 10 plates are required. Thus $(10/0.6) = 16.7$ actual plates are required and $\underline{\underline{17}}$ would be employed.

The feed tray lies between ideal trays 5 and 6, and in practice, the $\underline{\underline{\text{eighth actual tray}}}$ from the top would be used.
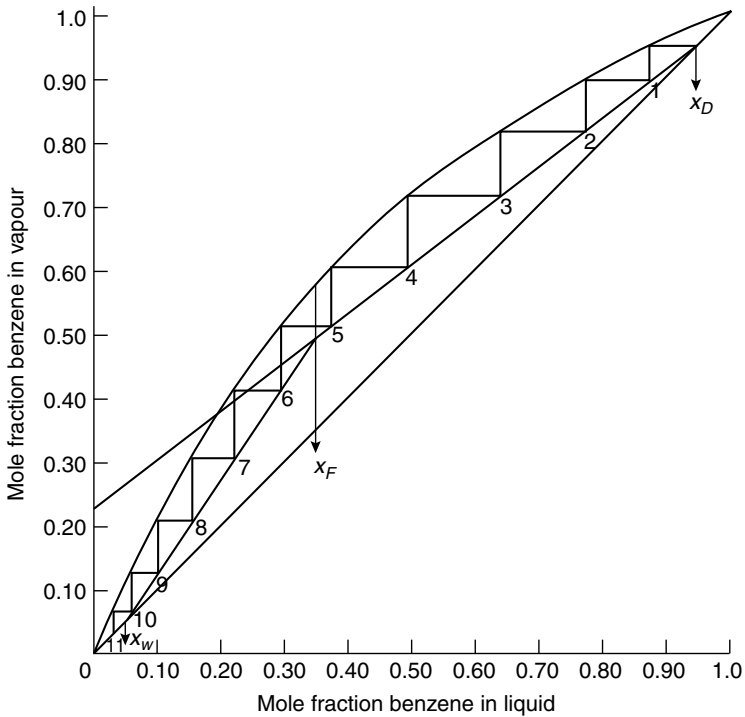
Figure 11i. Graphical construction for Problem 11.11

PROBLEM 11.12

The relationship between the mole fraction of carbon disulphide in the liquid and in the vapour during the distillation of a carbon disulphide–carbon tetrachloride mixture is:

x	0	0.20	0.40	0.60	0.80	1.00
y	0	0.445	0.65	0.795	0.91	1.00

Determine graphically the theoretical number of plates required for the rectifying and stripping portions of the column.

The reflux ratio = 3, the slope of the fractionating line = 1.4, the purity of product = 99 per cent, and the concentration of carbon disulphide in the waste liquors = 1 per cent.

What is the minimum slope of the rectifying line in this case?

Solution

The equilibrium data are plotted in Figure 11j. In this problem, no data are provided on the composition or the nature of the feed so that conventional location of the q-line
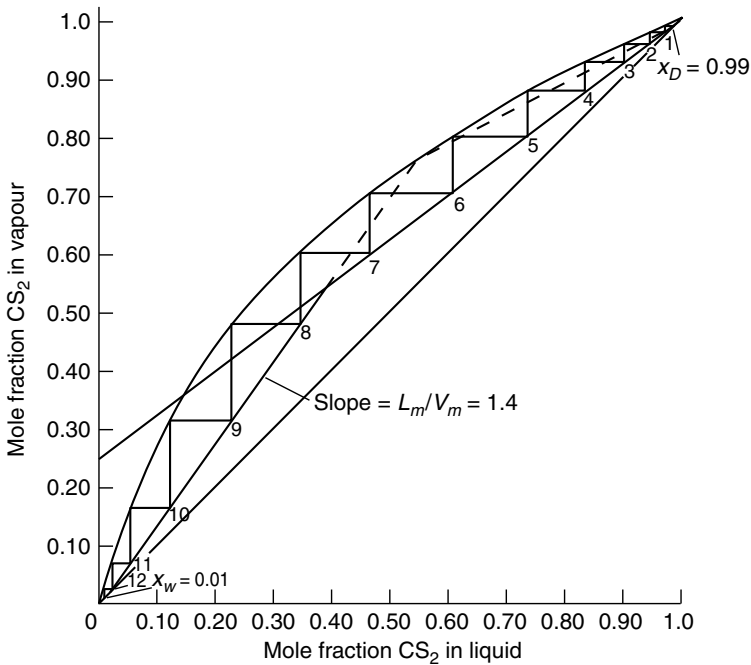
Figure 11j. Equilibrium data for Problem 11.12

is impossible. The rectifying line may be drawn, however, as both the top composition and the reflux ratio are known. The intercept on the y-axis is given by $x_d/(R+1) = (0.99/4) = 0.248$.

The slope of the lower operating line is given as 1.4. Thus the line may be drawn through the point (x_w, x_w) and the number of theoretical plates may be determined, as shown, as 12.

The minimum slope of the rectifying line corresponds to an infinite number of theoretical stages. If the slope of the stripping line remains constant, then production of that line to the equilibrium curve enables the rectifying line to be drawn as shown dotted in Figure 11j.

The slope of this line may be measured to give $L_n/V_n = 0.51$

PROBLEM 11.13

A fractionating column is required to distill a liquid containing 25 per cent benzene and 75 per cent toluene by mass, to give a product of 90 per cent benzene. A reflux ratio of 3.5 is to be used, and the feed will enter at its boiling point. If the plates used are 100 per cent efficient, calculate by the Lewis–Sorel method the composition of liquid on the third plate, and estimate the number of plates required using the McCabe–Thiele method.

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Solution

The equilibrium data for this problem are plotted as Figure 11k. Converting mass per cent to mole fraction gives $x_f = 0.282$ and $x_d = 0.913$. There are no data given on the bottom product so that usual mass balances cannot be applied. The equation of the top operating line is:

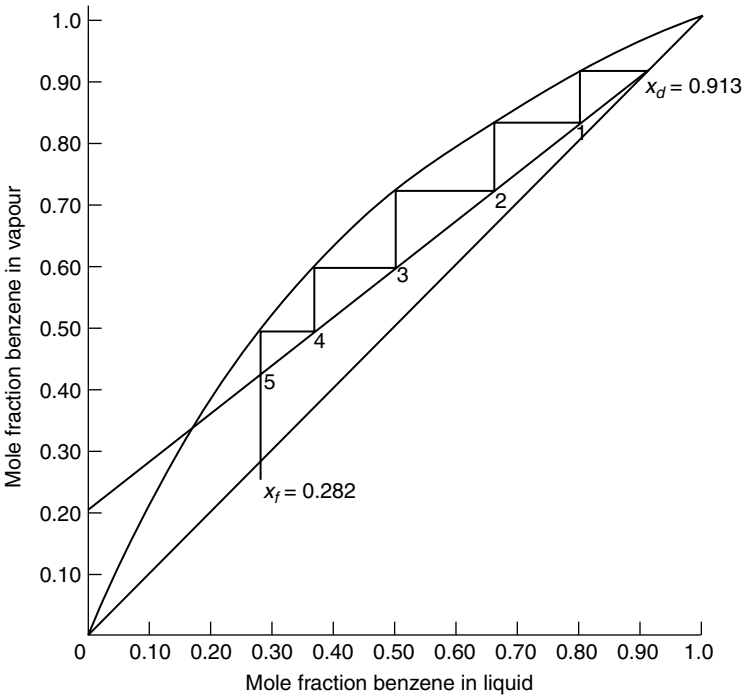$$y_n = \frac{L_n}{V_n}x_{n+1} + \frac{D}{V_n}x_d \qquad \text{(equation 11.35)}$$

Figure 11k. Equilibrium data for Problem 11.13

L_n/V_n is the slope of the top operating line which passes through the points (x_d, x_d) and 0, $x_d/(R + 1)$. This line is drawn in Figure 11k and its slope measured or calculated as 0.78. The reflux ratio which is equal to L_n/D is given as 3.5, so that D/V_n may then be found since:

$$\frac{D}{V_n} = \frac{D}{L_n} = \left(\frac{1}{3.5} \times 0.78\right) = 0.22$$

Thus:
$$y_n = 0.78_{n+1} + 0.22x_d = 0.78_{n+1} + 0.20$$

The composition of the vapour y_t leaving the top plate must be the same as the top product x_d since all the vapour is condensed. The composition of the liquid on the top plate x_t is found from the equilibrium curve since it is in equilibrium with vapour of composition $y_t = x_d = 0.913$.

Thus:
$$x_t = 0.805$$

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The composition of the vapour rising to the top plate y_{t-1} is found from the equation of the operating line. That is:

$$y_{t-1} = (0.78 \times 0.805) + 0.20 = 0.828$$

x_{t-1} is in equilibrium with y_{t-1} and is found to be 0.66 from the equilibrium curve.

Then: $y_{t-2} = (0.78 \times 0.66) + 0.20 = 0.715$

Similarly: $x_{t-2} = 0.50$

and: $y_{t-3} = (0.78 \times 0.50) + 0.20 = 0.557$

and: $\underline{\underline{x_{t-3} = 0.335}}$

The McCabe–Thiele construction in Figure 11k shows that $\underline{\underline{\text{5 theoretical plates}}}$ are required in the rectifying section.

PROBLEM 11.14

A 50 mole per cent mixture of benzene and toluene is fractionated in a batch still which has the separating power of 8 theoretical plates. It is proposed to obtain a constant quality product containing 95 mole per cent benzene, and to continue the distillation until the still has a content of 10 mole per cent benzene. What will be the range of reflux ratios used in the process? Show graphically the relation between the required reflux ratio and the amount of distillate removed.

Solution

If a constant product is to be obtained from a batch still, the reflux ratio must be constantly increased. Initially S_1 kmol of liquor is in the still with a composition x_{s_1} of the MVC and a reflux ratio of R_1 is required to give the desired product composition x_d. When S_2 kmol remain in the still of composition x_{s_2}, the amount of product is D kmol and the reflux ratio has increased to R_2.

From an overall mass balance: $(S_1 - S_2) = D$

For the MVC: $S_1 x_{s_1} - S_2 x_{s_2} = D x_d$

from which: $$D = S_1 \frac{(x_{s_1} - x_{s_2})}{(x_d - x_{s_2})}$$ (equation 11.98)

In this problem, $x_{s_1} = 0.5$ and $x_d = 0.95$ and there are 8 theoretical plates in the column. It remains, by using the equilibrium data, to determine values of x_{s_2} for selected reflux ratios. This is done graphically by choosing an intercept on the y-axis, calculating R, drawing in the resulting operating line, and stepping off in the usual way 8 theoretical plates and finding the still composition x_{s_2} and hence D. The results of this process are as follows for $S_1 = 100$ kmol.

$\phi = x_d/(R+1)$	R	x_{s_2}	D
0.4	1.375	0.48	4.2
0.35	1.71	0.405	17.3
0.30	2.17	0.335	26.8
0.25	2.80	0.265	34.3
0.20	3.75	0.195	40.3
0.15	5.33	0.130	45.1
0.10	8.50	0.090	47.7

The initial and final values of R are most easily determined by plotting R against x_{s_2} as shown in Figure 11l. The initial value of R corresponds to the initial still composition of 0.50 and is seen to be 1.3 and, at the end of the process when $x_{s_2} = 0.1$, $R = 7.0$.
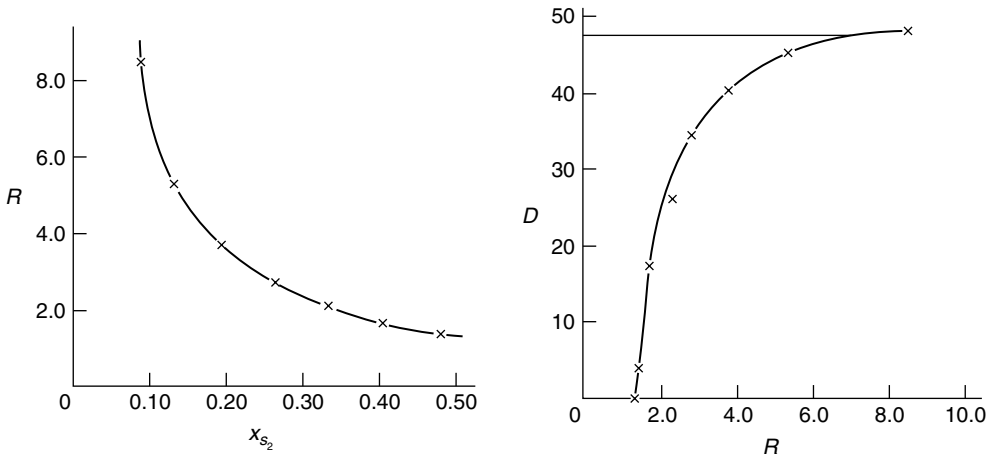
Figure 11l. Reflux ratio data for Problem 11.14

Figure 11l includes a plot of reflux ratio against the quantity of distillate. When $R = 7.0$, $D = 47.1$ kmol/100 kmol charged initially.

PROBLEM 11.15

The vapour composition on a plate of a distillation column is:

	C_1	C_2	$i - C_3$	$n - C_3$	$i - C_4$	$n - C_4$
mole fraction	0.025	0.205	0.210	0.465	0.045	0.050
relative volatility	36.5	7.4	3.0	2.7	1.3	1.0

What will be the composition of the liquid on the plate of it is in equilibrium with the vapour?

Solution

In a mixture of **A**, **B**, **C**, **D**, and so on, if the mole fractions in the liquid are x_A, x_B, x_C, x_D, and so on, and in the vapour y_A, y_B, y_C, and y_D, then:

$$x_A + x_B + x_C + \cdots = 1$$

Thus:

$$\frac{x_A}{x_B} + \frac{x_B}{x_B} + \frac{x_C}{x_B} + \cdots = \frac{1}{x_B}$$

But:

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

Thus:

$$\frac{y_A}{y_B \alpha_{AB}} + \frac{y_B}{y_B \alpha_{BB}} + \frac{y_C}{y_B \alpha_{CB}} + \cdots = \frac{1}{x_B}$$

or:

$$\sum \left(\frac{y_A}{\alpha_{AB}} \right) = \frac{y_B}{x_B}$$

But:

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

and substituting:

$$\sum \left(\frac{y_A}{\alpha_{AB}} \right) = \frac{y_A x_B}{\alpha_{AB} x_A x_B}$$

Thus

$$x_A = \frac{(y_A / \alpha_{AB})}{\sum (y_A / \alpha_{AB})}$$

Similarly:

$$x_B = \frac{(y_B / \alpha_{BB})}{\sum (y_A / \alpha_{AB})} \quad \text{and} \quad x_C = \frac{(y_C / \alpha_{BC})}{\sum (y_A / \alpha_{AB})}$$

These relationships may be used to solve this problem and the calculation is best carried out in tabular form as follows.

Component	y_i	α	y_i / α	$x_i = (y_i / \alpha) / \sum (y_i / \alpha)$
C_1	0.025	36.5	0.00068	0.002
C_2	0.205	7.4	0.0277	0.078
$i - C_3$	0.210	3.0	0.070	0.197
$n - C_3$	0.465	2.7	0.1722	0.485
$i - C_4$	0.045	1.3	0.0346	0.097
$n - C_4$	0.050	1.0	0.050	0.141
			$\sum (y_i / \alpha) = 0.355$	1.000

PROBLEM 11.16

A liquor of 0.30 mole fraction of benzene and the rest toluene is fed to a continuous still to give a top product of 0.90 mole fraction benzene and a bottom product of 0.95 mole fraction toluene.

If the reflux ratio is 5.0, how many plates are required:

(a) if the feed is saturated vapour?
(b) if the feed is liquid at 283 K?

Solution

In this problem, the q-lines have two widely differing slopes and the effect of the feed condition is to alter the number of theoretical stages as shown in Figure 11m.
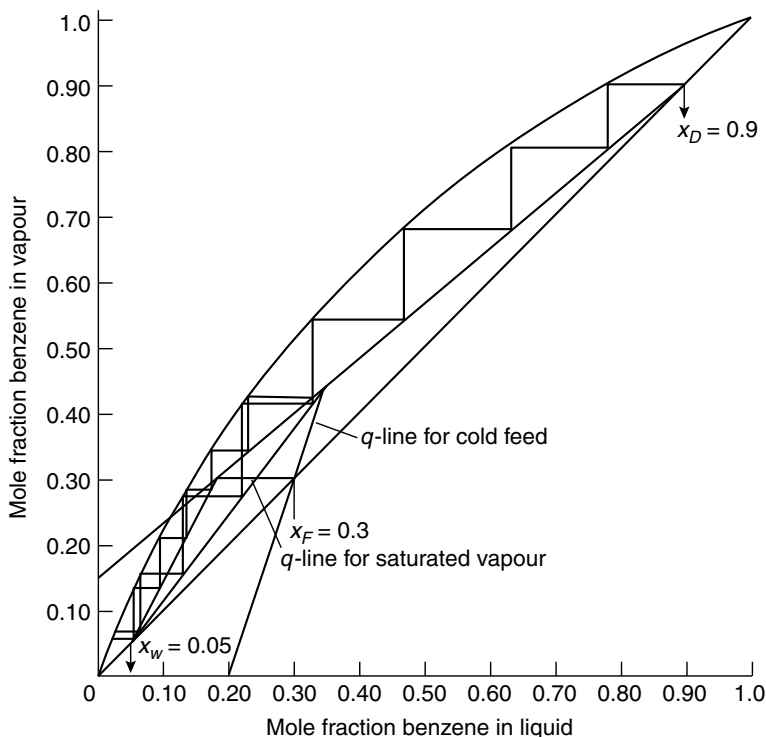
Figure 11m. Equilibrium data for Problem 11.16

$$q = \frac{\lambda + H_{fs} - H_f}{\lambda}$$

where λ is the molar latent heat of vaporisation, H_{fs} is the molar enthalpy of the feed at its boiling-point, and H_f is the molar enthalpy of the feed.

For benzene and toluene: $\qquad\qquad \lambda = 30$ MJ/kmol

and: $\qquad\qquad$ specific heat capacity $= 1.84$ kJ/kg K.

The boiling-points of benzene and toluene are 353.3 and 383.8 K respectively.

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(a) If the feed is a saturated vapour, $q = 0$.

(b) If the feed is a cold liquid at 283 K, the mean molecular mass is:

$$(0.3 \times 78) + (0.7 \times 92) = 87.8 \text{ kg/kmol}$$

and the mean boiling-point $= (0.3 \times 353.3) \times (0.7 \times 383.8) = 374.7$ K.

Using a datum of 273 K:

$$H_{fs} = 1.84 \times 87.8(374.7 - 273) = 16{,}425 \text{ kJ/kmol} \quad \text{or} \quad 16.43 \text{ MJ/kmol}$$

$$H_f = 1.84 \times 87.8(283 - 273) = 1615 \text{ kJ/kmol} \quad \text{or} \quad 1.615 \text{ MJ/kmol}$$

Thus: $\qquad\qquad\qquad q = (30 + 16.43 - 1.615)/30 = 1.49.$

From equation 11.46, the slope of the q-line is $q/(q - 1)$.

Hence the slope $= (1.49/0.49) = 3.05$.

Thus for (a) and (b) the slope of the q-line is zero and 3.05 respectively, and in Figure 11m these lines are drawn through the point (x_f, x_f).

By stepping off the ideal stages, the following results are obtained:

Feed	Theoretical plates		
	Stripping section	Rectifying section	Total
Saturated vapour	4	5	9
Cold liquid	4	3	7

Thus a cold feed requires fewer plates than a vapour feed although the capital cost saving is offset by the increased heat load on the reboiler.

PROBLEM 11.17

A mixture of alcohol and water containing 0.45 mole fraction of alcohol is to be continuously distilled in a column to give a top product of 0.825 mole fraction alcohol and a liquor at the bottom containing 0.05 mole fraction alcohol. How many theoretical plates are required if the reflux ratio used is 3? Indicate on a diagram what is meant by the Murphree plate efficiency.

Solution

This example is solved by a simple application of the McCabe–Thiele method and is illustrated in Figure 11n, where it is seen that 10 theoretical plates are required. The Murphree plate efficiency is discussed in the solution to Problem 11.9.
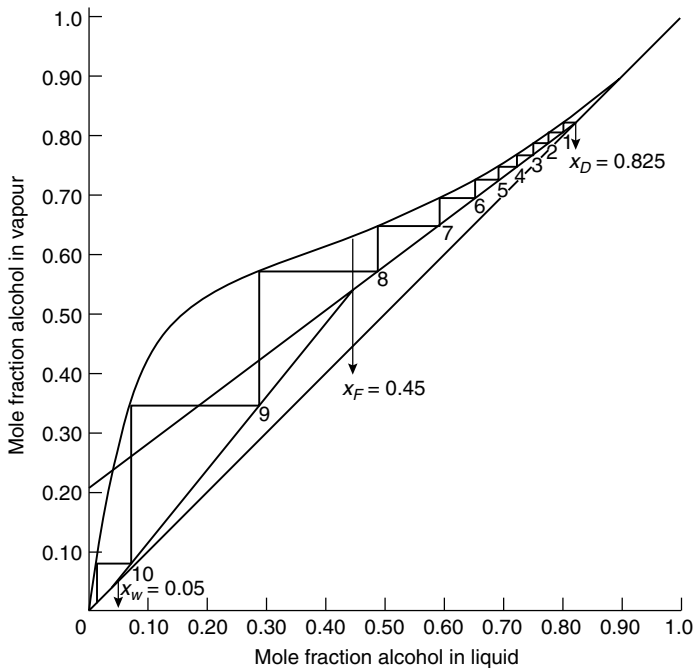
Figure 11n. Graphical construction for Problem 11.17

PROBLEM 11.18

It is desired to separate 1 kg/s of an ammonia solution containing 30 per cent NH_3 by mass into 99.5 per cent liquid NH_3 and a residual weak solution containing 10 per cent NH_3. Assuming the feed to be at its boiling point, a column pressure of 1013 kN/m^2, a plate efficiency of 60 per cent and that an 8 per cent excess over the minimum reflux requirements is used, how many plates must be used in the column and how much heat is removed in the condenser and added in the boiler?

Solution

Taking a material balance for the whole throughput and for the ammonia gives:

$$D' + W' = 1.0$$

and: $$(0.995D' + 0.1W') = (1.0 \times 0.3)$$

Thus: $$D' = 0.22 \text{ kg/s} \quad \text{and} \quad W' = 0.78 \text{ kg/s}$$

The enthalpy-composition chart for this system is shown in Figure 11o. It is assumed that the feed F and the bottom product W are liquids at their boiling-points.

Location of the poles N *and* M
N_m for minimum reflux is found by drawing a tie line through F, representing the feed, to cut the line $x = 0.995$ at N_m.
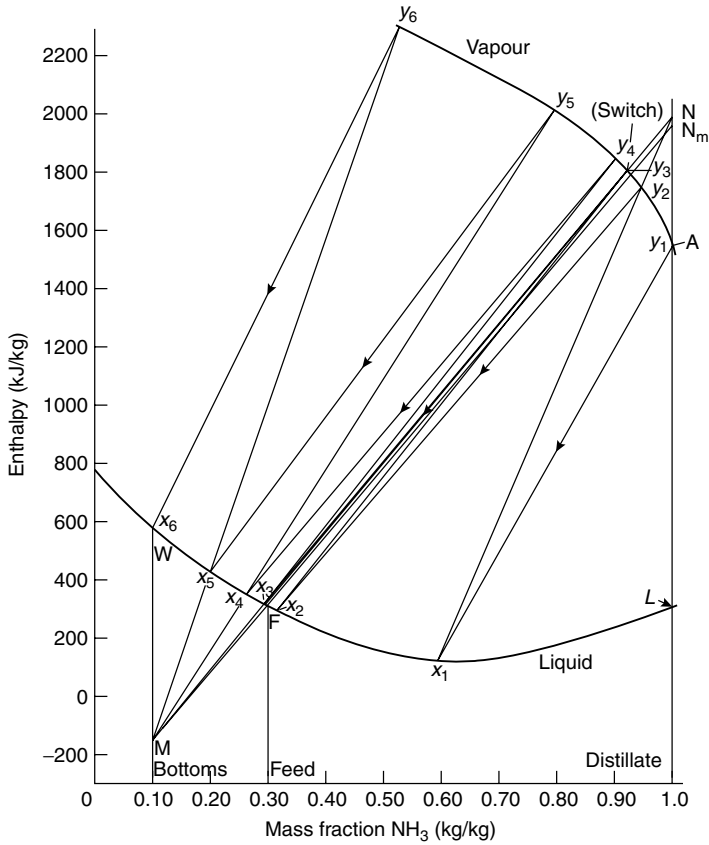
Figure 11o. Graphical construction for Problem 11.18

The minimum reflux ratio is given by:

$$R_m = \frac{\text{length } N_m A}{\text{length } AL} = \frac{(1952 - 1547)}{(1547 - 295)} = 0.323$$

Since the actual reflux is 8 per cent above the minimum,

$$NA = 1.08 N_m A = (1.08 \times 405) = 437$$

Point N therefore has an ordinate $(437 + 1547) = 1984$ and abscissa 0.995.

Point M is found by drawing NF to cut the line $x = 0.10$, through W, at M.

The number of theoretical plates is found, as on the diagram, to be 5+.

The number of plates to be provided $= (5/0.6) = 8.33$, say 9.

The feed is introduced just below the third ideal plate from the top, or just below the fifth actual plate.

The heat input at the boiler per unit mass of bottom product is given by:

$$Q_B/W = 582 - (-209) = 791 \text{ kJ/kg (from equation 11.88)}.$$

The heat input to boiler $= (791 \times 0.78) = \underline{617 \text{ kW}}$.

The condenser duty $=$ length NL $\times D$

$$= (1984 - 296) \times 0.22 = \underline{\underline{372 \text{ kW}}}.$$

PROBLEM 11.19

A mixture of 60 mole per cent benzene, 30 per cent of toluene and 10 per cent xylene is handled in a batch still. If the top product is to be 99 per cent benzene, determine:

(a) the liquid composition on each plate at total reflux,
(b) the composition on the 2nd and 4th plates for $R = 1.5$,
(c) as for (b) but $R = 5$,
(d) as for (c) but $R = 8$ and for the condition when the mol per cent benzene in the still is 10,
(e) as for (d) but with $R = 5$.

The relative volatility of benzene to toluene may be taken as 2.4, and the relative volatility of xylene to toluene as 0.43.

Solution

Although this problem is one of multicomponent batch distillation, the product remains of constant composition so that normal methods can be used for plate-to-plate calculations at a point value of the varying reflux ratio.

(a) At total reflux, for each component the operating line is:

$$y_n = x_{n+1}$$

Also:

$$y = \alpha x \Big/ \sum (\alpha x)$$

The solution is given in tabular form as:

	α	x_s	αx_s	$y_s = x_1$	αx_1	$y_1 = x_2$	αx_2	$y_2 = x_3$	Similarly x_4	x_5
B	2.4	0.60	1.44	0.808	1.164	0.867	2.081	0.942	0.975	0.989
T	1.0	0.30	0.30	0.168	0.168	0.125	0.125	0.057	0.025	0.011
X	0.43	0.10	0.043	0.024	0.010	0.008	0.003	0.001	—	—
			1.783	$\underline{\underline{1.000}}$	1.342	$\underline{\underline{1.000}}$	2.209	$\underline{\underline{1.000}}$	$\underline{\underline{1.000}}$	$\underline{\underline{1.000}}$

(b) The operating line for the rectifying section is:

$$y_n = \frac{L_n}{V_n} x_{n+1} + \frac{D}{V_n} x_d$$

$$R = L_n/D \quad \text{and} \quad V = L_n + D$$

Thus:

$$y_n = \frac{R}{R+1} x_{n+1} + \frac{x_d}{R+1}$$

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If $R = 1.5$:   for benzene, $y_{nb} = 0.6x_{n+1} + 0.396$,

toluene, $y_{nt} = 0.6x_{n+1} + 0.004$,   and   xylene, $y_{nx} = 0.6x_{n+1}$

The liquid composition on each plate is then found from these operating lines.

	y_s	x_1	αx_1	y_1	x_2	αx_2	y_2	x_3	αx_3	y_3	x_4
B	0.808	0.687	1.649	0.850	0.757	1.817	0.848	0.754	1.810	0.899	0.838
T	0.168	0.273	0.273	0.141	0.228	0.228	0.107	0.171	0.171	0.085	0.135
X	0.024	0.040	0.017	0.009	0.015	0.098	0.045	0.075	0.032	0.016	0.027
	1.000	1.000	1.939	1.000	1.000	2.143	1.000	1.000	2.013	1.000	1.000

(c) If $R = 5$, the operating line equations become:

$$y_{nb} = 0.833x_{n+1} + 0.165$$

$$y_{nt} = 0.833x_{n+1} + 0.0017$$

$$y_{nx} = 0.833x_{n+1}$$

	y_s	x_1	αx_1	y_1	x_2	αx_2	y_2	x_3	αx_3	y_3	x_4
B	0.808	0.772	1.853	0.897	0.879	2.110	0.947	0.939	2.254	0.974	0.971
T	0.168	0.200	0.200	0.097	0.114	0.114	0.051	0.059	0.059	0.025	0.028
X	0.024	0.028	0.012	0.006	0.007	0.003	0.002	0.002	0.001	0.001	0.001
	1.000	1.000	2.065	1.000	1.000	2.227	1.000	1.000	2.314	1.000	1.000

(d) When the benzene content is 10 per cent in the still, a mass balance gives the kmol of distillate removed, assuming 100 kmol initially, as:

$$D = 100(0.6 - 0.1)/(0.99 - 0.1) = 56.2 \text{ kmol}$$

Thus 43.8 kmol remain of which 4.38 are benzene, $x_b = 0.10$

29.42 are toluene, $x_t = 0.67$

and: 10.00 are xylene, $x_x = 0.23$

If $R = 8$, the operating lines become:

$$y_{nb} = 0.889x_{n+1} + 0.11, \quad y_{nt} = 0.889x_{n+1} + 0.001 \quad \text{and} \quad y_{nx} = 0.889x_{n+1}$$

	x_s	αx_s	y_s	x_1	αx_1	y_1	x_2	αx_2	y_2	x_3	αx_3	y_3	x_4
B	0.10	0.24	0.24	0.146	0.350	0.307	0.222	0.533	0.415	0.343	0.823	0.560	0.506
T	0.67	0.67	0.66	0.741	0.741	0.650	0.730	0.730	0.569	0.639	0.639	0.435	0.488
X	0.23	0.10	0.10	0.113	0.049	0.043	0.048	0.021	0.016	0.018	0.008	0.005	0.006
		1.01	1.00	1.000	1.140	1.000	1.000	1.284	1.000	1.000	1.470	1.000	1.000

(e) Exactly the same procedure is repeated for this part of the question, when the operating lines become:

$$y_{nb} = 0.833x_{n+1} + 0.165, \quad y_{nt} = 0.833x_{n+1} + 0.0017 \quad \text{and} \quad y_{nx} = 0.833x_{n+1}$$

PROBLEM 11.20

A continuous still is fed with a mixture of 0.5 mole fraction of the more volatile component, and gives a top product of 0.9 mole fraction of the more volatile component and a bottom product containing 0.10 mole fraction.

If the still operates with an L_n/D ratio of 3.5:1, calculate by Sorel's method the composition of the liquid on the third theoretical plate from the top:

(a) for benzene–toluene, and
(b) for n-heptane–toluene.

Solution

A series of mass balances as described in other problems enables the flows within the column to be calculated as follows.

For a basis of 100 kmol of feed and a reflux ratio of 3.5:

$$D = 50, \quad L_n = 175, \quad V_n = 225 \text{ kmol}$$

The top operating line equation is then:

$$y_n = 0.778x_{n+1} + 0.20$$

(a) Use is made of the equilibrium data from other examples involving benzene and toluene.

The vapour leaving the top plate has the same composition as the top product, or $y_t = 0.9$. From the equilibrium data, $x_t = 0.78$.

Thus: $\qquad y_{t-1} = (0.778 \times 0.78) + 0.20 = 0.807$

$$\text{and } x_{t-1}, \text{ from equilibrium data} = 0.640$$

Similarly: $\qquad y_{t-2} = (0.778 \times 0.640) + 0.20 = 0.698 \quad \text{and} \quad x_{t-2} = 0.49$

$$y_{t-3} = (0.778 \times 0.490) + 0.20 = 0.581 \quad \text{and} \quad x_{t-3} = \underline{\underline{0.36}}$$

(b) Vapour pressure data from Perry[1] for n-heptane and toluene are plotted in Figure 11p. These data may be used to calculate an mean value of the relative volatility α from:

$$\alpha = P_H^0 / P_T^0$$

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[1] PERRY, R. H., GREEN, D. W. and MALONEY, J. O.: *Perry's Chemical Engineers' Handbook*, 6th edn. (McGraw-Hill, New York, 1987).
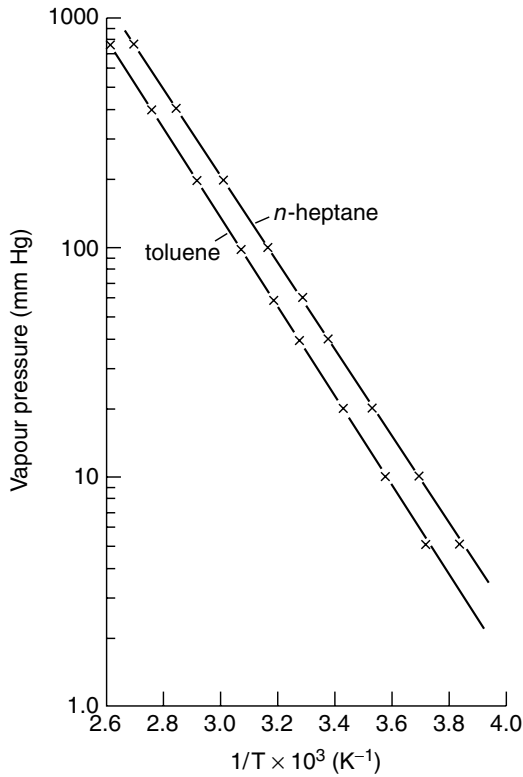
Figure 11p. Vapour pressure data for Problem 11.20

T	$10^3/T$	P_H^0	P_T^0	α	
370	2.7	730	510	1.43	
333	3.0	200	135	1.48	
303	3.3	55	35	1.57	Mean $\alpha = 1.52$
278	3.6	15	9.4	1.60	

As an alternative to drawing the equilibrium curve for the system, point values may be calculated from:

$$x = \frac{y}{\alpha - (\alpha - 1)y} \qquad \text{(equation 11.16)}$$

Thus: $y_t = 0.9$ and $x_t = 0.9/(1.52 - 0.52 \times 0.9) = 0.856$

From the same operating line equation:

$$y_{t-1} = (0.778 \times 0.856) + 0.20 = 0.865$$

Similarly: $x_{t-1} = 0.808$, $y_{t-2} = 0.829$, $x_{t-2} = 0.761$,

$$y_{t-3} = 0.792 \quad \text{and} \quad \underline{\underline{x_{t-3} = 0.715}}$$

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PROBLEM 11.21

A mixture of 40 mole per cent benzene with toluene is distilled in a column to give a product of 95 mole per cent benzene and a waste of 5 mole per cent benzene, using a reflux ratio of 4.

(a) Calculate by Sorel's method the composition on the second plate from the top.
(b) Using the McCabe and Thiele method, determine the number of plates required and the position of the feed if supplied to the column as liquid at the boiling-point.
(c) Find the minimum reflux ratio possible.
(d) Find the minimum number of plates.
(e) If the feed is passed in at 288 K find the number of plates required using the same reflux ratio.

Solution

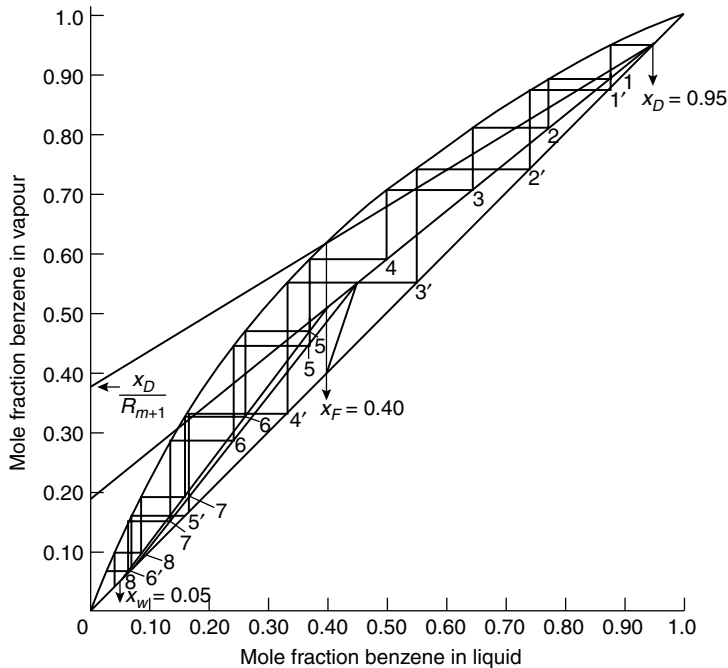The equilibrium data for benzene and toluene are plotted in Figure 11q.

Figure 11q. Equilibrium data for Problem 11.21

$$x_f = 0.40, \quad x_d = 0.95, \quad x_w = 0.05, \quad \text{and} \quad R = 4.0$$

and mass balances may be carried out to calculate the operating line equations. Taking a basis of 100 kmol, then:

$$100 = D + W$$

and:
$$(100 \times 0.4) = 0.95D + 0.05W$$

from which:
$$D = 38.9 \quad \text{and} \quad W = 61.1 \text{ kmol}$$
$$L_n/D = 4 \quad \text{so that} \quad L_n = 155.6 \text{ kmol}$$
$$V_n = L_n + D = 194.5 \text{ kmol}$$

(a) The top operating line equation is:

$$y_n = \frac{L_n}{V_n}x_{n+1} + \frac{D}{V_n}x_d \qquad \text{(equation 11.35)}$$

or:
$$y_n = (155.6/194.5)x_{n+1} + (38.9/194.5) \times 0.95$$
$$y_n = 0.8x_{n+1} + 0.19$$

Vapour y_t leaving the top tray has a composition, $x_d = 0.95$ so that x_t, the liquid composition on the top tray, is found from the equilibrium curve to be equal to 0.88. y_{t-1} is found from the operating line equation as:

$$y_{t-1} = (0.8 \times 0.88) + 0.19 = 0.894$$
$$x_{t-1} = 0.775, \text{ from the equilibrium curve.}$$

Thus:
$$y_{t-2} = (0.8 \times 0.775) + 0.19 = 0.810$$
$$x_{t-2} = \underline{\underline{0.645}}, \text{ from the equilibrium curve.}$$

(b) The steps in the McCabe–Thiele determination are shown in Figure 11q where 8 theoretical plates are required with a boiling liquid feed.

(c) The minimum reflux ratio corresponds to an infinite number of plates. This condition occurs when the top operating line passes through the intersection of the q-line and the equilibrium curve. This line is seen to give an intersection on the y-axis equal to 0.375.

Thus:
$$0.375 = x_d/(R_M + 1) \quad \text{and} \quad \underline{\underline{R_M = 1.53}}$$

(d) The minimum number of plates occurs at total reflux and may be determined by stepping between the equilibrium curve and the diagonal $y = x$ to give 6 theoretical plates as shown.

Alternatively, Fenske's equation may be used.

Thus:
$$n + 1 = \log\frac{[(x_A/x_B)_d(x_B/x_A)_s]}{\log \alpha_{AB}} \qquad \text{(equation 11.58)}$$
$$= \frac{\log(0.95/0.05)(0.95/0.05)}{\log 2.4} \quad \text{and} \quad n = 5.7 \text{ or } \underline{\underline{6 \text{ plates}}}$$

(e) If a cold feed is introduced, the q-line is no longer vertical. The slope of the line may be calculated as shown in Problem 11.16. In this problem, q is found to be 1.45 and the q-line has a slope of 3.22. This line is shown in Figure 11q and the number of theoretical plates is found to be unchanged at $\underline{\underline{8}}$.

PROBLEM 11.22

Determine the minimum reflux ratio using Fenske's equation and Colburn's rigorous method for the following three systems:

(a) 0.60 mole fraction C_6, 0.30 mole fraction C_7, and 0.10 mole fraction C_8 to give a product of 0.99 mole fraction C_6.

		Mole fraction	Relative volatility, α	x_d
(b) Components	A	0.3	2	1.0
	B	0.3	1	—
	C	0.4	0.5	—
(c) Components	A	0.25	2	1.0
	B	0.25	1	—
	C	0.25	0.5	—
	D	0.25	0.25	—

Solution

(a) Under conditions where the relative volatility remains constant, Underwood developed the following equations from which R_m may be calculated:

$$\frac{\alpha_A x_{fA}}{\alpha_A - \theta} + \frac{\alpha_B x_{fB}}{\alpha_B - \theta} + \frac{\alpha_C x_{fC}}{\alpha_C - \theta} + \cdots = 1 - q \qquad \text{(equation 11.114)}$$

and:

$$\frac{\alpha_A x_{dA}}{\alpha_A - \theta} + \frac{\alpha_B x_{dB}}{\alpha_B - \theta} + \frac{\alpha_C x_{dC}}{\alpha_C - \theta} + \cdots = R_m + 1 \qquad \text{(equation 11.115)}$$

where $x_{fA}, x_{fB}, x_{dA}, x_{dB}$, and so on, are the mole fractions of components **A** and **B**, and so on, in the feed and distillate with **A** the light key and **B** the heavy key.

$\alpha_A, \alpha_B, \alpha_C$, etc., are the volatilities with respect to the least volatile component.

θ is the root of equation 11.114 and lies between the values of α_A and α_B. Thus θ may be calculated from equation 11.114 and substituted into 11.115 to give R_m.

(b) Colburn's method allows the value of R_m to be calculated from approximate values of the pinch compositions of the key components. This value may then be checked again empirical relationships as shown in Example 11.15 in Volume 2.

The method is long and tedious and only the first approximation will be worked here.

$$R_m = \frac{1}{\alpha_{AB} - 1} \left[\left(\frac{x_{dA}}{x_{nA}} \right) - \alpha_{AB} \left(\frac{x_{dB}}{x_{nB}} \right) \right] \qquad \text{(equation 11.108)}$$

where x_{dA} and x_{nA} are the top and pinch compositions of the light key component, x_{dB} and x_{nB} are the top and pinch compositions of the heavy key component, and α_{AB} is the volatility of the light key relative to the heavy key component.

The difficulty in using this equation is that the values of x_{nA} and x_{nB} are known only for special cases where the pinch coincides with the feed composition. Colburn has suggested that an approximate value for x_{nA} is given by:

$$x_{nA} \text{ (approx.)} = \frac{r_f}{(1 + r_f)(1 + \Sigma \alpha x_{fh})} \qquad \text{(equation 11.109)}$$

and: $$x_{nB} \text{ (approx.)} = \frac{x_{nA}}{r_f} \qquad \text{(equation 11.110)}$$

where r_f is the estimated ratio of the key components on the feed plate.

For an all liquid feed at its boiling-point, r_f is equal to the ratio of the key components in the feed. Otherwise r_f is the ratio of the key components in the liquid part of the feed, x_{fh} is the mole fraction of each component in the liquid portion of feed heavier than the heavy key, and α is the volatility of the component relative to the heavy key.

(a) Relative volatility data are required and it will be assumed that $\alpha_{6,8} = 5$, $\alpha_{6,7} = 2.5$, and also that $x_{d7} = 0.01$ and that $q = 1$.

Substituting in Underwood's equation gives:

$$\left(\frac{5 \times 0.60}{5 - \theta}\right) + \left(\frac{2.5 \times 0.30}{2.5 - \theta}\right) + \left(\frac{0.1 \times 1}{1 - \theta}\right) = 1 - q = 0$$

from which by trial and error, $\theta = 3.1$.

Then: $$\left(\frac{5 \times 0.99}{5 - 3.1}\right) + \left(\frac{2.5 \times 0.01}{2.5 - 3.1}\right) = R_m + 1 \quad \text{and} \quad \underline{\underline{R_m = 1.57}}$$

In Colburn's equation, using C_6 and C_7 as the light and heavy keys respectively:

$$r_f = (0.6/0.3) = 2.0$$

$$\sum \alpha x_{fh} = (1/2.5) \times 0.1 = 0.04, \quad x_{nA} = 2/(3 \times 1.04) = 0.641$$

$$\alpha_{AB} = (5.0/2.5) = 2.0, \quad x_{nB} = (0.641/2) = 0.32$$

$$R_m = \frac{1}{1}\left[\left(\frac{0.99}{0.641}\right) - \left(\frac{2 \times 0.01}{0.32}\right)\right] \quad \text{and} \quad \underline{\underline{R_m = 1.48}}$$

(b) The light key $= A$, the heavy key $= B$, and $\alpha_A = 4$, $\alpha_B = 2$, $\alpha_C = 1$, and if q is assumed to equal 1, then substitution into Underwood's equations gives $\theta = 2.8$ and $\underline{\underline{R_m = 2.33}}$.

In Colburn's method, $x_{dB} = 0$ and $r_f = (0.3/0.3) = 1.0$.

$$\sum \alpha n_{fh} = (1/2) \times 0.4 = 0.2$$

$$x_{nA} = 1/(2 \times 1.2) = 0.417$$

$$\alpha_{AB} = (4/2) = 2.0 \quad \text{and} \quad \underline{\underline{R_m = 2.40}}$$

(c) In this case, $\alpha_A = 8$, $\alpha_B = 4$, $\alpha_C = 2$, $\alpha_D = 1$. If $q = 1$, then θ is found from Underwood's equation to be equal to 5.6 and $\underline{\underline{R_m = 2.33}}$.

In Colburn's method, $x_{dB} = 0$ and $r_f = 1.0$, the light key $= A$, the heavy key $= B$.

$$\sum \alpha x_{fh} = (0.5 \times 0.25) + (0.25 \times 0.25) = 0.188$$

$$x_{nA} = 1/(2 \times 1.188) = 0.421$$

$$\alpha_{AB} = 2 \quad \text{and} \quad \underline{\underline{R_m = 2.38}}$$

In all cases, good agreement is shown between the two methods.

PROBLEM 11.23

A liquor consisting of phenol and cresols with some xylenols is fractionated to give a top product of 95.3 mole per cent phenol. The compositions of the top product and of the phenol in the bottoms are:

	Compositions (mole per cent)		
	Feed	Top	Bottom
phenol	35	95.3	5.24
o-cresol	15	4.55	—
m-cresol	30	0.15	—
xylenols	20	—	—
	100	100	

If a reflux ratio of 10 is used,

(a) Complete the material balance over the still for a feed of 100 kmol.
(b) Calculate the composition on the second plate from the top.
(c) Calculate the composition on the second plate from the bottom.
(d) Calculate the minimum reflux ratio by Underwood's equation and by Colburn's approximation.

The heavy key is m-cresol and the light key is phenol.

Solution

(a) An overall mass balance and a phenol balance gives, on a basis of 100 kmol:

$$100 = D + W$$

and:
$$(100 \times 0.35) = 0.953D + 0.0524W$$

from which:
$$D = 33.0 \text{ kmol} \quad \text{and} \quad W = 67.0 \text{ kmol.}$$

Balances on the remaining components give the required bottom product composition as:

o-cresol: $(100 \times 0.15) = (0.0455 \times 33) + 67x_{wo}$ and $x_{wo} = \underline{\underline{0.2017}}$

m-cresol: $(100 \times 0.30) = (0.0015 \times 33) + 67x_{wm}$ and $x_{wm} = \underline{\underline{0.4472}}$

xylenols: $(100 \times 0.20) = 0 + 67x_{wx}$, and $x_{wx} = \underline{\underline{0.2987}}$

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(b)
$$L_n/D = 10 \quad \text{and} \quad L_n = 330 \text{ kmol}$$
$$V_n = L_n + D \quad \text{and} \quad V_n = 363 \text{ kmol}$$

The equation of the top operating line is:

$$y_n = \frac{L_n}{V_n}x_{n+1} + \frac{D}{V_n}x_d = (330/363)x_{n+1} + (33/330)x_d = 0.91x_{n+1} + 0.091x_d$$

The operating lines for each component then become:

$$\text{phenol:} \quad y_{np} = 0.91x_{n+1} + 0.0867$$
$$\text{o-cresol:} \quad y_{no} = 0.91x_{n+1} + 0.0414$$
$$\text{m-cresol:} \quad y_{nm} = 0.91x_{n+1} + 0.00014$$
$$\text{xylenols:} \quad y_{nx} = 0.91x_{n+1}$$

Mean α-values are taken from the data given in Volume 2, Table 11.2 as:

$$\alpha_{PO} = 1.25, \quad \alpha_{OO} = 1.0, \quad \alpha_{MO} = 0.63, \quad \alpha_{XO} = 0.37$$

The solution may be set out as a table as follows, using the operating line equations and the equation:

$$x = \frac{y/\alpha}{\sum(y/\alpha)}$$

	$y = x_d$	y_t/α	x_t	y_{t-1}	y_{t-1}/α	x_{t-1}
phenol	0.953	0.762	0.941	0.943	0.754	0.928
o-cresol	0.0455	0.0455	0.056	0.054	0.054	0.066
m-cresol	0.0015	0.0024	0.003	0.003	0.005	0.006
xylenols	—	—	—	—	—	—
	$\Sigma(y_t/\alpha) = 0.8099$		1.000	1.000	0.813	1.000

(c) In the bottom of the column:

$$L_m = L_n + F = 430 \text{ kmol}$$
$$V_m = L_m - W = 363 \text{ kmol}$$

and:
$$y_m = \frac{L_m}{V_m}x_{n+1} - \frac{W}{V_m}x_w = 1.185x_{m+1} - 0.185x_w$$

Hence for each component:

$$\text{phenol:} \quad y_{mp} = 1.185x_{m+1} - 0.0097$$
$$\text{o-cresol:} \quad y_{mo} = 1.185x_{m+1} - 0.0373$$
$$\text{m-cresol:} \quad y_{mm} = 1.185x_{m+1} - 0.0827$$
$$\text{xylenols:} \quad y_{mx} = 1.185x_{m+1} - 0.0553$$

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Using these operating lines to calculate x and also $y = \alpha x / \Sigma \alpha x$ gives the following data:

	x_s	αx_s	y_s	x_1	αx_1	y_1	x_2
phenol	0.0524	0.066	0.100	0.093	0.116	0.156	0.140
o-cresol	0.2017	0.202	0.305	0.289	0.289	0.387	0.358
m-cresol	0.4472	0.282	0.427	0.430	0.271	0.363	0.376
xylenols	0.2987	0.111	0.168	0.188	0.070	0.094	0.126
	1.000	$\Sigma = 0.661$	1.000	1.000	$\Sigma = 0.746$	1.000	1.000

(d) Underwood's equations defined in, Problem 11.22, are used with $\alpha_p = 3.4$, $\alpha_o = 2.7$, $\alpha_m = 1.7$, $\alpha_x = 1.0$ to give:

$$\left(\frac{3.4 \times 0.35}{3.4 - \theta}\right) + \left(\frac{2.7 \times 0.15}{2.7 - \theta}\right) + \left(\frac{1.7 \times 0.30}{1.7 - \theta}\right) + \left(\frac{1.0 \times 0.20}{1 - \theta}\right) = (1 - q) = 0$$

$3.4 > \theta > 1.7$ and θ is found by trial and error to be 2.06.

Then: $\left(\dfrac{3.4 \times 0.953}{3.4 - 2.06}\right) + \left(\dfrac{2.7 \times 0.0455}{2.7 - 2.06}\right) + \left(\dfrac{1.7 \times 0.0015}{1.7 - 2.06}\right) = R_{m+1}$

and: $$\underline{\underline{R_{m+1} = 1.60}}$$

Colburn's equation states that:

$$R_m = \frac{1}{\alpha_{AB} - 1}\left[\left(\frac{x_{dA}}{x_{nA}}\right) - \alpha_{AB}\left(\frac{x_{dB}}{x_{nB}}\right)\right] \qquad \text{(equation 11.108)}$$

$$x_{nA} = \frac{r_f}{(1 + r_f)(1 + \Sigma x_{fh})} \qquad \text{(equation 11.109)}$$

$$x_{nB} = x_{nA}/r_f \qquad \text{(equation 11.110)}$$

where **A** and **B** are the light and heavy keys, that is phenol and m-cresol.

$$r_f = (0.35/0.30) = 1.17$$
$$\Sigma \alpha x_{fh} = (0.37/0.63) \times 0.20 = 0.117$$
$$x_{nA} = 1.17/(2.17 \times 1.117) = 0.482$$
$$x_{nB} = (0.482/1.17) = 0.413$$
$$\alpha_{AB} = (1.25/0.63) = 1.98$$

Thus: $R_m = \dfrac{1}{0.98}\left[\left(\dfrac{0.953}{0.482}\right) - \left(\dfrac{1.98 \times 0.0015}{0.413}\right)\right] = \underline{\underline{1.95}}$

This is the first approximation by Colburn's method and provides a good estimate of R_m.

PROBLEM 11.24

A continuous fractionating column is to be designed to separate 2.5 kg/s of a mixture of 60 per cent toluene and 40 per cent benzene, so as to give an overhead of 97 per cent benzene and a bottom product containing 98 per cent toluene by mass. A reflux ratio of 3.5 kmol of reflux/kmol of product is to be used and the molar latent heat of benzene and toluene may be taken as 30 MJ/kmol. Calculate:

(a) The mass flow of top and bottom products.
(b) The number of theoretical plates and position of the feed if the feed is liquid at 295 K, of specific heat capacity 1.84 kJ/kg K.
(c) How much steam at 240 kN/m² is required in the still.
(d) What will be the required diameter of the column if it operates at atmospheric pressure and a vapour velocity of 1 m/s.
(e) If the vapour velocity is to be 0.75 m/s, based on free area of column, determine the necessary diameter of the column.
(f) The minimum possible reflux ratio, and the minimum number of plates for a feed entering at its boiling-point.

Solution

(a) An overall mass balance and a benzene balance permit the mass of product and waste to be calculated directly:

$$2.5 = D' + W'$$

and:

$$2.5 \times 0.4 = 0.97D' + 0.02W'$$

from which:

$$\underline{\underline{W' = 1.5 \text{ kg/s}}} \quad \text{and} \quad \underline{\underline{D' = 1.0 \text{ kg/s}}}$$

(b) This part of the problem is solved by the McCabe–Thiele method. If the given compositions are converted to mole fractions, then:

$$x_f = 0.44, \quad x_w = 0.024, \quad x_d = 0.974$$

and a mass balance gives for 100 kmol of feed:

$$100 = D + W$$

$$(100 \times 0.44) = 0.974D + 0.024W$$

from which $D = 43.8$ and $W = 56.2$ kmol/100 kmol of feed

If $R = 3.5$, then: $L_n/D = 3.5$ and $L_n = 153.3$ kmol

$$V_n = L_n + D \quad \text{and} \quad V_n = 197.1 \text{ kmol}$$

The intercept on the y-axis $= x_d/(R + 1) = 0.216$.

As the feed is a cold liquid, the slope of the q-line must be found. Using the given data and employing the method used in earlier problems, q is found to be 1.41 and the slope $= q/(q + 1) = 3.44$. This enables the diagram to be completed as shown in

Figure 11r from which it is seen that 10 theoretical plates are required with the feed tray as the fifth from the top.
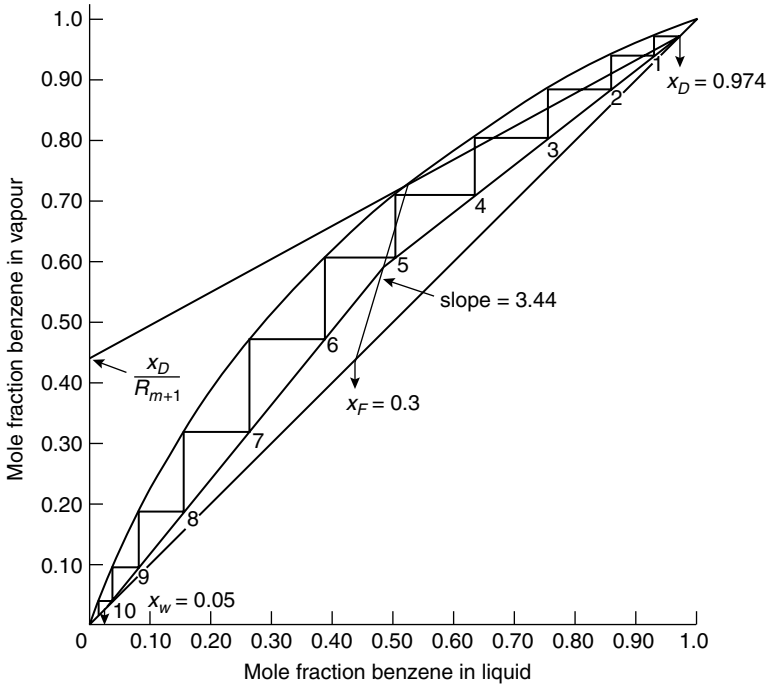

Figure 11r. Equilibrium data for Problem 11.24

(c) The boil-up rate at the bottom of the column $= V_m$.

$$V_m = 238.1 \text{ kmol/100 kmol feed}$$

$$\text{feed rate} = 2.5/(\text{mean mol mass}) = (2.5/86.4) = 0.0289 \text{ kmol/s}$$

Thus: vapour rate $= (238.1/100) \times 0.0289 = 0.069 \text{ kmol/s}$

The heat load $= (0.069 \times 30) = 2.07 \text{ MW or } 2070 \text{ kW}$

The latent heat of steam at $240 \text{ kN/m}^2 = 2186 \text{ kJ/kg}$ (from the Appendix in Volume 2)

Thus: steam required $= (2070/2186) = \underline{\underline{0.95 \text{ kg/s}}}$

(d) At the top of the column the temperature is the boiling-point of essentially pure benzene, that is 353.3 K.

Thus: $C = (1/22.4)(273/353.3) = 0.034 \text{ kmol/m}^3$

and: $V_n = 197.1 \text{ kmol/100 kmol of feed.}$

Vapour flow $= (197.1/100) \times 0.0289 = 0.057 \text{ kmol/s}$

Thus: volumetric flowrate $= (0.057/0.034) = 1.68 \text{ m}^3/\text{s}$

If the vapour velocity is 1.0 m/s, then:

$$\text{the area} = 1.68 \text{ m}^2 \quad \text{and} \quad \text{the diameter} = 1.46 \text{ m}$$

If the diameter is calculated from the velocity at the bottom of the column, the result is a diameter of 1.67 m so that, if the velocity is not to exceed 1 m/s in any part of the column, its diameter must be <u>1.67 m</u>.

(e) The velocity based on the free area (tower area − downcomer area) must not exceed 0.75 m/s. The vapour rate in the bottom of the column is 2.17 m^3/s and, for a single-pass crossflow tray, the free area is approximately 88 per cent of the tower area.

Thus: $\qquad\qquad\qquad A_t = 2.17/(0.75 \times 0.88) = 3.28 \text{ m}^2$

and: $\qquad\qquad\qquad \underline{\underline{D_t = 2.05 \text{ m}}}$

PROBLEM 11.25

For a system that obeys Raoult's law, show that the relative volatility α_{AB} is P_A^0/P_B^0, where P_A^0 and P_B^0 are the vapour pressures of the components **A** and **B** at the given temperature. From vapour pressure curves of benzene, toluene, ethyl benzene and of o-, m- and p-xylenes, obtain a plot of the volatilities of each of the materials relative to m-xylene in the range 340–430 K.

Solution

The volatility of $\mathbf{A} = P_A/x_A$ and the volatility of $\mathbf{B} = P_B/x_B$ and the relative volatility α_{AB} is the ratio of these volatilities,

that is: $\qquad\qquad\qquad \alpha_{AB} = \dfrac{P_A x_B}{x_A P_B}$

For a system that obeys Raoult's law, $P = x P^0$.

Thus: $\qquad\qquad\qquad \alpha_{AB} = \dfrac{(x_A P_A^0) x_B}{x_A (x_B P_B^0)} = \underline{\underline{\dfrac{P_A^0}{P_B^0}}}$

The vapour pressures of the compounds given in the problem are plotted in Figure 11s and are taken from Perry.[1]

For convenience, the vapour pressures are plotted on a logarithmic scale against the reciprocal of the temperature (1/K) given a straight line. The relative volatilities may then be calculated in tabular form as follows:

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[1] PERRY, R. H., GREEN, D. W., and MALONEY, J. O.: *Perry's Chemical Engineer's Handbook*, 6th edn. (McGraw-Hill, New York, 1987).
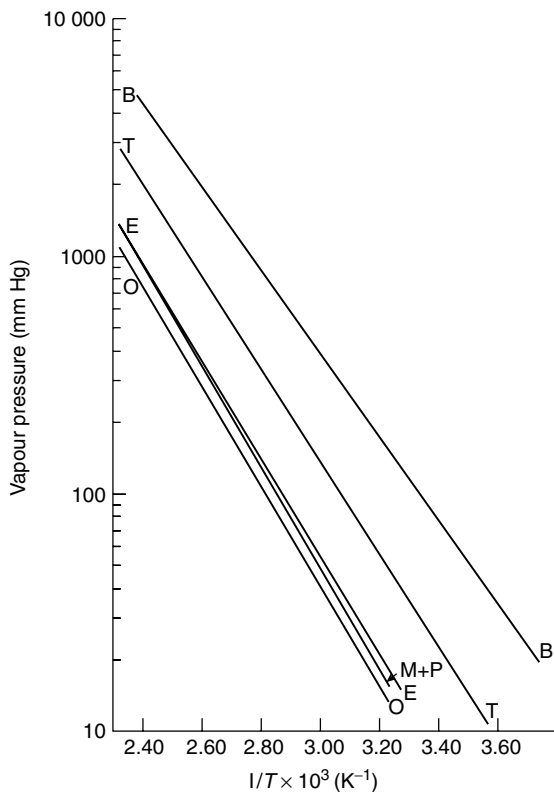
Figure 11s. Vapour pressure data for Problem 11.25

Temperature (K)	$10^3/T$ (1/K)	$(m-x)$	P^0 (mmHg)/ $(p-x)$	$(o-x)$	E	B	T
340	2.94	65	64	56	72	490	180
360	2.78	141	140	118	155	940	360
380	2.63	292	290	240	310	1700	700
400	2.50	554	550	450	570	2900	1250
415	2.41	855	850	700	860	4200	1900
430	2.32	1315	1300	1050	1350	6000	2800

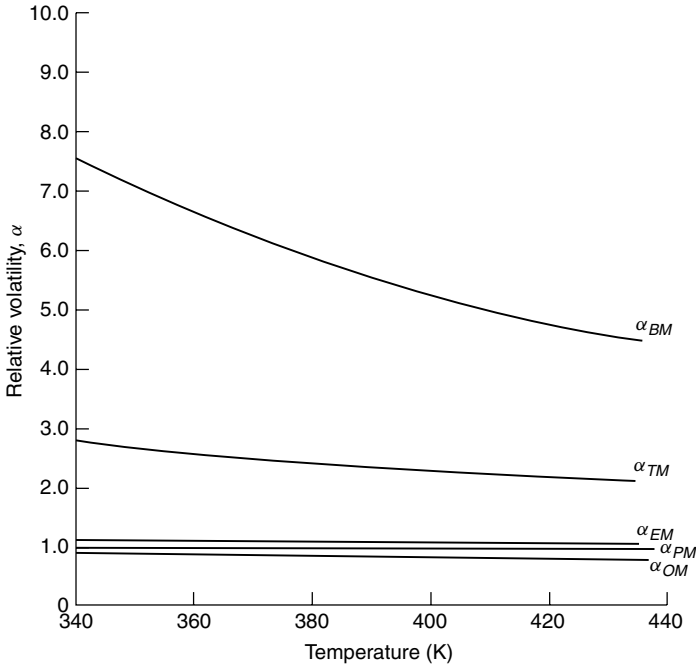| Temperature (K)	α_{pm}	α_{om}	α_{Em}	α_{Bm}	α_{Tm}
340	0.98	0.86	1.11	7.54	2.77
360	0.99	0.84	1.10	6.67	2.55
380	0.99	0.82	1.06	5.82	2.40
400	0.99	0.81	1.03	5.23	2.26
415	0.99	0.82	1.01	4.91	2.22
430	0.99	0.80	1.03	4.56	2.13

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These data are plotted in Figure 11t.

Figure 11t. Relative volatility data for Problem 11.25

PROBLEM 11.26

A still contains a liquor composition of *o*-xylene 10 per cent, *m*-xylene 65 per cent, *p*-xylene 17 per cent, benzene 4 per cent and ethyl benzene 4 per cent. How many plates are required at total reflux to give a product of 80 per cent *m*-xylene, and 14 per cent *p*-xylene? The data are given as mass per cent.

Solution

Fenske's equation may be used to find the number of plates at total reflux.

Thus:
$$n + 1 = \frac{\log[(x_A/x_B)_d(x_B/x_A)_s]}{\log \alpha_{AB}}$$
(equation 11.58)

In multicomponent distillation, **A** and **B** are the light and heavy key components respectively. In this problem, the only data given for both top and bottom products are for *m*- and *p*-xylene and these will be used with the mean relative volatility calculated in the previous problem. Thus:

$$x_A = 0.8, \quad x_B = 0.14, \quad x_{B_s} = 0.17, \quad x_{A_s} = 0.65$$

$$\alpha_{AB} = (1/0.99) = 1.0101$$

Thus: $n + 1 = \log[(0.8/0.14)(0.17/0.65)]/\log 1.0101$ and $\underline{\underline{n = 39 \text{ plates}}}$

PROBLEM 11.27

The vapour pressures of *n*-pentane and of *n*-hexane are:

Pressure (kN/m^2)	1.3	2.6	5.3	8.0	13.3	26.6	53.2	101.3
(mm Hg)	10	20	40	60	100	200	400	760
Temperature (K)								
C_5H_{12}	223.1	233.0	244.0	257.0	260.6	275.1	291.7	309.3
C_6H_{14}	248.2	259.1	270.9	278.6	289.0	304.8	322.8	341.9

The equilibrium data at atmospheric pressure are:

x	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
y	0.21	0.41	0.54	0.66	0.745	0.82	0.875	0.925	0.975

(a) Determine the relative volatility of pentane to hexane at 273, 293 and 313 K.
(b) A mixture containing 0.52 mole fraction pentane is to be distilled continuously to give a top product of 0.95 mole fraction pentane and a bottom of 0.1 mole fraction pentane. Determine the minimum number of plates, that is the number of plates at total reflux, by the graphical McCabe–Thiele method, and analytically by using the relative volatility method.
(c) Using the conditions in (b), determine the liquid composition on the second plate from the top by Lewis's method, if a reflux ratio of 2 is used.
(d) Using the conditions in (b), determine by the McCabe–Thiele method the total number of plates required, and the position of the feed.

It may be assumed that the feed is all liquid at its boiling-point.

Solution

The vapour pressure data and the equilibrium data are plotted in Figures 11u and 11v.

(a) Using:
$$\alpha_{PH} = P_p^0 / P_H^0$$

The following data are obtained:

Temperature (K)	P_p^0	P_H^0	α_{PH}
273	24	6.0	4.0
293	55	16.0	3.44
313	116	36.5	3.18
		Mean =	3.54

(b) The McCabe–Thiele construction is shown in Figure 11v where it is seen that 4 theoretical plates are required at total reflux.

Using Fenske's equation at total reflux:

$$n + 1 = \log[(0.95/0.05)(0.90/0.10)]/\log 3.54 \quad \text{and} \quad n = 3.07$$
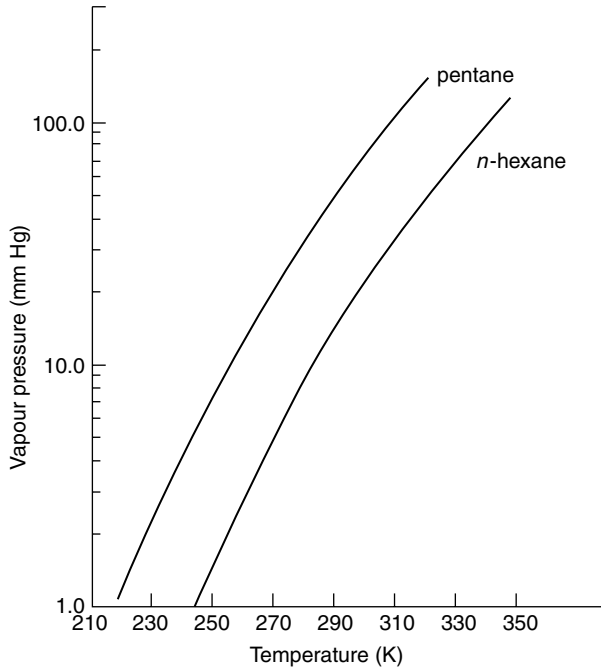
Figure 11u. Vapour pressure data for Problem 11.27

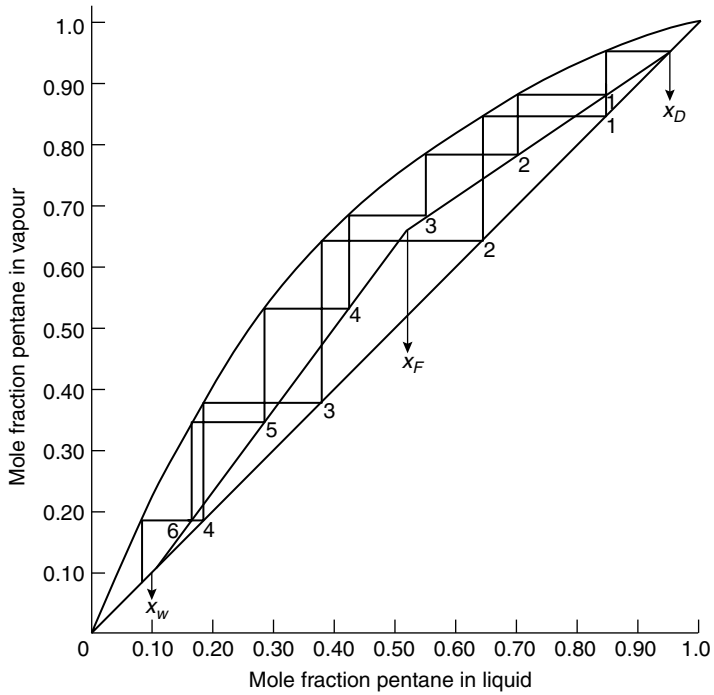**Figure 11v.** Equilibrium data for Problem 11.27

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The discrepancy here is caused by using a mean value of α although α does in fact vary considerably.

(c) From a mass balance it is found that for 100 kmol of feed and $R = 2$:

$$D = 49.4, \quad W = 50.6, \quad L_n = 98.8, \quad V_n = 148.2$$

Then:
$$y_n = \frac{L_n}{V_n}x_{n+1} + \frac{D}{V_n}x_d \qquad \text{(equation 11.34)}$$

and:
$$y_n = 0.67x_{n+1} + 0.317$$

The vapour leaving the top plate has the composition of the distillate, that is: $y_t = x_d = 0.95$.

The liquid on the top plate is in equilibrium with this vapour and from the equilibrium curve has a composition $x_t = 0.845$.

The vapour rising to the top tray y_{t-1} is found from the operating line:

$$y_{t-1} = 0.67 \times 0.845 + 0.317 = 0.883$$

$$x_{t-1} = \text{from the equilibrium curve} = 0.707$$

$$y_{t-2} = (0.67 \times 0.707) + 0.317 = 0.790$$

and:
$$x_{t-2} = \underline{\underline{0.56}}$$

(d) From Figure 11v, <u>6 theoretical plates</u> are required and the feed tray is the <u>third from</u> <u>the top of the column.</u>

PROBLEM 11.28

The vapour pressures of n-pentane and n-hexane are as given in Problem 11.27. Assuming that both Raoult's and Dalton's Laws are obeyed,

(a) Plot the equilibrium curve for a total pressure of 13.3 kN/m^2.
(b) Determine the relative volatility of pentane to hexane as a function of liquid composition for a total pressure of 13.3 kN/m^2.
(c) Would the error caused by assuming the relative volatility constant at its mean value be considerable?
(d) Would it be more advantageous to distil this mixture at a higher pressure?

Solution

(a) The following equations are used where **A** is n-pentane and **B** is n-hexane:

$$x_A = \frac{P - P_B^0}{P_A^0 - P_B^0} \qquad \text{(equation 11.5)}$$

$$y_A = P_A^0 x_A / P \qquad \text{(equation 11.4)}$$

At $P = 13.3 \text{ kN/m}^2$:

Temperature (K)	P_A^0	P_B^0	x_A	y_A	$\alpha = P_A^0/P_B^0$
260.6	13.3	2.85	1.0	1.0	4.67
265	16.5	3.6	0.752	0.933	4.58
270	21.0	5.0	0.519	0.819	4.20
275	26.0	6.7	0.342	0.669	3.88
280	32.5	8.9	0.186	0.455	3.65
285	40.0	11.0	0.079	0.238	3.64
289	47.0	13.3	0	0	3.53
				Mean $\alpha =$	4.02

These figures are plotted in Figure 11w.

(b) The relative volatility is plotted as a function of liquid composition in Figure 11w.

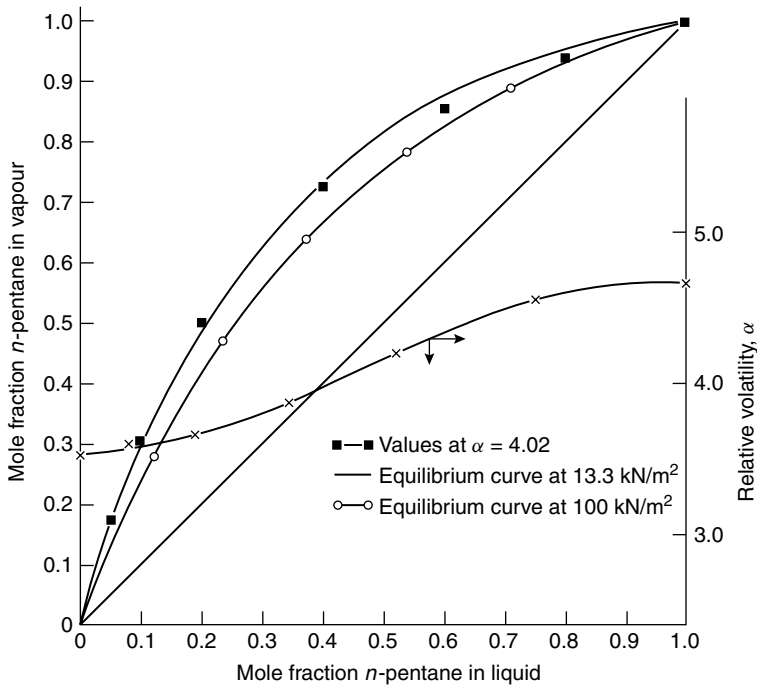**Figure 11w.** Equilibrium data for Problem 11.28

(c) If α is taken as 4.02, y_A may be calculated from:

$$y_A = \frac{\alpha x_A}{1 + (\alpha - 1)x_A} \qquad \text{(equation 11.15)}$$

Using equation 11.15, a new equilibrium curve may be calculated as follows:

x_A	0	0.05	0.10	0.20	0.40	0.60	0.80	1.0
y_A	0	0.174	0.308	0.500	0.727	0.857	0.941	1.0

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These points are shown in Figure 11w where it may be seen that little error is introduced by the use of this mean value.

(d) If a higher pressure is used, the method used in (a) may be repeated. If $P = 100$ kN/m^2, the temperature range increases to 309–341 K and the new curve is drawn in Figure 11w. Clearly, the higher pressure demands a larger number of plates for the same separation and is not desirable.

PROBLEM 11.29

It is desired to separate a binary mixture by simple distillation. If the feed mixture has a composition of 0.5 mole fraction, calculate the fraction which it is necessary to vaporise in order to obtain:

(a) a product of composition 0.75 mole fraction, when using a continuous process, and
(b) a product whose composition is not less than 0.75 mole fraction at any instant, when using a batch process.

If the product of batch distillation is all collected in a single receiver, what is its mean composition?

It may be assumed that the equilibrium curve is given by:

$$y = 1.2x + 0.3$$

for liquid compositions in the range 0.3–0.8.

Solution

(a) If F = number of kmol of feed of composition x_f,
$\quad$ L = kmol remaining in still with composition x, and
$\quad$ V = kmol of vapour formed with composition y, then:

$$F = V + L \quad \text{and} \quad Fx_f = Vy + Lx$$

For 1 kmol of feed:

$$x_f = Vy + Lx \quad \text{and} \quad y = \frac{x_f}{V} - \frac{L}{V}x$$

This equation is a straight line of slope $-L/V$ which passes through the point (x_f, x_f), so that, if y is known, L/V may be found. This is illustrated in Figure 11x where:

$$-L/V = -5.0$$

As: $\qquad F = 1, \quad 1 = V + L$

and: $\qquad V = 0.167$ kmol/kmol of feed or <u>16.7 per cent is vaporised</u>

(b) For a batch process it may be shown that:

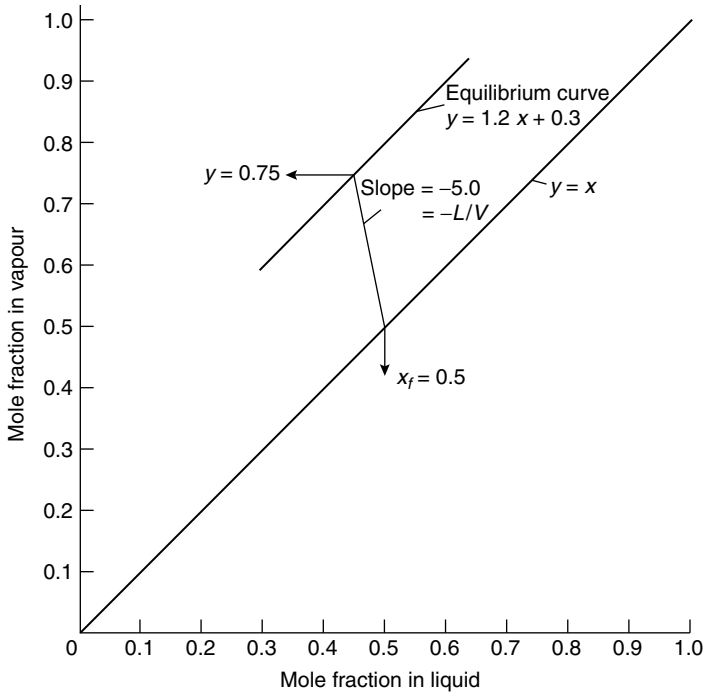$$\left(\frac{y - x}{y_0 - x_0}\right) = \left(\frac{S}{S_0}\right)^{m-1} \qquad \text{(equation 11.29)}$$

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Figure 11x. Graphical construction for Problem 11.29

where S is the number of kmol charged initially $= 100$ kmol (say), S_0 is the number of kmol remaining, x is the initial still composition $= 0.5$, y is the initial vapour composition $= (1.2 \times 0.5) + 0.3 = 0.90$, y_0 is the final vapour composition $= 0.75$ and x_0 is the final liquid composition, is found from:

$$0.75 = 1.2x_0 + 0.3 \quad \text{or} \quad x_0 = 0.375$$

and m is the slope of equilibrium curve $= 1.2$.

Thus: $$\frac{(0.90 - 0.50)}{(0.75 - 0.375)} = 1.07 = \left(\frac{100}{S_0}\right)^{0.2}$$

and: $$S_0 = 71.3 \text{ kmol/100 kmol feed}$$

Therefore: amount vaporised $= \left(\dfrac{100 - 71.3}{100}\right) \times 100 = \underline{\underline{28.7 \text{ per cent}}}$

The distillate composition may be found from a mass balance as follows:

	Total kmol	kmol **A**	kmol **B**
Charge	100	50	50
Distillate	28.7	$(50 - 26.7) = 23.3$	$(50 - 44.6) = 5.4$
Residue	71.3	$(0.374 \times 71.3) = 26.7$	$(71.3 - 26.7) = 44.6$

Distillate composition $= (23.3/28.7) \times 100 = \underline{\underline{81.2 \text{ per cent}}}$.

PROBLEM 11.30

A liquor, consisting of phenol and cresols with some xylenol, is separated in a plate column. Given the following data complete the material balance:

Component	Mole per cent		
	Feed	Top	Bottom
C_6H_5OH	35	95.3	5.24
o-C_7H_7OH	15	4.55	—
m-C_7H_7OH	30	0.15	—
C_8H_9OH	20	—	—
Total	100	100	—

Calculate:

    (a) the composition on the second plate from the top,
    (b) the composition on the second plate from the bottom.

A reflux ratio of 4 is used.

Solution

The mass balance is completed as in Problem 11.24, where it was shown that:

$$x_{wo} = \underline{\underline{0.2017}}, \quad x_{wm} = \underline{\underline{0.4472}}, \quad x_{wx} = \underline{\underline{0.2987}}$$

(a) For 100 kmol feed, from mass balances with $R = 4.0$, the following values are obtained:

$$L_n = 132, \quad V_n = 165, \quad L_n = 232, \quad V_n = 165$$

The equations for the operating lines of each component are obtained from:

$$y_n = \frac{L_n}{V_n}x_{n+1} + \frac{D}{V_n}x_d \qquad \text{(equation 11.35)}$$

as: phenols: $y_{np} = 0.8x_{n+1} + 0.191$

                 o-cresol: $y_{no} = 0.8x_{n+1} + 0.009$

               m-cresol: $y_{nm} = 0.8x_{n+1} + 0.0003$

               xylenols: $y_{nx} = 0.8x_{n+1}$

The compositions on each plate may then be found by calculating y from the operating line equations and x from $x = \dfrac{y/\alpha}{\Sigma(y/\alpha)}$ to give the following results:

	α	$y_t = x_d$	y_t/α	$x_t = (y_t/\alpha)/0.81$	y_{t-1}	y_{t-1}/α	x_{t-1}
phenols	1.25	0.953	0.762	0.940	0.943	0.762	0.843
o-cresol	1.0	0.0455	0.046	0.057	0.055	0.055	0.061
m-cresol	0.63	0.0015	0.002	0.003	0.002	0.087	0.096
xylenols	0.37	—	—	—	—	—	—
		1.000	$\Sigma = 0.81$	1.000	1.000	0.904	1.000

(b) In a similar way, the following operating lines may be derived for the bottom of the column:

$$\text{phenols:} \quad y_{mp} = 1.406x_{n+1} - 0.0212$$

$$\text{o-cresol:} \quad y_{mo} = 1.406x_{n+1} - 0.0819$$

$$\text{m-cresol:} \quad y_{mm} = 1.406x_{n+1} - 0.1816$$

$$\text{xylenols:} \quad y_{mx} = 1.406x_{n+1} - 0.1213$$

Thus:

	α	x_s	αx_s	$y_s = \alpha x_s/0.661$	x_1	αx_1	y_1	x_2
phenols	1.25	0.0524	0.066	0.100	0.086	0.108	0.148	0.121
o-cresol	1	0.2017	0.202	0.305	0.275	0.275	0.375	0.324
m-cresol	0.63	0.4472	0.282	0.427	0.433	0.273	0.373	0.394
xylenols	0.37	0.2987	0.111	0.168	0.206	0.076	0.104	0.161
			$\Sigma = 0.661$	1.000	1.000	0.732	1.000	1.000

PROBLEM 11.31

A mixture of 60, 30, and 10 mole per cent benzene, toluene, and xylene respectively is separated by a plate-column to give a top product containing at least 90 mole per cent benzene and a negligible amount of xylene, and a waste containing not more than 60 mole per cent toluene.

Using a reflux ratio of 4, and assuming that the feed is boiling liquid, determine the number of plates required in the column, and the approximate position of the feed.

The relative volatility of benzene to toluene is 2.4 and of xylene to toluene is 0.45, and it may be assumed that these values are constant throughout the column.

Solution

Assuming 100 kmol of feed, the mass balance may be completed to give:

$$D = 60, \quad W = 40 \text{ kmol}$$

and: $\qquad x_{dt} = 0.10, \quad x_{wb} = 0.15, \quad x_{wx} = 0.25$

If $R = 4$ and the feed is at its boiling-point then:

$$L_n = 240, \quad V_n = 300, \quad L_m = 340, \quad V_m = 300$$

and the top and bottom operating lines are:

$$y_n = 0.8x_{n+1} + 0.2x_d$$

and:
$$y_m = 1.13x_{n+1} - 0.133x_w$$

A plate-to-plate calculation may be carried out as follows:
In the bottom of the column.

α		x_s	αx_s	y_s	x_1	αx_1	y_1	x_2	αx_2	y_2	x_3
2.4	benzene	0.15	0.360	0.336	0.314	0.754	0.549	0.503	1.207	0.724	0.65
1.0	toluene	0.60	0.600	0.559	0.564	0.564	0.411	0.432	0.432	0.258	0.29
0.45	xylene	0.25	0.113	0.105	0.122	0.055	0.040	0.065	0.029	0.018	0.04
			1.073	1.000	1.000	1.373	1.000	1.000	1.668	1.000	1.00

The composition on the <u>third plate from the bottom</u> corresponds most closely to the feed and above this tray the rectifying equations will be used.

	x_3	αx_3	y_3	x_4	αx_4	y_4
benzene	0.657	1.577	0.832	0.815	1.956	0.917
toluene	0.298	0.298	0.157	0.171	0.171	0.080
xylene	0.045	0.020	0.011	0.014	0.006	0.003
	1.000	1.895	1.000	1.000	2.133	1.000

As the vapour leaving the top plate will be totally condensed to give the product, <u>4 theoretical plates</u> will be required to meet the given specification.

PROBLEM 11.32

It is desired to concentrate a mixture of ethyl alcohol and water from 40 mole per cent to 70 mole per cent alcohol. A continuous fractionating column, 1.2 m in diameter with 10 plates is available. It is known that the optimum superficial vapour velocity in the column at atmosphere pressure is 1 m/s, giving an overall plate efficiency of 50 per cent.

Assuming that the mixture is fed to the column as a boiling liquid and using a reflux ratio of twice the minimum value possible, determine the location of the feed plate and the rate at which the mixture can be separated.

Equilibria data:

Mole fraction alcohol in liquid	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.89
Mole fraction alcohol in vapour	0.43	0.526	0.577	0.615	0.655	0.70	0.754	0.82	0.89

Solution

The equilibrium data are plotted in Figure 11y, where the operating line corresponding to the minimum reflux ratio is drawn from the point (x_d, x_d) through the intersection of the vertical q-line and the equilibrium curve to give an intercept of 0.505.
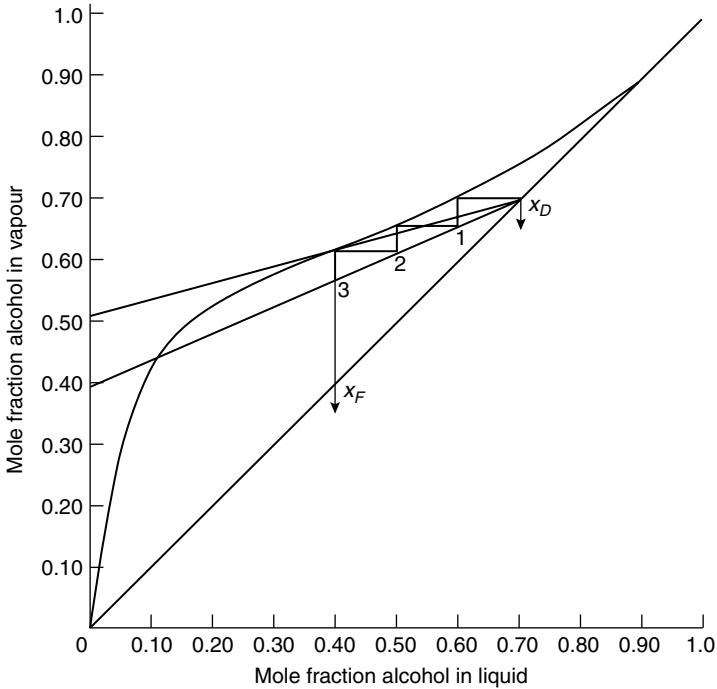

Figure 11y. Equilibrium data for Problem 11.32

Thus:
$$x_d/(R_m + 1) = 0.505 \quad \text{and} \quad R_m = 0.386$$

The actual value of R is then $(2 \times 0.386) = 0.772$ so that the top operating line may be constructed as shown.

This column contains the equivalent of (10×0.5), that is 5 theoretical plates, so that these may be stepped off from the point (x_d, x_d) to give the feed plate as the third from the top.

The problem as stated gives no bottom-product composition, so that whilst all flow-rates in the top of the column may be calculated, no information about the lower half can be derived. In the absence of these data, the feed rate cannot be determined, though the rate of distillate removal may be calculated as follows.

Mean molecular mass of top product = $(46 \times 0.7 + 18 \times 0.3) = 37.6$ kg/kmol
If the top temperature is assumed to be 353 K, then:

$$C = (1/22.4)(273/353) = 0.0345 \text{ kmol/s}^3$$

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If the vapour velocity $= 1$ m/s, the volumetric vapour flow at the top of the column is:

$$(\pi/4)(1.2)^2 \times 1 = 1.13 \text{ m}^3/\text{s}.$$

Hence: $\qquad\qquad\qquad\qquad V_n = (1.13 \times 0.0345) = 0.039 \text{ kmol/s}$

From the slope of the operating line:

$$L_n/V_n = 0.436$$

Thus: $\qquad\qquad\qquad\qquad L_n = (0.436 \times 0.039) = 0.017 \text{ kmol/s}$

As $R = 0.772$, then:

$$D = (L_n/0.772) = 0.022 \text{ kmol/s} \quad \text{or} \quad \underline{\underline{0.828 \text{ kg/s distillate}}}$$