

## Binary Distillation

In *distillation (fractionation)*, a feed mixture of two or more components is separated into two or more products, including, and often limited to, an overhead distillate and a bottoms, whose compositions differ from that of the feed. Most often, the feed is a liquid or a vapor–liquid mixture. The bottoms product is almost always a liquid, but the distillate may be a liquid or a vapor or both. The separation requires that (1) a second phase be formed so that both liquid and vapor phases are present and can contact each other on each stage within a separation column, (2) the components have different volatilities so that they will partition between the two phases to different extents, and (3) the two phases can be separated by gravity or other mechanical means. Distillation differs from absorption and stripping in that the second fluid phase is usually created by thermal means (vaporization and condensation) rather than by the introduction of a second phase that may contain an additional component or components not present in the feed mixture.

According to Forbes [1], the art of distillation dates back to at least the first century A.D. By the eleventh century, distillation was being used in Italy to produce alcoholic beverages. At that time, distillation was probably a batch process based on the use of just a single stage, the boiler. The feed to be separated, a liquid, was placed in a vessel to which heat was applied, causing part of the liquid to evaporate. The vapor passed out of the heating vessel and was cooled in

another chamber by transfer of heat through the wall of the chamber to water, producing condensate that dripped into a product receiver. The word *distillation* is derived from the Latin word *destillare*, which means dripping or trickling down. By at least the sixteenth century, it was known that the extent of separation could be improved by providing multiple vapor–liquid contacts (stages) in a so-called Rectificatorium. The term *rectification* is derived from the Latin words *recte facere*, meaning to improve. Modern distillation derives its ability to produce almost pure products from the use of multistage contacting.

Throughout the twentieth century, multistage distillation was by far the most widely used industrial method for separating liquid mixtures of chemical components. Unfortunately, distillation is a very energy-intensive technique, especially when the relative volatility,  $\alpha$ , of the components being separated is low ( $<1.50$ ). Mix et al. [2] report that the energy consumption for distillation in the United States for 1976 totaled  $2 \times 10^{15}$  Btu (2 quads), which was nearly 3% of the entire national energy consumption. Approximately two-thirds of the distillation energy was consumed by petroleum refining, where distillation is widely used to separate crude oil into petroleum fractions, light hydrocarbons ( $C_2$ 's to  $C_5$ 's), and aromatic chemicals. The separation of other organic chemicals, often in the presence of water, is widely practiced in the chemical industry.

### 7.0 INSTRUCTIONAL OBJECTIVES

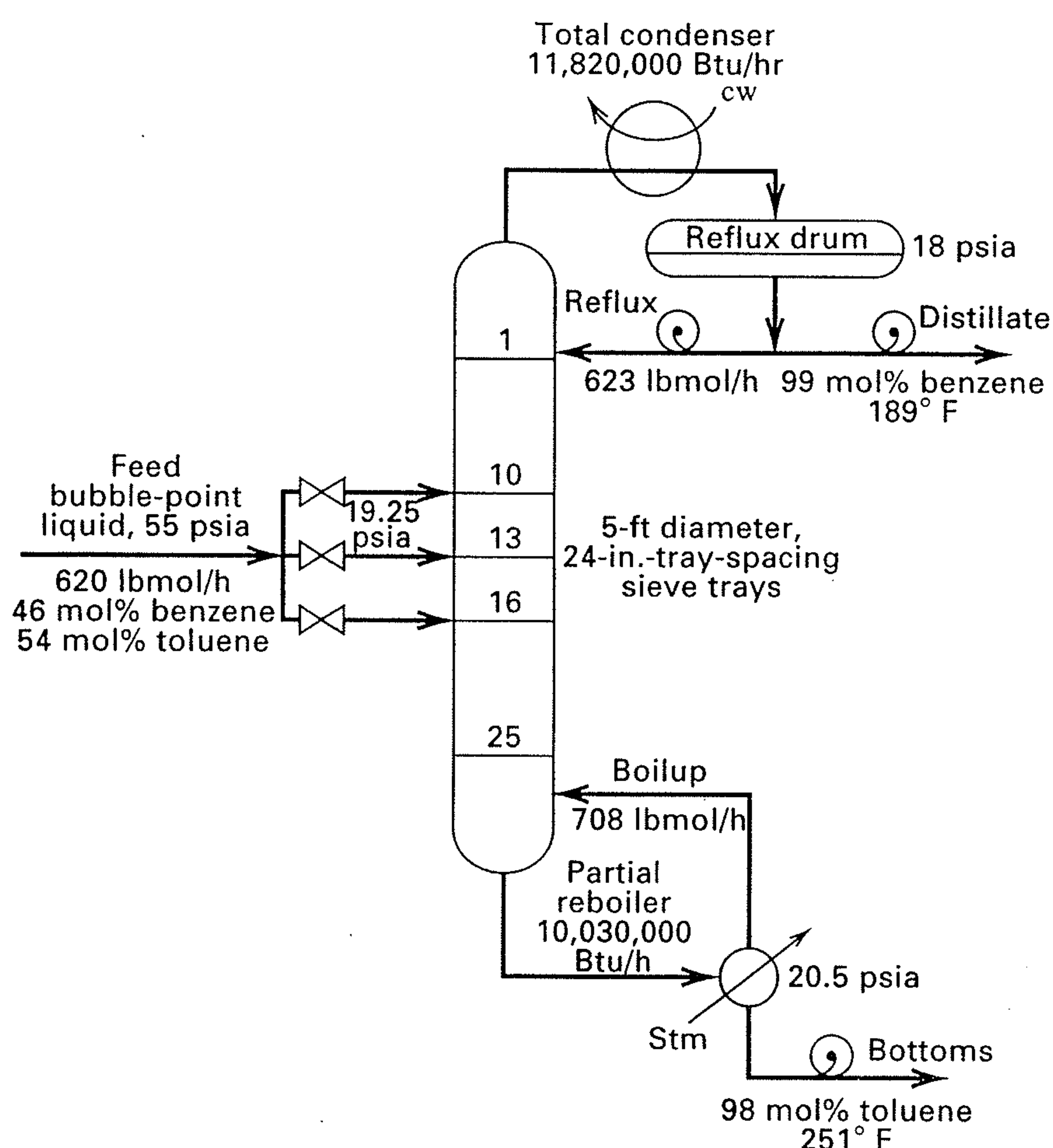
After completing this chapter, you should be able to:

- Explain the difference between distillation and absorption or stripping.
- Explain the need in distillation for a condenser to produce reflux and a reboiler to produce boilup
- Enumerate factors that influence design of a distillation column.
- Distinguish between required specifications and results that can be obtained from the McCabe–Thiele method for continuous binary distillation.
- Determine the five construction lines used in the McCabe–Thiele method using component material balances and vapor–liquid equilibrium relations.
- Explain the concept of “constant molar overflow,” assumptions required for its validity, and why it eliminates the need for energy balances around stages.
- Distinguish among five possible phase conditions of the feed.
- Apply the McCabe–Thiele method for determining minimum reflux ratio, minimum number of equilibrium stages, number of equilibrium stages for a specified reflux ratio greater than minimum, and optimal feed-stage location, given the required split between the two feed components.

- Select an appropriate condenser and a suitable operating pressure for a distillation separation.
- Explain differences among the three most common types of reboilers.
- Calculate condenser and reboiler heat duties and consider use of a feed preheater.
- Determine the optimal reflux ratio.
- Use the Murphree vapor stage efficiency to determine actual number of stages (plates) from the number of equilibrium stages.
- Extend the McCabe–Thiele method to multiple feeds, side streams, and open steam (in place of a reboiler).
- Estimate overall stage efficiency for binary distillation from correlations and laboratory column data.
- Determine the diameter of a trayed tower and size of the reflux drum.
- Determine packed height and diameter of a packed column.
- Use an enthalpy-concentration diagram when assumption of “constant molar overflow” is not valid.

### Industrial Example

The fundamentals of distillation are best understood by the study of binary distillation, the separation of a two-component mixture, which is the subject of this chapter. The more general and much more difficult case of a multicomponent mixture is covered in Chapters 10 and 11. A representative binary distillation operation is shown in Figure 7.1 for the separation of 620 lbmol/h (0.0781 kmol/s) of a binary mixture of 46 mol% benzene (the more volatile component) and 54 mol% toluene. The purpose of the 25-sieve-tray (equivalent to 20 theoretical stages plus a partial reboiler that acts as an additional theoretical stage)



**Figure 7.1** Distillation of a binary mixture of benzene and toluene.

distillation column is to separate the feed into a liquid distillate of 99 mol% benzene and a liquid bottoms product of 98 mol% toluene. The column operates at a pressure in the reflux drum of 18 psia (124 kPa), just slightly above ambient pressure. For a negligible pressure drop across the condenser and a vapor pressure drop of 0.1 psi/tray (0.69 kPa/tray), the pressure in the reboiler is  $18 + 0.1(25) = 20.5$  psia (141 kPa). In this range of pressure, benzene and toluene form nearly ideal mixtures with a relative volatility of from 2.26 at conditions of the bottom tray to 2.52 at the top tray, as determined from Raoult's law by (2-44). The reflux ratio (reflux rate to distillate rate) is 2.215. If an infinite number of stages were used, the required reflux ratio would be a minimum value of 1.708. Thus, the ratio of reflux rate to minimum reflux rate for this example is 1.297. Most distillation columns are designed to operate with optimal-reflux-to-minimum-reflux ratios of 1.1 to 1.5. If an infinite ratio of reflux to minimum reflux were used, only 10.7 theoretical stages would be required. Thus, the ratio of theoretical stages to minimum theoretical stages for this example is  $21/10.7 = 1.96$ . For most distillation columns, this ratio is approximately 2. The stage efficiency is 20/25 or 80%. This is close to the average efficiency observed for distillation.

The feed to the separation operation of Figure 7.1 is a saturated liquid at 55 psia (379 kPa). A bubble-point calculation gives a temperature of 294°F (419 K). When this feed is flashed adiabatically across the feed valve to the feed tray pressure of 19.25 psia (133 kPa), the feed temperature drops to 220°F (378 K), causing 23.4 mol% of the feed to be vaporized. A total condenser is used to obtain saturated liquid reflux and liquid distillate at a bubble-point temperature of 189°F (360 K) at 18 psia (124 kPa). The duty of the condenser is 11,820,000 Btu/h (3.46 MW). At the bottom of the column, a partial reboiler is used to produce vapor boilup and a liquid bottoms product. Assuming that the boilup and bottoms are in physical equilibrium, the

partial reboiler functions as an additional theoretical stage, giving a total of 21 theoretical stages. Because the bottoms product is a saturated liquid, its temperature of 251°F (395 K) corresponds to the bubble point of the bottoms at 20.5 psia (141 kPa). The duty of the reboiler is 10,030,000 Btu/h (2.94 MW), which is within 15% of the condenser duty.

The inside diameter of the distillation column in Figure 7.1 is a constant 5 ft (1.53 m). At the top tray this diameter corresponds to 84% of flooding, while at the bottom tray the percent flooding is 81%. As shown, the column can be fed at any one of three trays. For the design conditions, the optimal feed entry is between trays 12 and 13. However, should the feed composition or product specifications change, one of the other two feed trays could become optimal.

Distillation columns similar to that of Figure 7.1 have been built for diameters up to at least 30 ft (9.14 m). With a 24-in. (0.61-m) tray spacing, the maximum number of trays included in a single column is usually no greater than 150. In general, for the sharp separation of a binary mixture with a relative volatility less than 1.05, distillation can require many hundreds of trays, so a more efficient separation

technique should be sought. Even when distillation is the most economical separation technique, its second-law efficiency, using the calculational procedure developed in Chapter 2, can be less than 10%.

Technically, distillation is the most mature separation operation. Design and operation procedures are well established; for example, see Kister [3, 4]. Only when vapor-liquid equilibrium or other data are uncertain is a laboratory and/or pilot-plant study necessary prior to the design of a commercial unit. Table 7.1, taken partially from the study of Mix et al. [2], lists just some of the more common commercial binary distillation operations in decreasing order of difficulty of separation. Included are representative nominal values of relative volatility, number of trays, column operating pressure, and reflux-to-minimum-reflux ratio. Although the data in Table 7.1 refer to trayed towers, distillation can also be carried out in packed columns. More and more frequently, additional distillation capacity is being achieved with existing trayed towers by replacing all or some of the trays with sections of random or structured packing.

**Table 7.1** Representative Commercial Binary Distillation Operations [2]

Binary Mixture	Average Relative Volatility	Number of Trays	Typical Operating Pressure, psia	Reflux-to-Minimum-Reflux Ratio
1,3-Butadiene/vinyl acetylene	1.16	130	75	1.70
Vinyl acetate/ethyl acetate	1.16	90	15	1.15
<i>o</i> -Xylene/ <i>m</i> -xylene	1.17	130	15	1.12
Isopentane/ <i>n</i> -pentane	1.30	120	30	1.20
Isobutane/ <i>n</i> -butane	1.35	100	100	1.15
Ethylbenzene/styrene	1.38	34	1	1.71
Propylene/propane	1.40	138	280	1.06
Methanol/ethanol	1.44	75	15	1.20
Water/acetic acid	1.83	40	15	1.35
Ethylene/ethane	1.87	73	230	1.07
Acetic acid/acetic anhydride	2.02	50	15	1.13
Toluene/ethylbenzene	2.15	28	15	1.20
Propane/1,3-butadiene	2.18	40	120	1.13
Ethanol azeotrope/water	2.21	60	15	1.35
Isopropanol/water	2.23	12	15	1.28
Benzene/toluene	3.09	34	15	1.15
Methanol/water	3.27	60	45	1.31
Cumene/phenol	3.76	38	1	1.21
Benzene/ethylbenzene	6.79	20	15	1.14
HCN/water	11.20	15	50	1.36
Ethylene oxide/water	12.68	50	50	1.19
Formaldehyde/methanol	16.70	23	50	1.17
Water/ethylene glycol	81.20	16	4	1.20

In this chapter, equipment for conducting distillation operations is discussed and fundamental equilibrium-based and rate-based calculational procedures are developed for binary mixtures. Trayed and packed distillation columns are

identical in most respects to the absorption and stripping columns discussed in the previous chapter. Therefore, where appropriate, reference is made to Chapter 6 and only important differences are discussed in this chapter.

## 7.1 EQUIPMENT AND DESIGN CONSIDERATIONS

Industrial distillation operations are most commonly conducted in trayed towers, but packed columns are finding increasing use. Occasionally, distillation columns contain both trays and packing. Types of trays and packings are identical to those used for absorption and stripping, as described in Section 6.1, shown in Figures 6.2 to 6.7, and compared in Tables 6.2 and 6.3.

Factors that influence the design or analysis of a binary-distillation operation include:

1. Feed flow rate, composition, temperature, pressure, and phase condition
2. Desired degree of separation between two components
3. Operating pressure (which must be below the critical pressure of the mixture)
4. Vapor pressure drop, particularly for vacuum operation
5. Minimum reflux ratio and actual reflux ratio
6. Minimum number of equilibrium stages and actual number of equilibrium stages (stage efficiency)
7. Type of condenser (total, partial, or mixed)
8. Degrees of subcooling, if any, of the liquid reflux
9. Type of reboiler (partial or total)
10. Type of contacting (trays or packing or both)
11. Height of the column
12. Feed-entry stage
13. Diameter of the column
14. Column internals.

The phase condition (also called thermal condition) of the feed is determined at the feed-tray pressure by an adiabatic-flash calculation across the feed valve. As the molar fraction of vapor in the feed increases, the required reflux ratio ( $L/D$ ) increases, but the corresponding boilup ratio ( $V/B$ ) decreases. The column operating pressure in the reflux drum should correspond to a distillate temperature somewhat higher (e.g., 10 to 50°F or 6 to 28°C) than the supply temperature of the cooling water used as the coolant in the overhead condenser. However, if this pressure approaches the critical pressure of the more volatile component, then a lower operating pressure must be used and a refrigerant is required as coolant. For example, in Table 7.1, the separation of ethylene/ethane is

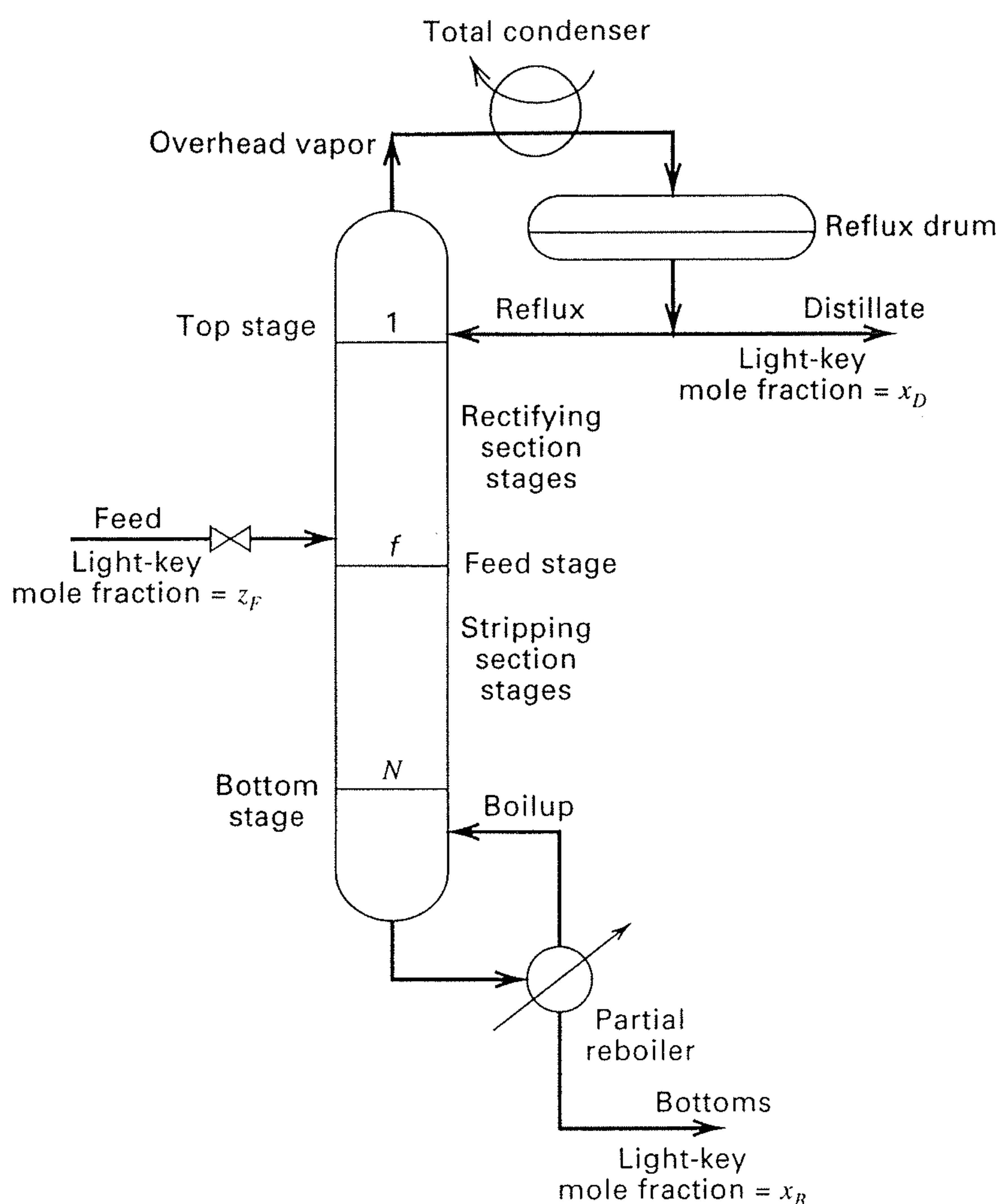
conducted at 230 psia (1,585 kPa), giving a column top temperature of  $-40^{\circ}\text{F}$  (233 K), which requires a refrigerant. Water at  $80^{\circ}\text{F}$  (300 K) cannot be used in the condenser because the critical temperature of ethylene is  $48.6^{\circ}\text{F}$  (282 K). If the estimated pressure is less than atmospheric pressure, the operating pressure at the top of the column is often set just above atmospheric pressure to avoid vacuum operation, unless the temperature at the bottom of the column is found to exceed a bottoms temperature limited by decomposition, polymerization, excessive corrosion, or other chemical reaction. In that case, vacuum operation is necessary. In Table 7.1, vacuum operation is required for the separation of ethylbenzene from styrene to maintain a bottoms temperature sufficiently low to prevent polymerization of styrene.

For given (1) feed, (2) desired degree of separation, and (3) operating pressure, a minimum reflux ratio exists that corresponds to an infinite number of theoretical stages; and a minimum number of theoretical stages exists that corresponds to an infinite reflux ratio. A design trade-off is usually made between the number of stages and the reflux ratio. A graphical method for determining the data needed to make this trade-off and to determine the optimal feed-stage location is developed in the next section.

## 7.2 MCCABE–THIELE GRAPHICAL EQUILIBRIUM-STAGE METHOD FOR TRAYED TOWERS

Consider the general countercurrent-flow, multistage, binary-distillation operation shown in Figure 7.2. The operation consists of a column containing the equivalent of  $N$  theoretical stages; a total condenser in which the overhead vapor leaving the top stage is totally condensed to give a liquid distillate product and liquid reflux that is returned to the top stage; a partial reboiler in which liquid from the bottom stage is partially vaporized to give a liquid bottoms product and vapor boilup that is returned to the bottom stage, and an intermediate feed stage. By means of multiple, countercurrent contacting stages arranged in a two-section cascade with reflux and boilup, as discussed in Section 5.4, it is possible to achieve a sharp separation between the two components in the feed unless an azeotrope is formed, in which case one of the two products will approach the azeotropic composition.

The feed, which contains a more-volatile (light) component (the *light key*, LK), and a less-volatile (heavy)



**Figure 7.2** Distillation operation using a total condenser and partial reboiler.

component (the *heavy key*, HK), enters the column at a feed stage,  $f$ . At the feed-stage pressure, the feed may be liquid, vapor, or a mixture of liquid and vapor, with its overall mole-fraction composition with respect to the light component denoted by  $z_F$ . The mole fraction of the light key in the distillate is  $x_D$ , while the mole fraction of the light key in the bottoms product is  $x_B$ . Corresponding compositions with respect to the heavy key are  $1 - z_F$ ,  $1 - x_D$ , and  $1 - x_B$ .

The goal of distillation is to produce from the feed a distillate, rich in the light key (i.e.,  $x_D$  approaching 1.0), and a bottoms product, rich in the heavy key (i.e.,  $x_B$  approaching 0.0). The ease or difficulty with which the separation can be achieved depends on the relative volatility,  $\alpha$ , of the two components (LK = 1 and HK = 2), where

$$\alpha_{1,2} = K_1/K_2 \quad (7-1)$$

Methods for estimating  $K$ -values are discussed in Chapter 2.

If the two components form ideal solutions and follow the ideal-gas law in the vapor phase, Raoult's law applies to give

$$K_1 = P_1^s/P \quad \text{and} \quad K_2 = P_2^s/P$$

and from (7-1), the relative volatility is given simply by the ratio of vapor pressures,  $\alpha_{1,2} = P_1^s/P_2^s$  and thus is a function only of temperature. As discussed in Section 4.2, as the temperature (and therefore the pressure) increases,  $\alpha_{1,2}$

decreases. At the convergence pressure of the mixture,  $\alpha_{1,2} = 1.0$  and a separation cannot be achieved at this or any higher pressure.

The relative volatility can be expressed in terms of equilibrium vapor and liquid compositions from the definition of the  $K$ -value as  $K_i = y_i/x_i$ . For a binary mixture,

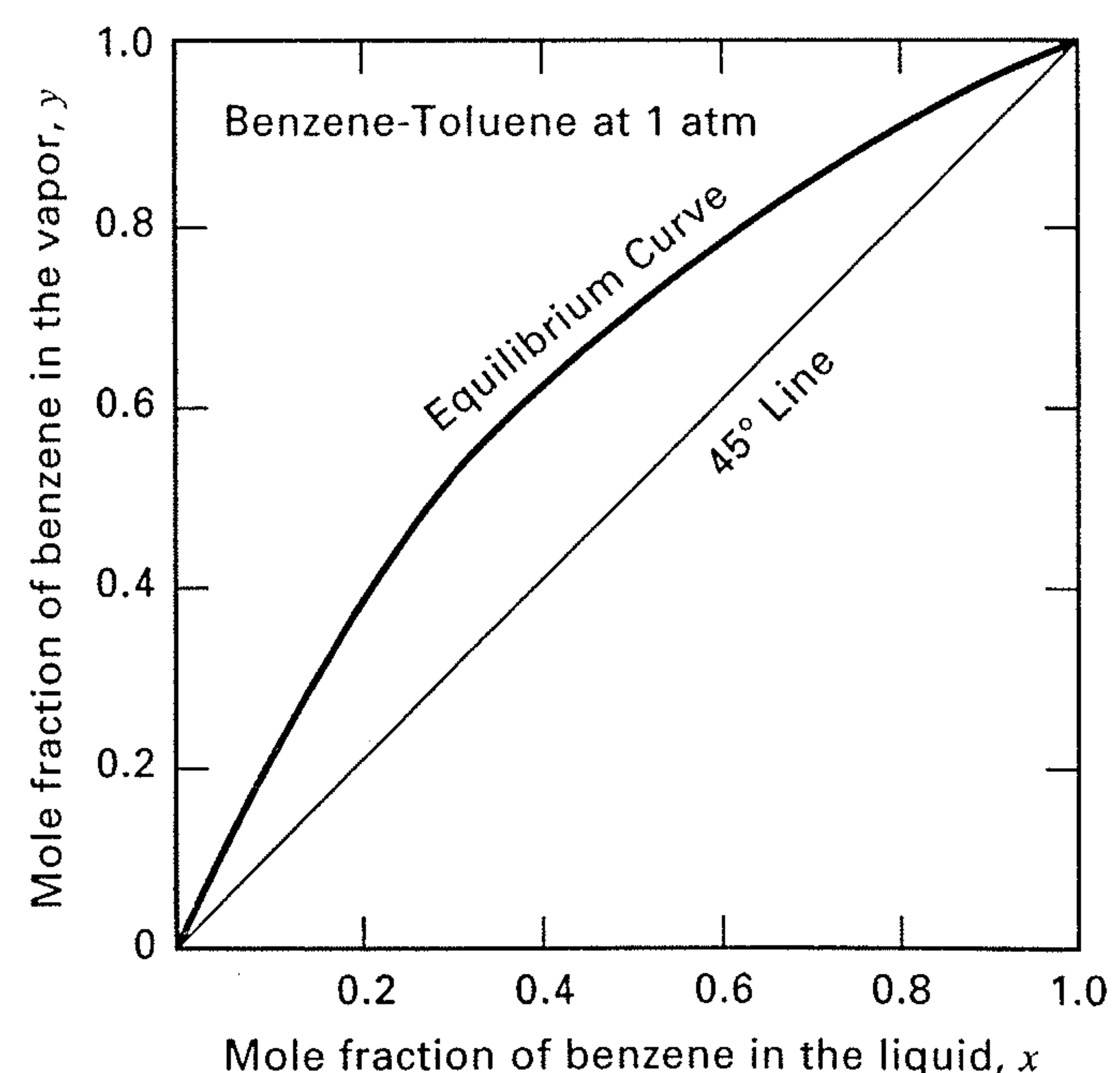
$$\alpha_{1,2} = \frac{y_1/x_1}{y_2/x_2} = \frac{y_1(1-x_1)}{x_1(1-y_1)} \quad (7-2)$$

Solving (7-2) for  $y_1$ ,

$$y_1 = \frac{\alpha_{1,2}x_1}{1 + x_1(\alpha_{1,2} - 1)} \quad (7-3)$$

For ideal binary mixtures of components with close boiling points, the temperature change over the column is small and  $\alpha_{1,2}$  is almost constant. In any case, for a given pressure  $P$  and liquid-phase composition  $x_1$ , the Gibbs phase rule, discussed in Chapter 4, fixes the temperature and equilibrium-vapor composition. An equilibrium curve for the benzene-toluene system is shown in Figure 7.3, where  $y$  and  $x$  correspond to the light key, benzene, and the pressure is 1 atm, at which pure benzene and pure toluene boil at 176 and 231°F, respectively. Thus, these two components are not close-boiling. Using (7-3) with this curve,  $\alpha$  varies from about 2.6 at the bottom of the curve to about 2.35 at the top of the curve. Representative equilibrium curves for some average values of  $\alpha$  are shown in Figure 4.5. The higher the average value of  $\alpha$ , the easier it is to achieve the desired separation. Average values of  $\alpha$  for the distillation operations in Table 7.1 range from 1.16 to 81.2.

In 1925, McCabe and Thiele [5] published an approximate graphical method for combining the equilibrium curve of Figure 7.3 with operating-line curves to estimate, for a given binary-feed mixture and column operating pressure, the number of equilibrium stages and the amount of reflux required for a desired degree of separation of the feed.



**Figure 7.3** Equilibrium curve for benzene-toluene at 1 atm.

Although computer-aided methods, discussed later in Chapter 10, are more accurate and easier to apply, the graphical construction of the McCabe–Thiele method greatly facilitates the visualization of many of the important aspects of multistage distillation, and therefore the effort required to learn the method is well justified.

Typical problem specifications for and results from the McCabe–Thiele method are summarized in Table 7.2. This table applies to a simple, binary-distillation operation, like that in Figure 7.2, for a single feed and two products. The distillate can be a liquid from a total condenser, as shown in Figure 7.2, or a vapor from a partial condenser. The feed phase condition must be known at the column pressure, which is assumed to be uniform throughout the column for the McCabe–Thiele method. The type of condenser and reboiler must be specified, as well as the ratio of reflux to minimum reflux. From the specification of  $x_D$  and  $x_B$  for the light key, the distillate and bottoms flow rates,  $D$  and  $B$ , are fixed by material balance, since

$$Fz_F = x_D D + x_B B$$

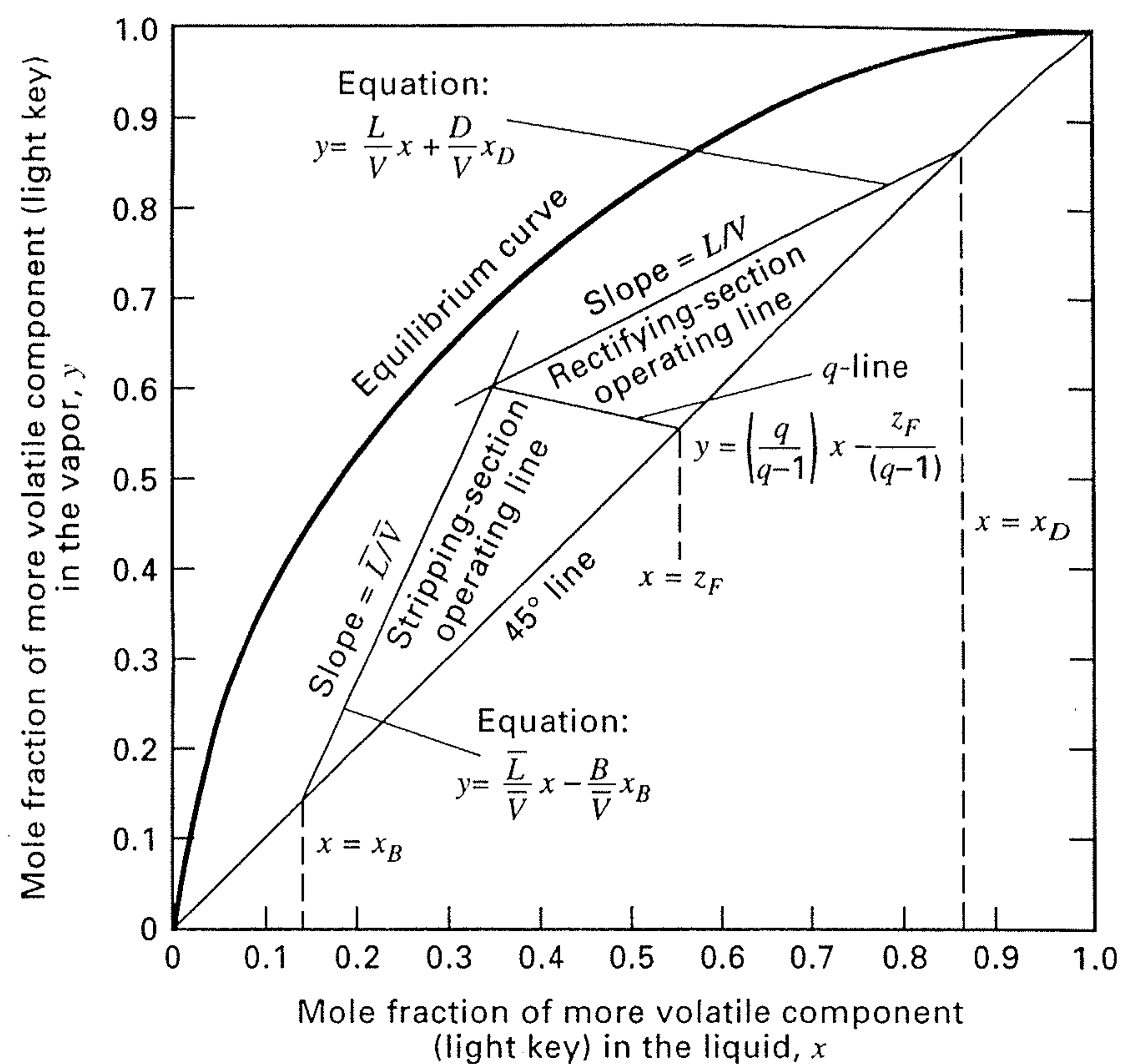
But,  $B = F - D$  and therefore

$$Fz_F = x_D D + x_B (F - D)$$

**Table 7.2** Specifications for and Results from the McCabe–Thiele Method for Binary Distillation

Specifications	
$F$	Total feed rate
$z_F$	Mole-fraction composition of the feed
$P$	Column operating pressure (assumed uniform throughout the column)
	Phase condition of the feed at column pressure
	Vapor–liquid equilibrium curve for the binary mixture at column pressure
	Type of overhead condenser (total or partial)
	Type of reboiler (usually partial)
$x_D$	Mole-fraction composition of the distillate
$x_B$	Mole-fraction composition of the bottoms
$R/R_{\min}$	Ratio of reflux to minimum reflux
Results	
$D$	Distillate flow rate
$B$	Bottoms flow rate
$N_{\min}$	Minimum number of equilibrium stages
$R_{\min}$	Minimum reflux ratio, $L_{\min}/D$
$R$	Reflux ratio, $L/D$
$V_B$	Boilup ratio, $\bar{V}/B$
$N$	Number of equilibrium stages
	Optimal feed-stage location
	Stage vapor and liquid compositions

All mole fraction compositions are for the light key.



**Figure 7.4** Construction lines for McCabe–Thiele method.

or

$$D = F \left( \frac{z_F - x_B}{x_D - x_B} \right)$$

This result requires  $x_B < z_F < x_D$ .

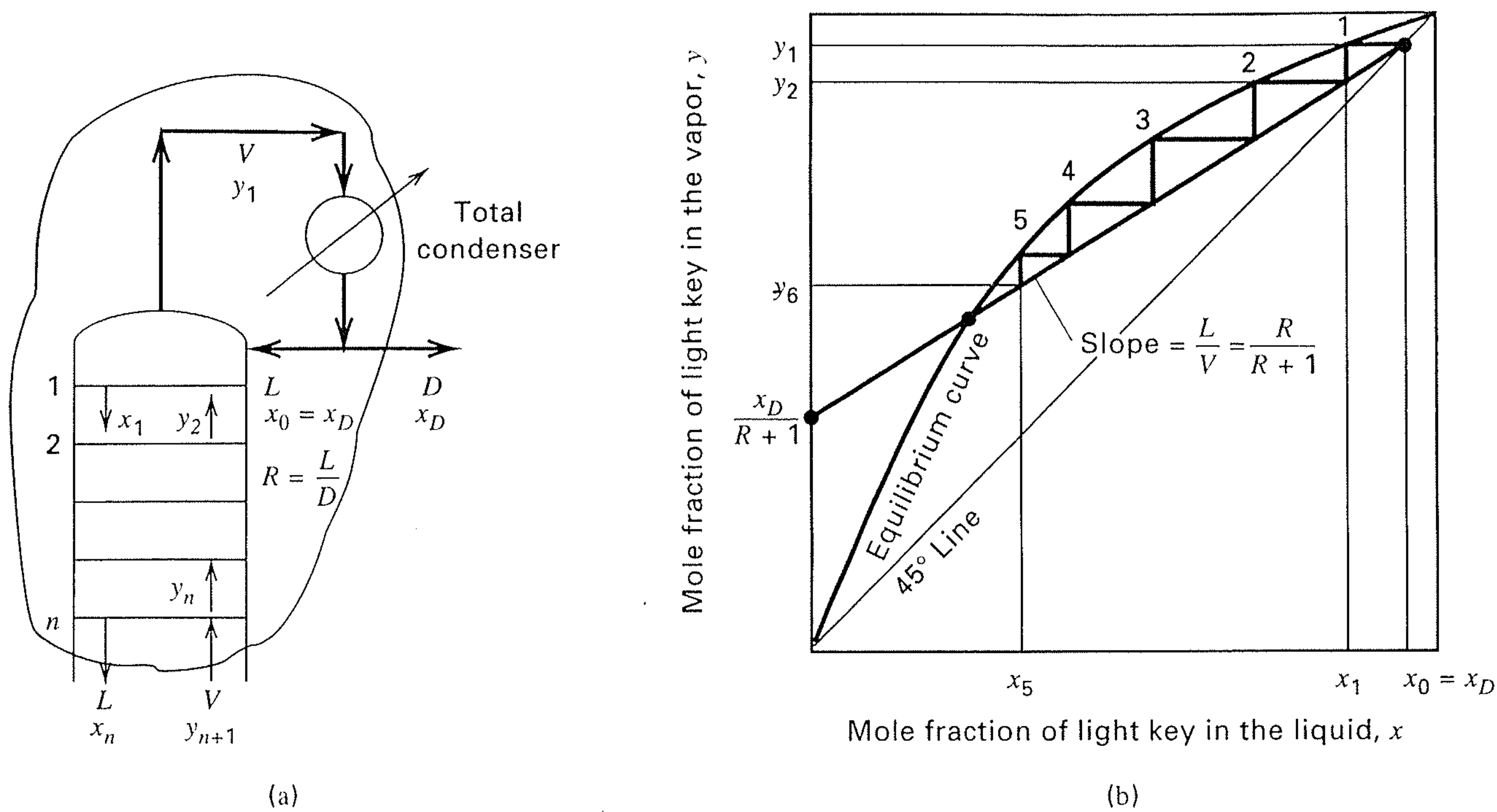
The McCabe–Thiele method determines not only  $N$ , the number of equilibrium stages, but also  $N_{\min}$ ,  $R_{\min}$ , and the optimal stage for feed entry. Following the application of the McCabe–Thiele method, energy balances are applied to estimate condenser and reboiler heat duties.

Besides the equilibrium curve, the McCabe–Thiele method involves a 45° reference line, separate operating lines for the upper *rectifying* (enriching) section of the column and the lower *stripping* (exhausting) section of the column, and a fifth line (the *q*-line or feed line) for the phase or thermal condition of the feed. A typical set of these lines is shown in Figure 7.4. Equations for these lines are derived in the following subsection.

### Rectifying Section

As shown in Figure 7.2, the rectifying section of equilibrium stages extends from the top stage, 1, to just above the feed stage,  $f$ . Consider a top portion of the rectifying stages, including the total condenser. A material balance for the light key over the envelope shown in Figure 7.5a for the total condenser and stages 1 to  $n$  is as follows, where  $y$  and  $x$  refer to vapor and liquid mole fractions, respectively, for the light key:

$$V_{n+1}y_{n+1} = L_n x_n + D x_D \quad (7-4)$$



**Figure 7.5** McCabe–Thiele operating line for the rectifying section.

Solving for  $y_{n+1}$ :

$$y_{n+1} = \frac{L_n}{V_{n+1}}x_n + \frac{D}{V_{n+1}}x_D \quad (7-5)$$

Equation (7-5) relates the compositions,  $y_{n+1}$  and  $x_n$ , of two passing streams,  $V_{n+1}$  and  $L_n$ , respectively. For (7-5) to plot as a straight line of the form  $y = mx + b$ , which is the locus of compositions of all passing streams in the rectifying section, total molar flow rates  $L$  and  $V$  must not vary from stage to stage. This is the case if:

1. The two components have equal and constant molar enthalpies of vaporization (latent heats).
2. Component sensible-enthalpy changes ( $C_P\Delta T$ ) and heat of mixing are negligible compared to latent heat changes.
3. The column is well insulated so that heat loss is negligible.
4. The pressure is uniform throughout the column (no pressure drop).

These assumptions are referred to as the *McCabe–Thiele assumptions* leading to the condition of *constant molar overflow* in the rectifying section, which refers to a molar liquid flow rate that remains constant as the liquid overflows each weir from one stage to the next. Since a total material balance for the rectifying-section envelope in Figure 7.5a gives  $V_{n+1} = L_n + D$ , if  $L$  is constant, then  $V$  is also constant for a particular value of  $D$ . Thus, (7-5) can be rewritten as

$$y = \frac{L}{V}x + \frac{D}{V}x_D \quad (7-6)$$

as shown in Figure 7.4. Thus, the slope of the operating line is  $L/V$ , which is constant. Because  $V > L$ ,  $L/V < 1$  in the rectifying section, as seen in Figure 7.5b.

For constant molar overflow, it is not necessary to consider energy balances in either the rectifying or stripping

sections; only material balances and a vapor–liquid equilibrium curve are required. However, energy balances are needed to determine condenser and reboiler duties, as discussed later.

The liquid entering the top stage is the external reflux rate,  $L_0$ , and its ratio to the distillate rate,  $L_0/D$ , is the reflux ratio,  $R$ . Because of the assumption of constant molar overflow,  $R$  is a constant in the rectifying section, equal to  $L/D$ . Since  $V = L + D$ , the slope of the operating line is readily related to the reflux ratio:

$$\frac{L}{V} = \frac{L}{L + D} = \frac{L/D}{L/D + D/D} = \frac{R}{R + 1} \quad (7-7)$$

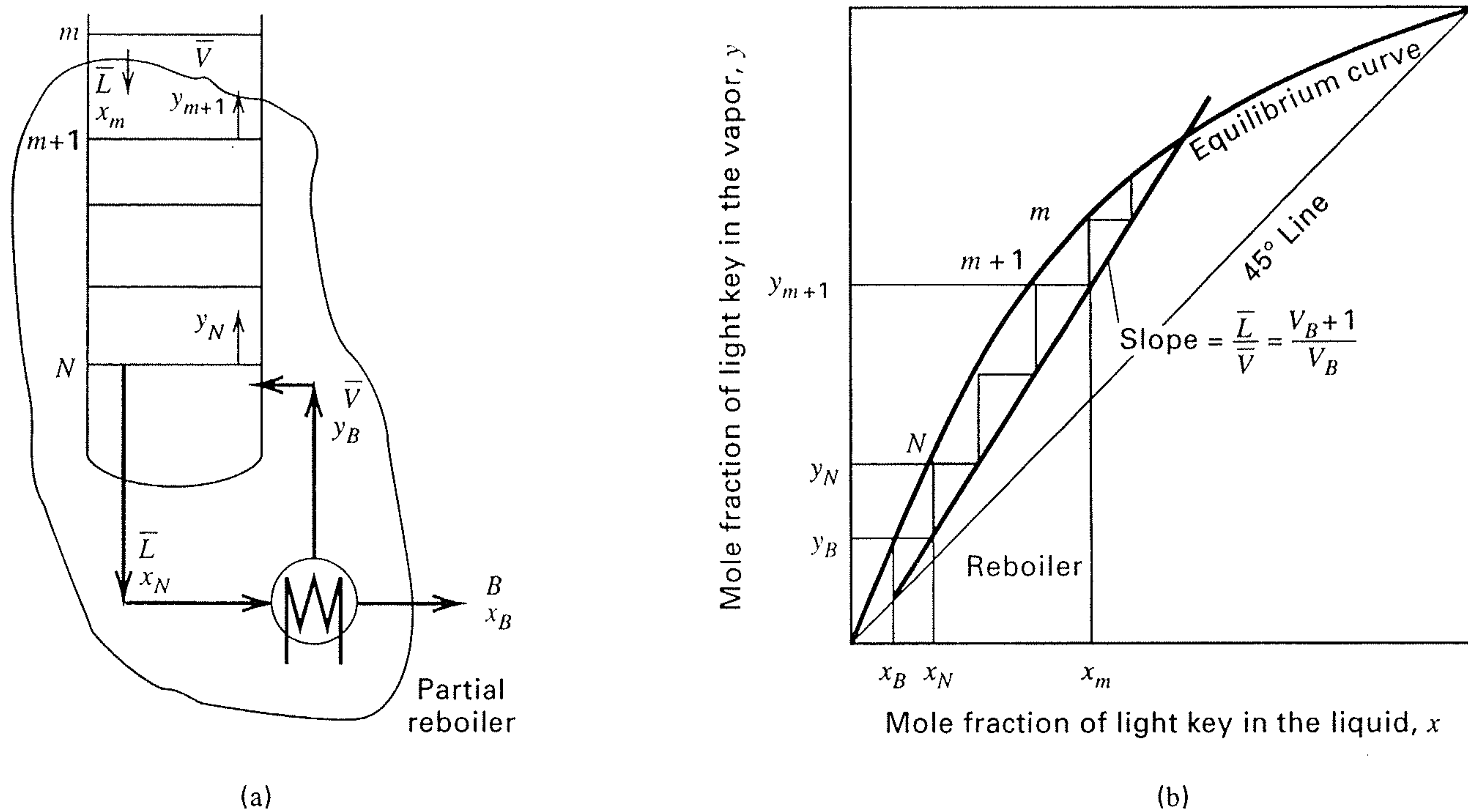
Similarly,

$$\frac{D}{V} = \frac{D}{L + D} = \frac{1}{R + 1} \quad (7-8)$$

Combining (7-6), (7-7), and (7-8) produces the most useful form of the operating line for the rectifying section:

$$y = \left(\frac{R}{R + 1}\right)x + \left(\frac{1}{R + 1}\right)x_D \quad (7-9)$$

If values of  $R$  and  $x_D$  are specified, (7-9) plots as a straight line with an intersection at  $y = x_D$  on the 45° line, a slope of  $L/V = R/(R + 1)$ , and an intersection at  $y = x_D/(R + 1)$  for  $x = 0$ , as shown in Figure 7.5b, which also contains a 45° line and an equilibrium curve. The equilibrium stages are stepped off in the manner described in Section 6.3 for absorption. Starting from the point  $(y_1 = x_D, x_0 = x_D)$  on the operating line and the 45° line, a horizontal line is drawn to the left until it intersects the equilibrium curve at  $(y_1, x_1)$ , that is, the compositions of the *equilibrium phases* leaving the top equilibrium stage. A vertical line is now dropped until it intersects the operating line at the point  $(y_2, x_1)$ , the compositions of the two *phases passing* each other between stages 1 and 2. The horizontal- and vertical-line constructions are



**Figure 7.6** McCabe–Thiele operating line for the stripping section.

continued down the rectifying section in the manner shown in Figure 7.5b to give the staircase construction shown, which is arbitrarily terminated at stage 5. The optimal stage for termination is considered later.

### Stripping Section

As shown in Figure 7.2, the stripping section of equilibrium stages extends from the feed to the bottom stage. In Figure 7.6a, consider a bottom portion of the stripping stages, including the partial reboiler and extending up from stage  $N$  to stage  $m + 1$ , located somewhere below the feed. A material balance for the light key over the envelope shown in Figure 7.6a results in

$$\bar{L}x_m = \bar{V}y_{m+1} + Bx_B \quad (7-10)$$

Solving for  $y_{m+1}$ :

$$y_{m+1} = \frac{\bar{L}}{\bar{V}}x_m - \frac{B}{\bar{V}}x_B$$

or

$$y = \frac{\bar{L}}{\bar{V}}x - \frac{B}{\bar{V}}x_B \quad (7-11)$$

where  $\bar{L}$  and  $\bar{V}$  are the total molar flows, which by the constant-molar-overflow assumption remain constant from stage to stage. The slope of this operating line for the compositions of passing streams in the stripping section is seen to be  $\bar{L}/\bar{V}$ . Because  $\bar{L} > \bar{V}$ ,  $\bar{L}/\bar{V} > 1$ , as seen in Figure 7.6b. This is the inverse of conditions in the rectifying section.

The vapor leaving the partial reboiler is assumed to be in equilibrium with the liquid bottoms product. Thus, the partial reboiler acts as an additional equilibrium stage. The vapor rate leaving it is called the *boilup*,  $\bar{V}_{N+1}$ , and its ratio to the bottoms product rate,  $V_B = \bar{V}_{N+1}/B$ , is the *boilup ratio*. Because of the constant-molar-overflow assumption,  $V_B$  is constant in the stripping section. Since  $\bar{L} = \bar{V} + B$ ,

$$\frac{\bar{L}}{\bar{V}} = \frac{\bar{V} + B}{\bar{V}} = \frac{V_B + 1}{V_B} \quad (7-12)$$

Similarly,

$$\frac{B}{\bar{V}} = \frac{1}{V_B} \quad (7-13)$$

Combining (7-11), (7-12), and (7-13), the operating-line equation for the stripping section becomes

$$y = \left( \frac{V_B + 1}{V_B} \right) x - \left( \frac{1}{V_B} \right) x_B \quad (7-14)$$

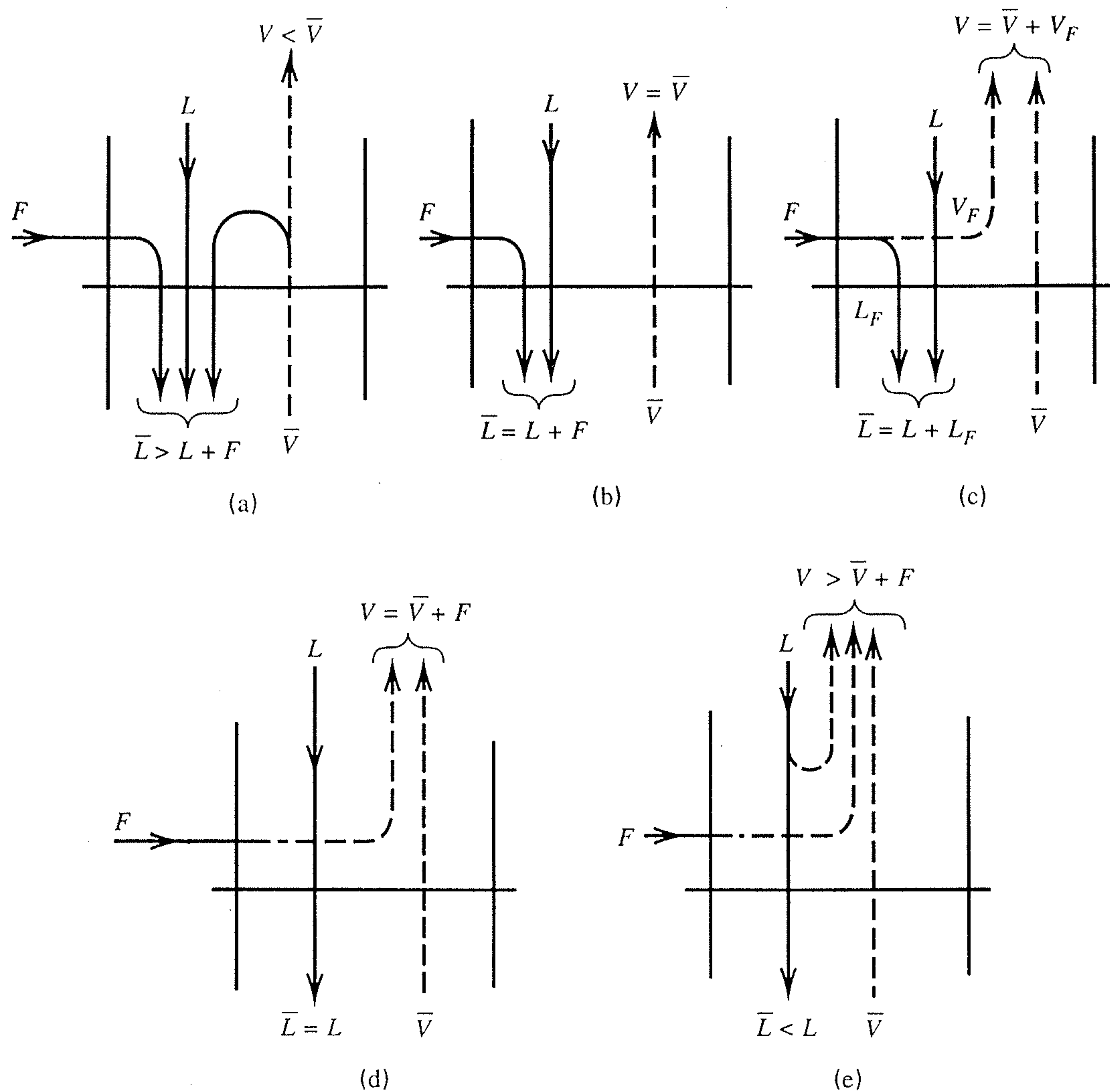
If values of  $V_B$  and  $x_B$  are known, (7-14) can be plotted, together with the equilibrium curve and a 45° line, as a straight line with an intersection at  $y = x_B$  on the 45° line and a slope of  $\bar{L}/\bar{V} = (V_B + 1)/V_B$ , as shown in Figure 7.6b. The equilibrium stages are stepped off, in a manner similar to that described for the rectifying section, starting from the point  $(y = x_B, x = x_B)$  on the operating and 45° lines and moving upward on a vertical line until the equilibrium curve is intersected at  $(y = y_B, x = x_B)$ , which represents the equilibrium mole fractions in the vapor and liquid leaving the partial reboiler. From that point, the staircase is constructed by drawing horizontal and then vertical lines, moving back and forth between the operating line and equilibrium curve, as observed in Figure 7.6b, where the staircase is arbitrarily terminated at stage  $m$ . Next, we determine where to terminate the two operating lines.

### Feed-Stage Considerations

Thus far, the McCabe–Thiele construction has not considered the feed to the column. In determining the operating lines for the rectifying and stripping sections, it is very important to note that although  $x_D$  and  $x_B$  can be selected independently,  $R$  and  $V_B$  are related by the feed phase condition.

Consider the five possible feed conditions shown in Figure 7.7, which assumes that the feed has been flashed adiabatically to the feed-stage pressure. If the feed is a bubble-point liquid, it adds to the reflux,  $L$ , coming from the stage above to give  $\bar{L} = L + F$ . If the feed is a dew-point





**Figure 7.7** Possible feed conditions: (a) subcooled-liquid feed; (b) bubble-point liquid feed; (c) partially vaporized feed; (d) dew-point vapor feed; (e) superheated-vapor feed.

[Adapted from W.L. McCabe, J.C. Smith, and P. Harriott, *Unit Operations of Chemical Engineering*, 5th ed., McGraw-Hill, New York (1993).]

vapor, it adds to the boilup vapor,  $\bar{V}$ , coming from the stage below to give  $V = \bar{V} + F$ . For a partially vaporized feed, as shown in Figure 7.7c,  $F = L_F + V_F$  and  $\bar{L} = L + L_F$  and  $V = \bar{V} + V_F$ . If the feed is a subcooled liquid, it will cause a portion of the boilup,  $\bar{V}$ , to condense, giving  $\bar{L} > L + F$  and  $V < \bar{V}$ . If the feed is a superheated vapor, it will cause a portion of the reflux,  $L$ , to vaporize, giving  $\bar{L} < L$  and  $V > \bar{V} + F$ .

For cases (b), (c), and (d) of Figure 7.7, covering a range of feed conditions from a saturated liquid to a saturated vapor, the boilup  $\bar{V}$  is related to the reflux  $L$  by the material balance:

$$\bar{V} = L + D - V_F \quad (7-15)$$

and the boilup ratio,  $V_B = \bar{V}/B$ , is

$$V_B = \frac{L + D - V_F}{B} \quad (7-16)$$

Alternatively, the reflux can be determined from the boilup by

$$L = \bar{V} + B - L_F \quad (7-17)$$

Although distillation operations can be specified by either the reflux ratio  $R$  or the boilup ratio  $V_B$ , by tradition  $R$  or  $R/R_{\min}$  is used because the distillate product is most often the more important product.

For the other two cases, (a) and (e) of Figure 7.7,  $V_B$  and  $R$  cannot be related by simple material balances alone. It is necessary to consider an energy balance to convert sensible enthalpy into latent enthalpy of phase change. This is most

conveniently done by defining a parameter,  $q$ , as the ratio of the increase in molar reflux rate across the feed stage to the molar feed rate,

$$q = \frac{\bar{L} - L}{F} \quad (7-18)$$

or by material balance around the feed stage,

$$q = 1 + \frac{\bar{V} - V}{F} \quad (7-19)$$

Values of  $q$  for the five feed conditions are

Feed condition	$q$
Subcooled liquid	$>1$
Bubble-point liquid	1
Partially vaporized	$L_F/F = 1 - \text{molar fraction vaporized}$
Dew-point vapor	0
Superheated vapor	$<0$

To determine values of  $q$  for subcooled liquid and superheated vapor, a more general definition of  $q$  is applied:

$q$  = enthalpy change to bring the feed to a dew-point vapor divided by enthalpy of vaporization of the feed (dew-point vapor minus bubble-point liquid), that is,

$$q = \frac{(h_F)_{\text{sat'd vapor temperature}} - (h_F)_{\text{feed temperature}}}{(h_F)_{\text{sat'd vapor temperature}} - (h_F)_{\text{sat'd liquid temperature}}} \quad (7-20)$$

For a subcooled liquid feed, (7-20) becomes

$$q = \frac{\Delta H^{\text{vap}} + C_{PL}(T_b - T_F)}{\Delta H^{\text{vap}}} \quad (7-21)$$

For a superheated vapor, (7-20) becomes

$$q = \frac{C_{P_V}(T_d - T_F)}{\Delta H^{\text{vap}}} \quad (7-22)$$

where  $C_{P_L}$  and  $C_{P_V}$  are the liquid and vapor molar heat capacities, respectively,  $\Delta H^{\text{vap}}$  is the molar enthalpy change from the bubble point to the dew point, and  $T_F$ ,  $T_d$ , and  $T_b$  are the feed, dew-point, and bubble-point temperatures, respectively, of the feed at the column operating pressure.

Instead of using (7-14) to locate the stripping operating line on the McCabe–Thiele diagram, it is more common to use an alternative method that involves a  $q$ -line (feed line), which is included in Figure 7.4. The  $q$ -line, one point of which is the intersection of the rectifying and stripping operating lines, is derived in the following manner. Combining (7-11) with (7-6) gives

$$y(V - \bar{V}) = (L - \bar{L})x + Dx_D + Bx_B \quad (7-23)$$

But

$$Dx_D + Bx_B = Fz_F \quad (7-24)$$

and a material balance around the feed stage gives

$$F + \bar{V} + L = V + \bar{L} \quad (7-25)$$

Combining (7-23) to (7-25) with (7-18) gives

$$y = \left( \frac{q}{q-1} \right) x - \left( \frac{z_F}{q-1} \right) \quad (7-26)$$

which is the equation for the  $q$ -line. This line is located on the McCabe–Thiele diagram by noting that when  $x = z_F$ , (7-26) reduces to the point  $y = z_F = x$ , which lies on the 45° line. From (7-26), the slope of the line is  $q/(q-1)$ . This construction is shown in Figure 7.4 for a partially vaporized feed, for which  $0 < q < 1$  and  $-\infty < [q/(q-1)] < 0$ . Following the placement of the rectifying-section operating line and the  $q$ -line, the stripping-section operating line is located by drawing a straight line from the point  $(y = x_B, x = x_B)$  on the 45° line to and through the point of intersection of the  $q$ -line and the rectifying-section operating line as shown in Figure 7.4. The point of intersection must lie somewhere between the equilibrium curve and the 45° line.

As  $q$  changes from a value greater than 1 (subcooled liquid) to a value less than 0 (superheated vapor), the slope of the  $q$ -line,  $q/(q-1)$ , changes from a positive value to a negative value and back to a positive value, as shown in Figure 7.8. For a saturated liquid feed, the  $q$ -line is vertical; for a saturated vapor, the  $q$ -line is horizontal.

### Determination of Number of Equilibrium Stages and Feed-Stage Location

Following the construction of the five lines shown in Figure 7.4, the number of equilibrium stages required for the entire column, as well as the location of the feed stage, are determined by stepping off stages by any of several ways. The stages can be stepped off first from the top down and

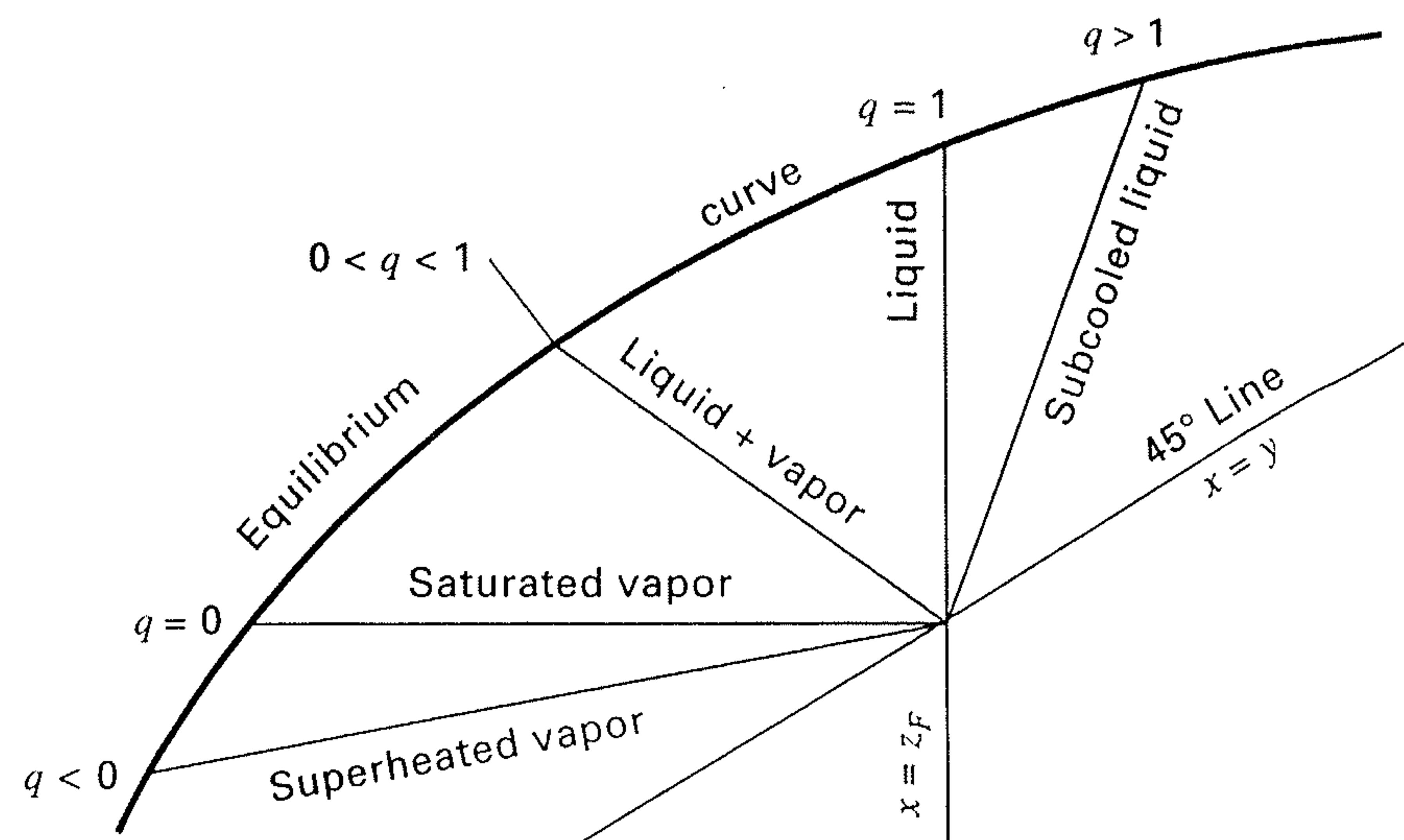
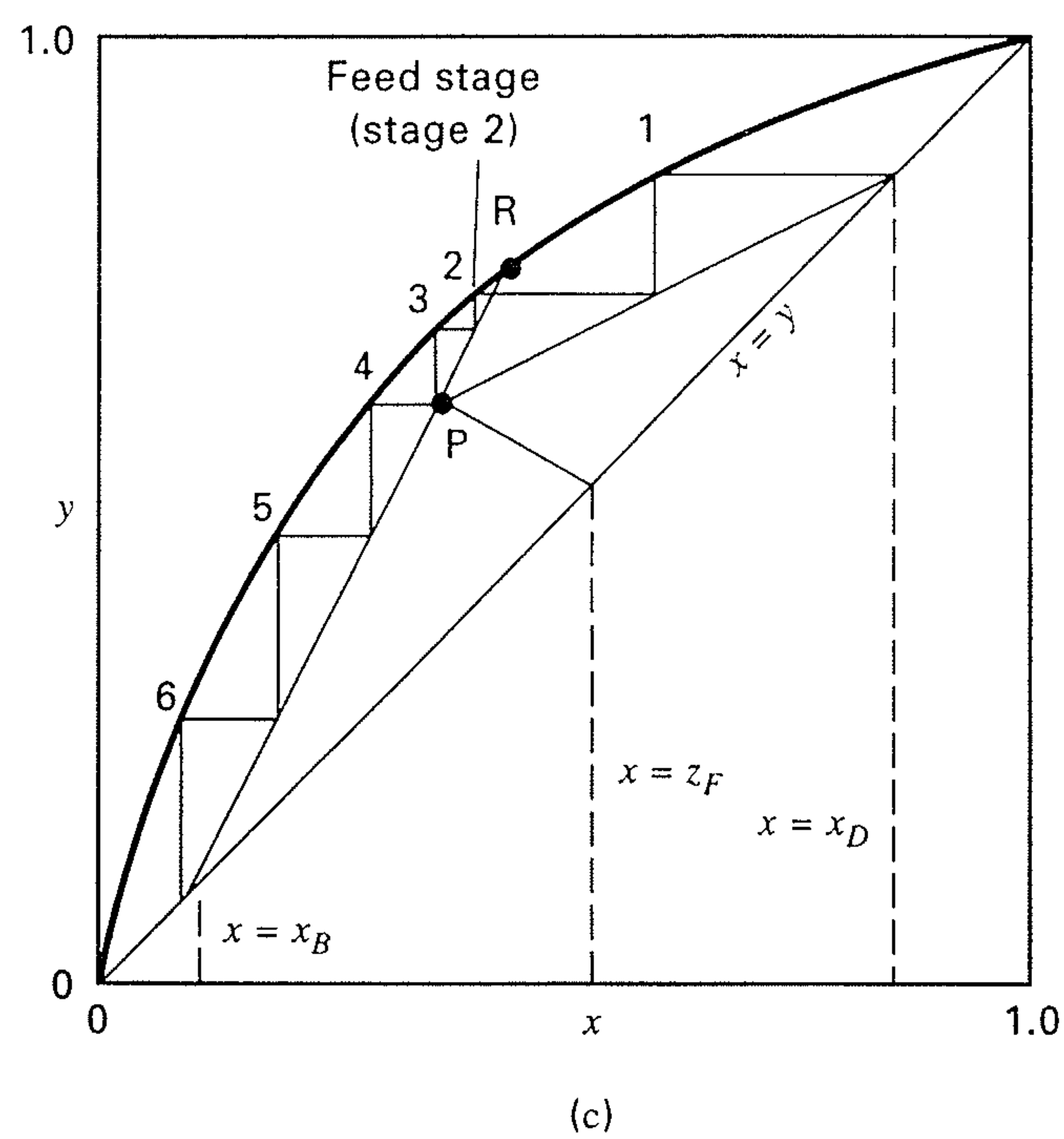
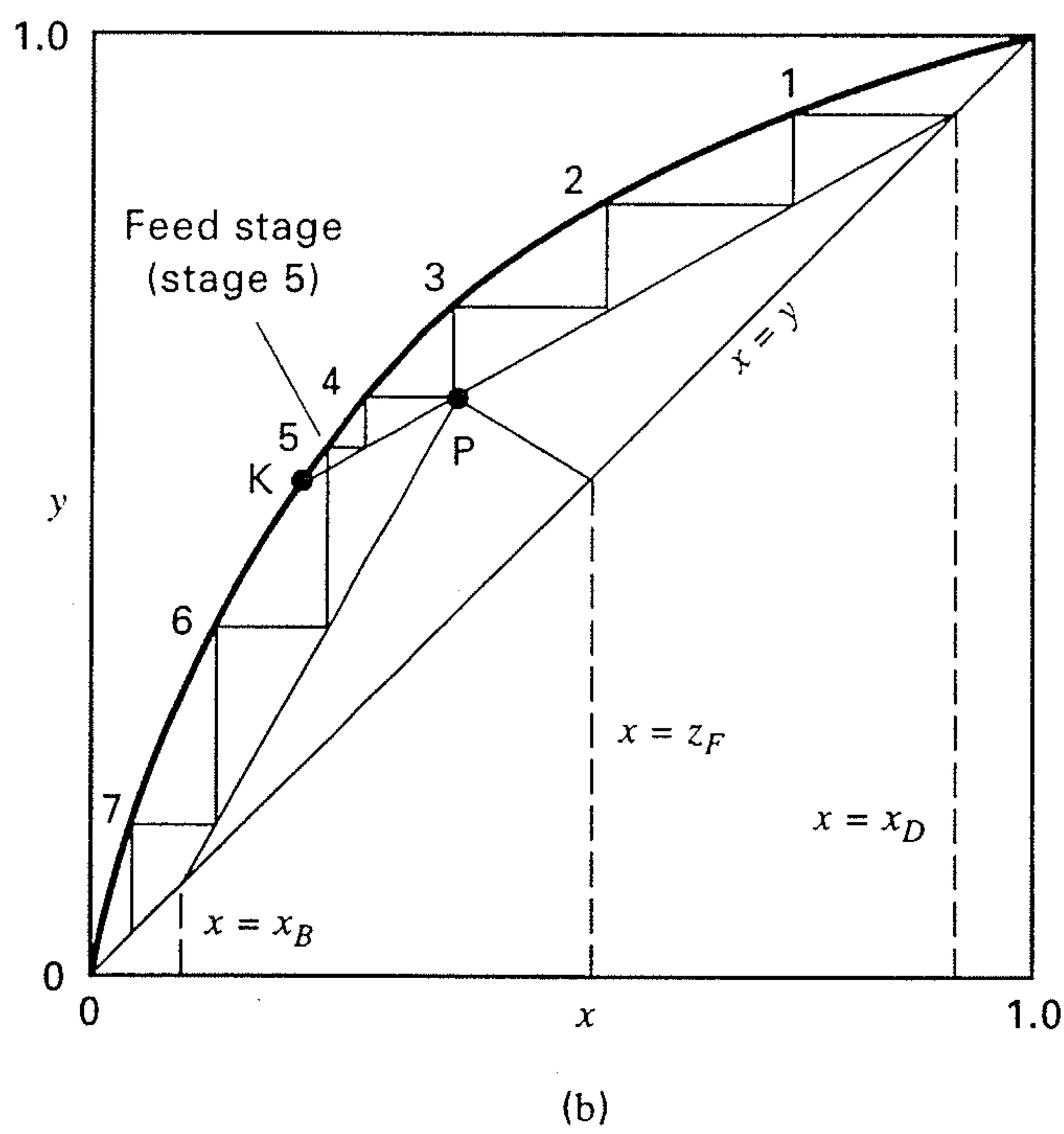
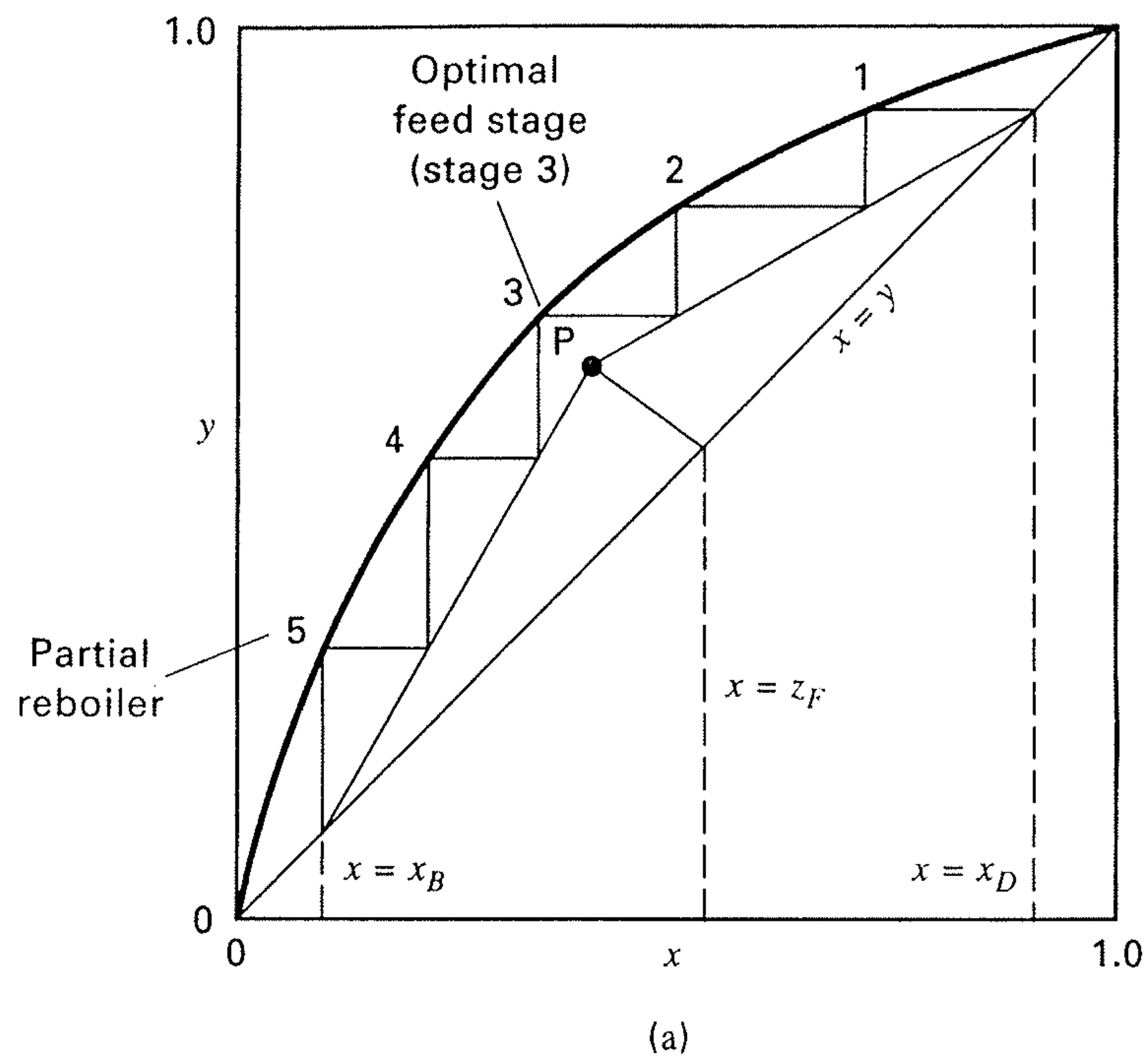


Figure 7.8 Effect of thermal condition of feed on slope of  $q$ -line.

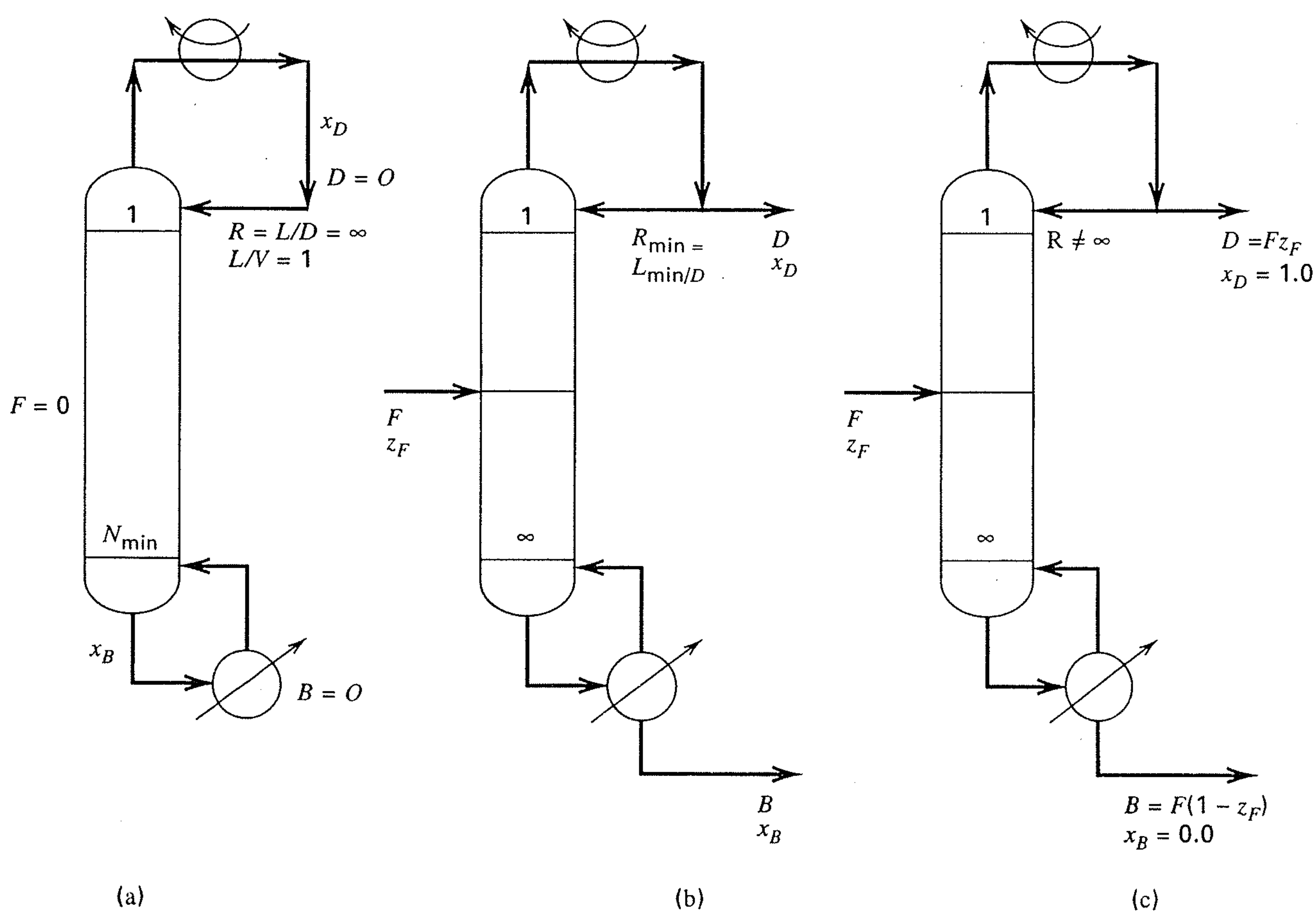
then from the bottom up, as described above, until a point of merger is found for the feed stage. Alternatively, the stages can be stepped off from the bottom all the way to the top, or vice versa. Hardly ever will an integer number of stages result, but rather a fractional stage will appear near the middle, at the top, or at the bottom. Usually the staircase is stepped off from the top and continued all the way to the bottom, starting from the point  $(y = x_D, x = x_D)$  on the 45° line, as shown in Figure 7.9 for the case of a partially vaporized feed. In that figure, point P is the intersection of the  $q$ -line with the two operating lines. The transfer point for stepping off stages between the rectifying-section operating line and the equilibrium curve to stepping off stages between the stripping-section operating line and the equilibrium curve occurs at the feed stage. In Figure 7.9a, the feed stage is stage 3 from the top and a fortuitous total of exactly five stages is required, where the last stage is the partial reboiler. In Figure 7.9b the feed stage is stage 5 and a total of about 6.4 stages is required. In Figure 7.9c, the feed stage is stage 2 and a total of about 5.9 stages is required. In Figure 7.9b, the stepping off of stages in the rectifying section can be continued indefinitely, finally approaching, but never reaching, point K. In Figure 7.9c, if the stepping off of stages had started from the partial reboiler at the point  $(y = x_B, x = x_B)$  and proceeded upward, the staircase in the stripping section could have been continued indefinitely, finally approaching, but never reaching, point R. In Figure 7.9, it is seen that the smallest number of total stages occurs when the transfer is made at the first opportunity after a horizontal line of the staircase passes over point P, as in Figure 7.9a. This feed-stage location is optimal.

### Limiting Conditions

For a given specification (Table 7.2), a reflux ratio can be selected anywhere from the minimum,  $R_{\min}$ , to an infinite value (total reflux) where all of the overhead vapor is condensed and returned to the top stage (thus, no distillate is withdrawn). As shown in Figure 7.10b, the minimum reflux corresponds to the need for an infinite number of stages, while in Figure 7.10a the infinite reflux ratio corresponds to



**Figure 7.9** Optimal and nonoptimal locations of feed stage: (a) optimal feed-stage location; (b) feed-stage location below optimal stage; (c) feed-stage location above optimal stage.



**Figure 7.10** Limiting conditions for distillation: (a) total reflux, minimum stages; (b) minimum reflux, infinite stages; (c) perfect separation for nonazeotropic system.

the minimum number of equilibrium stages. The McCabe–Thiele graphical method can quickly determine the two limits,  $N_{\min}$  and  $R_{\min}$ . Then, for a practical operation,  $N_{\min} < N < \infty$  and  $R_{\min} < R < \infty$ .

### Minimum Number of Equilibrium Stages

As the reflux ratio is increased, the slope of the rectifying-section operating line, given by (7-7), increases from  $L/V < 1$  to a limiting value of  $L/V = 1$ . Correspondingly, the boilup ratio increases and the slope of the stripping section operating line, given by (7-12), decreases from  $\bar{L}/\bar{V} > 1$  to a limiting value of  $\bar{L}/\bar{V} = 1$ . Thus, at this limiting condition, both the rectifying and stripping operating lines coincide with the 45° line and neither the feed composition,  $z_F$ , nor the  $q$ -line influences the staircase construction. This is total reflux because when  $L = V$ ,  $D = B = 0$ , and the total condensed overhead is returned to the column as reflux. Furthermore, all liquid leaving the bottom stage is vaporized and returned as boilup to the column. If both distillate and bottoms flow rates are zero, the feed to the column is also zero, which is consistent with the lack of influence of the feed condition. It is possible to operate a column at total reflux, and such an operation is convenient for the experimental measurement of tray efficiency because a steady-state operating condition is readily achieved.

A simple example of the McCabe–Thiele construction for this limiting condition is shown in Figure 7.11 for two equilibrium stages. Because the operating lines are located as far away as possible from the equilibrium curve, a minimum number of stages is required.

### Minimum Reflux Ratio

As the reflux ratio decreases from the limiting case of infinity (i.e., total reflux), the intersection of the two operating lines and the  $q$ -line moves away from the 45° line toward the equilibrium curve. The number of equilibrium stages required increases because the operating lines move closer and closer to the equilibrium curve, thus requiring more and

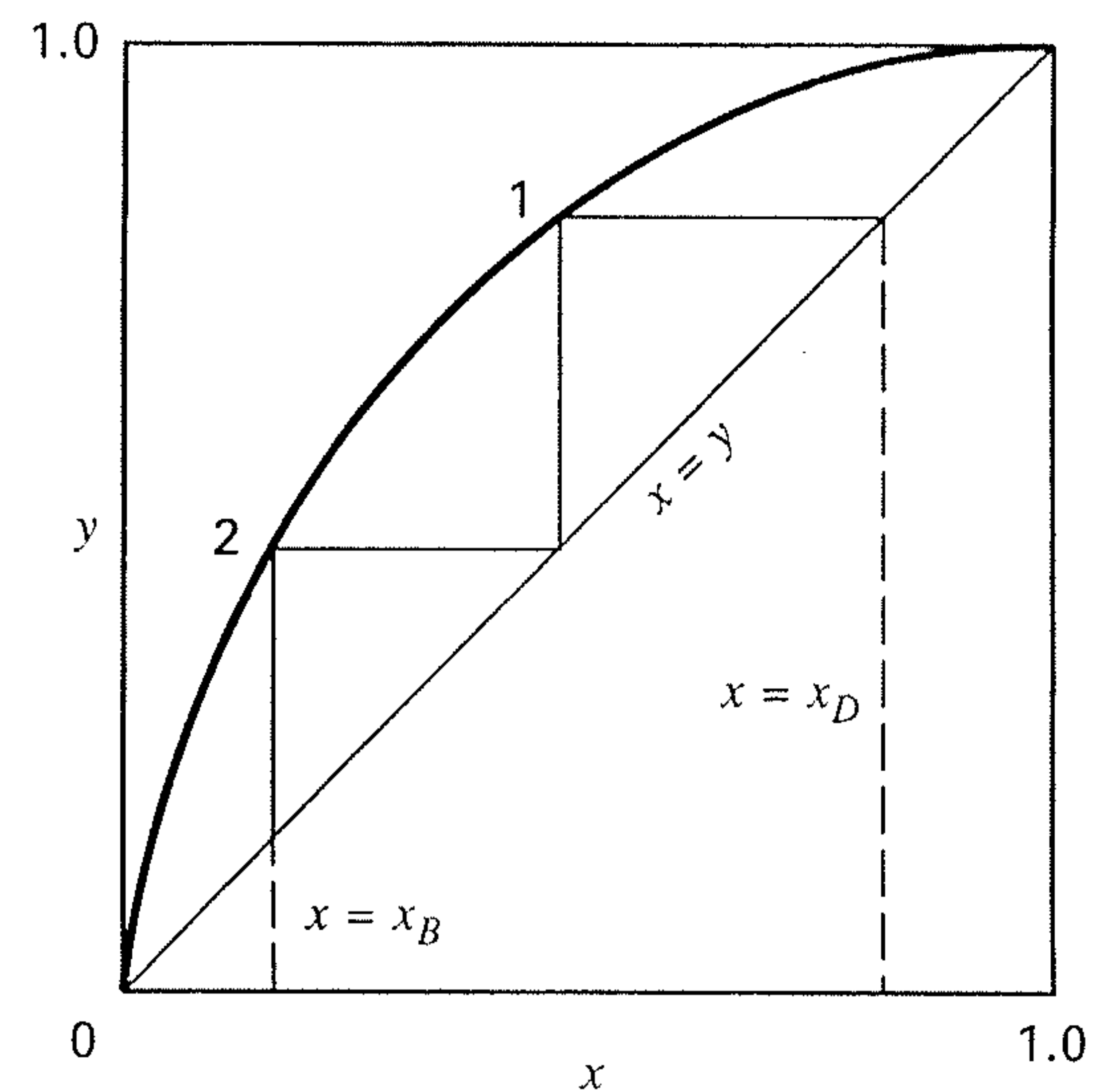


Figure 7.11 Construction for minimum stages at total reflux.

more stairs to move from the top of the column to the bottom. Finally a limiting condition is reached when the point of intersection is on the equilibrium curve, as shown in Figure 7.12. For binary mixtures that are not highly nonideal, the typical case is shown in Figure 7.12a, where the intersection, P, is at the feed stage. To reach that stage from either the rectifying section or the stripping section, an infinite number of stages is required. The point P is called a *pinch point* because the two operating lines each pinch the equilibrium curve.

For a highly nonideal binary system, the pinch point may occur at a stage above or below the feed stage. The former case is illustrated in Figure 7.12b, where the operating line for the rectifying section intersects the equilibrium curve before the feed stage is reached. The slope of this operating line cannot be reduced further because it would then cross over the equilibrium curve and thereby violate the second law of thermodynamics because of a reversal in the direction of mass transfer. This would require spontaneous mass transfer from a region of low concentration to a region of high concentration. This is similar to a second-law violation by a temperature crossover in a heat exchanger. Now, the pinch point occurs entirely in the rectifying section, where an infinite number of stages exists; the stripping section contains a finite number of stages.

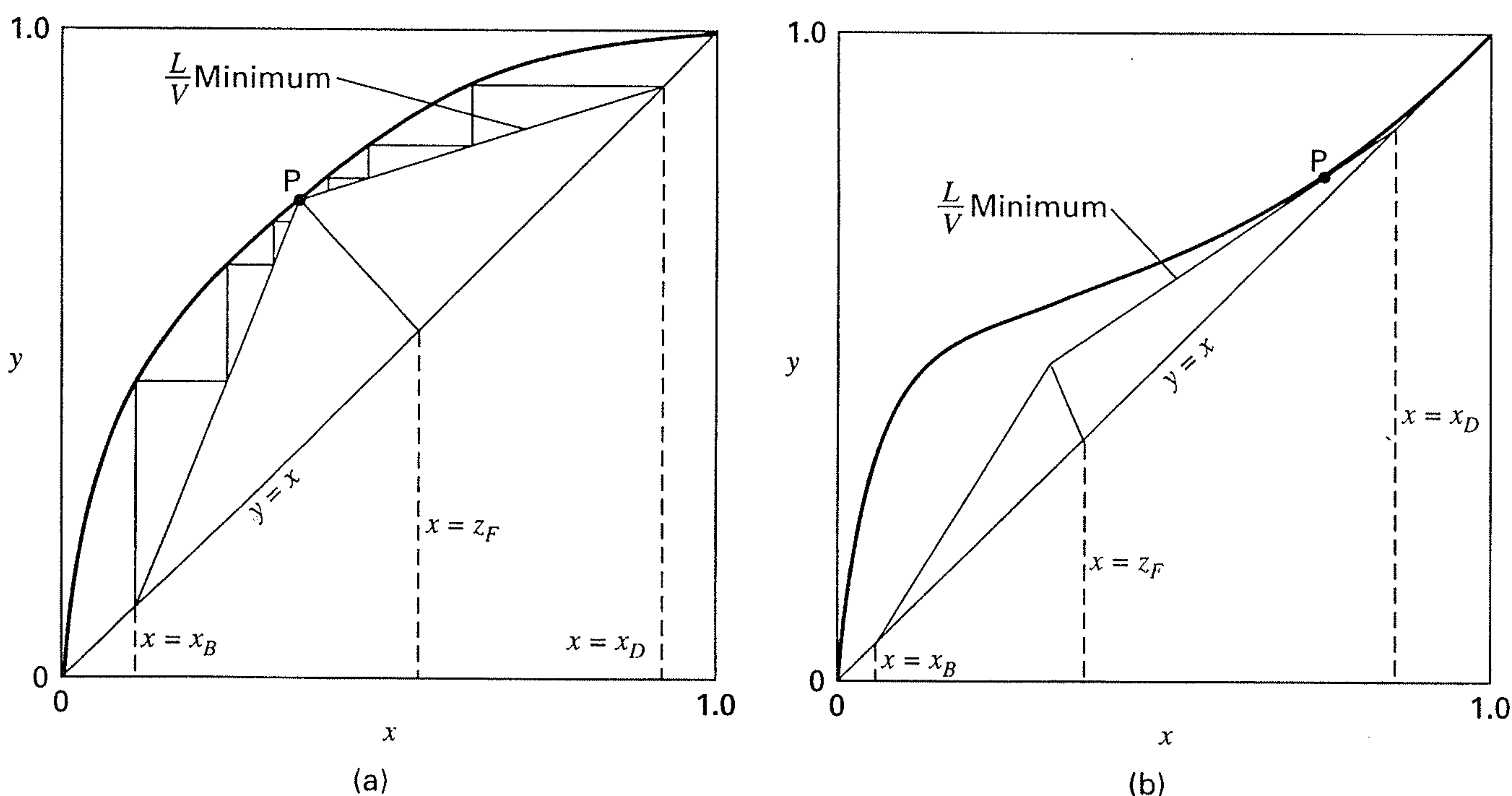


Figure 7.12 Construction for minimum reflux at infinite stages: (a) typical ideal or near-ideal system, pinch point at the feed stage; (b) typical nonideal system, pinch point above the feed stage.

From the slope of the limiting operating line for the rectifying section, the minimum reflux ratio can be determined. From (7-7), the minimum feasible slope is

$$(L/V)_{\min} = R_{\min}/(R_{\min} + 1)$$

or

$$R_{\min} = (L/V)_{\min}/[1 - (L/V)_{\min}] \quad (7-27)$$

Alternatively, the limiting condition of infinite stages corresponds to a minimum boilup ratio for  $(\bar{L}/\bar{V})_{\max}$ . From (7-12),

$$(V_B)_{\min} = 1/[(\bar{L}/\bar{V})_{\max} - 1] \quad (7-28)$$

### Perfect Separation

A third limiting condition of interest involves the degree of separation. As a perfect split ( $x_D = 1$ ,  $x_B = 0$ ) is approached, for a reflux ratio at or greater than the minimum value, the number of stages required near the top and near the bottom

### EXAMPLE 7.1

A trayed tower is to be designed to continuously distill 450 lbmol/h (204 kmol/h) of a binary mixture of 60 mol% benzene and 40 mol% toluene. A liquid distillate and a liquid bottoms product of 95 mol% and 5 mol% benzene, respectively, are to be produced. The feed is preheated so that it enters the column with a molar percent vaporization equal to the distillate-to-feed ratio. Use the McCabe–Thiele method to compute the following, assuming a uniform pressure of 1 atm (101.3 kPa) throughout the column: (a) Minimum number of theoretical stages,  $N_{\min}$ ; (b) Minimum reflux ratio,  $R_{\min}$ ; and (c) Number of equilibrium stages  $N$ , for a reflux-to-minimum reflux ratio,  $R/R_{\min}$ , of 1.3 and the optimal location of the feed stage.

### SOLUTION

Calculate  $D$  and  $B$ . An overall material balance on benzene gives

$$0.60(450) = 0.95D + 0.05B \quad (1)$$

$$\text{A total balance gives } 450 = D + B \quad (2)$$

Combining (1) and (2) to eliminate  $B$ , followed by solving the resulting equation for  $D$  and (2) for  $B$  gives  $D = 275$  lbmol/h,  $B = 175$  lbmol/h, and  $D/F = 0.611$

Calculate the slope of the  $q$ -line:

$V_F/F = D/F$  for this example = 0.611 and  $q$  for a partially vaporized feed is

$$\frac{L_F}{F} = \frac{(F - V_F)}{F} = 1 - \frac{V_F}{F} = 0.389$$

From (7-26),

$$\text{the slope of the } q\text{-line is } \frac{q}{q-1} = \frac{0.389}{0.389-1} = -0.637$$

- (a) In Figure 7.13, where  $y$  and  $x$  refer to benzene, the more-volatile component, with  $x_D = 0.95$  and  $x_B = 0.05$ , the number of minimum equilibrium stages is stepped off from the top between the equilibrium curve and the 45° line, giving  $N_{\min} = 6.7$ .
- (b) In Figure 7.14, a  $q$ -line is drawn that has a slope of  $-0.637$  and passes through the feed composition ( $z_F = 0.60$ ) on the 45° line. For the minimum-reflux condition, an operating line for the rectifying section passes through the point  $x = x_D = 0.95$  on

of the column increases rapidly and without limit until pinches are encountered at  $x_D = 1$  and  $x_B = 0$ . Thus, a perfect separation of a binary mixture that does not form an azeotrope requires an infinite number of stages in both sections of the column. However, this is not the case for the reflux ratio. In Figure 7.12a, as  $x_D$  is moved from, say, 0.90 toward 1.0, the slope of the operating line at first increases, but in the range of  $x_D$  from 0.99 to 1.0 the slope changes only slightly. Furthermore, the value of the slope, and therefore the value of  $R$ , is finite for a perfect separation. For example, if the feed is a saturated liquid, application of (7-4) and (7-7) gives the following equation for the minimum reflux of a perfect binary separation:

$$R_{\min} = \frac{1}{z_F(\alpha - 1)} \quad (7-29)$$

where the relative volatility,  $\alpha$ , is evaluated at the feed condition.

the 45° line and through the point of intersection of the  $q$ -line and the equilibrium curve ( $y = 0.684$ ,  $x = 0.465$ ). The slope of this operating line is 0.55, which from (7-9) equals  $R/(R + 1)$ . Therefore,  $R_{\min} = 1.22$ .

(c) The operating reflux ratio is  $1.3R_{\min} = 1.3(1.22) = 1.59$

From (7-9), the slope of the operating line for the rectifying section is

$$\frac{R}{R+1} = \frac{1.59}{1.59+1} = 0.614$$

The construction for the resulting two operating lines, together with the  $q$ -line, is shown in Figure 7.15, where the operating line for the stripping section is drawn to pass through the point  $x = x_B = 0.05$  on the 45° line and the point of intersection of the  $q$ -line and the operating line for the stripping section. The number of equilibrium stages is stepped off between, first, the rectifying-section operating line and the equilibrium curve and then the stripping-section operating line and the equilibrium curve, starting from point A

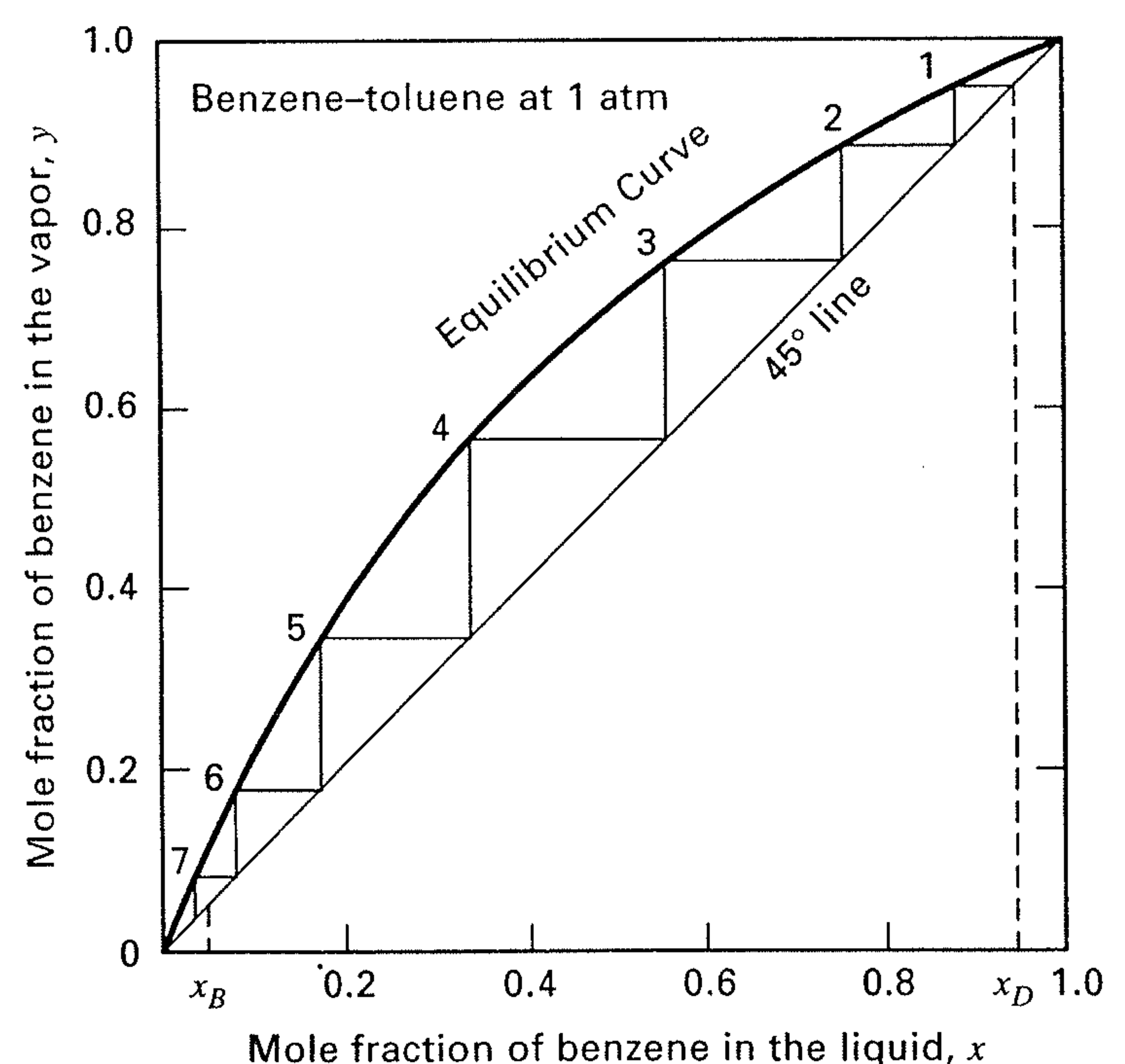


Figure 7.13 Determination of minimum stages for Example 7.1.

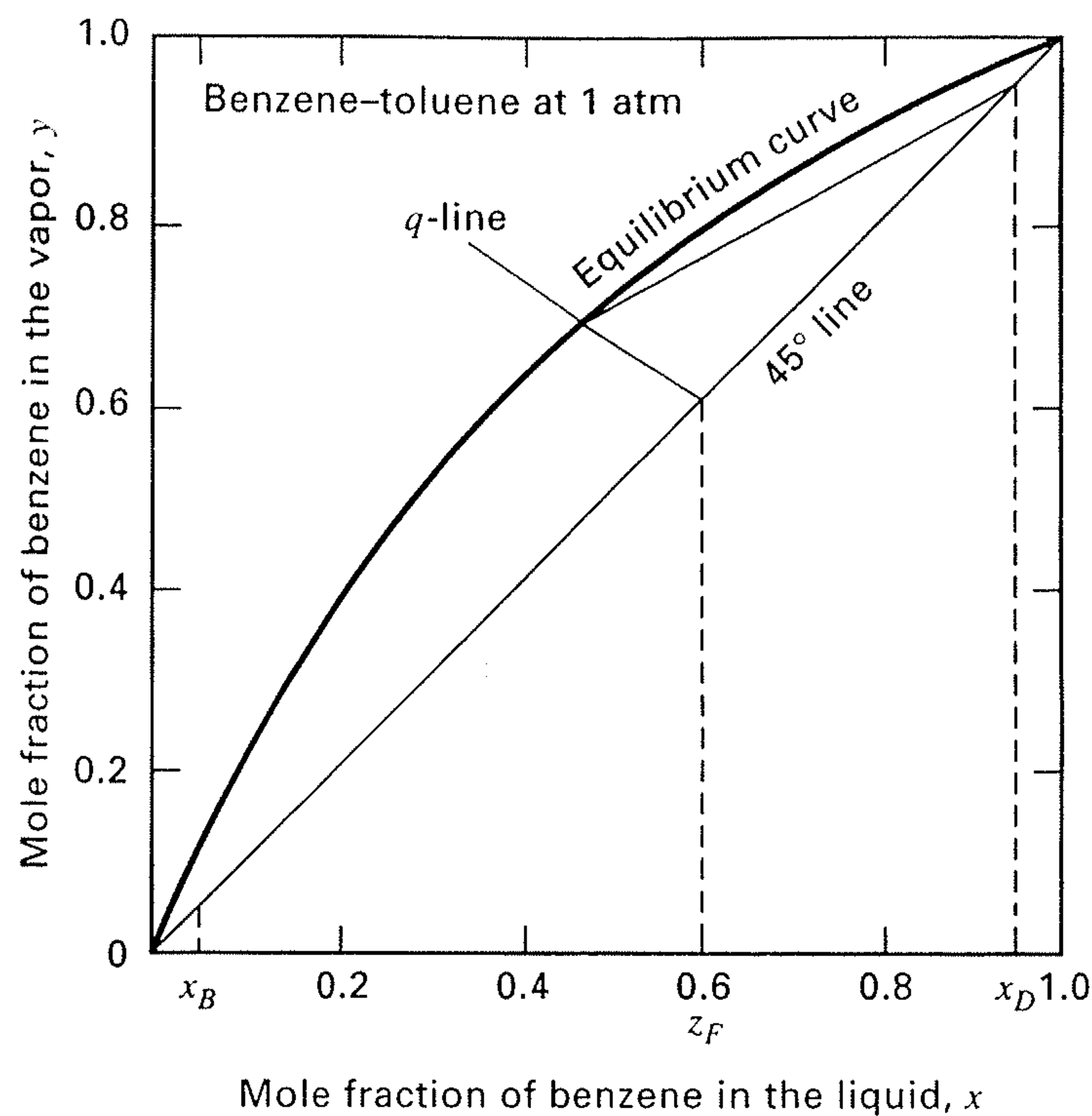


Figure 7.14 Determination of minimum reflux for Example 7.1.

(at  $x = x_D = 0.95$ ) and finishing at point B (to the left of  $x = x_B = 0.05$ ). For the optimal feed-stage location, the transfer from the rectifying-section operating line to the stripping-section operating line takes place at point P. The result is  $N = 13.2$  equilibrium

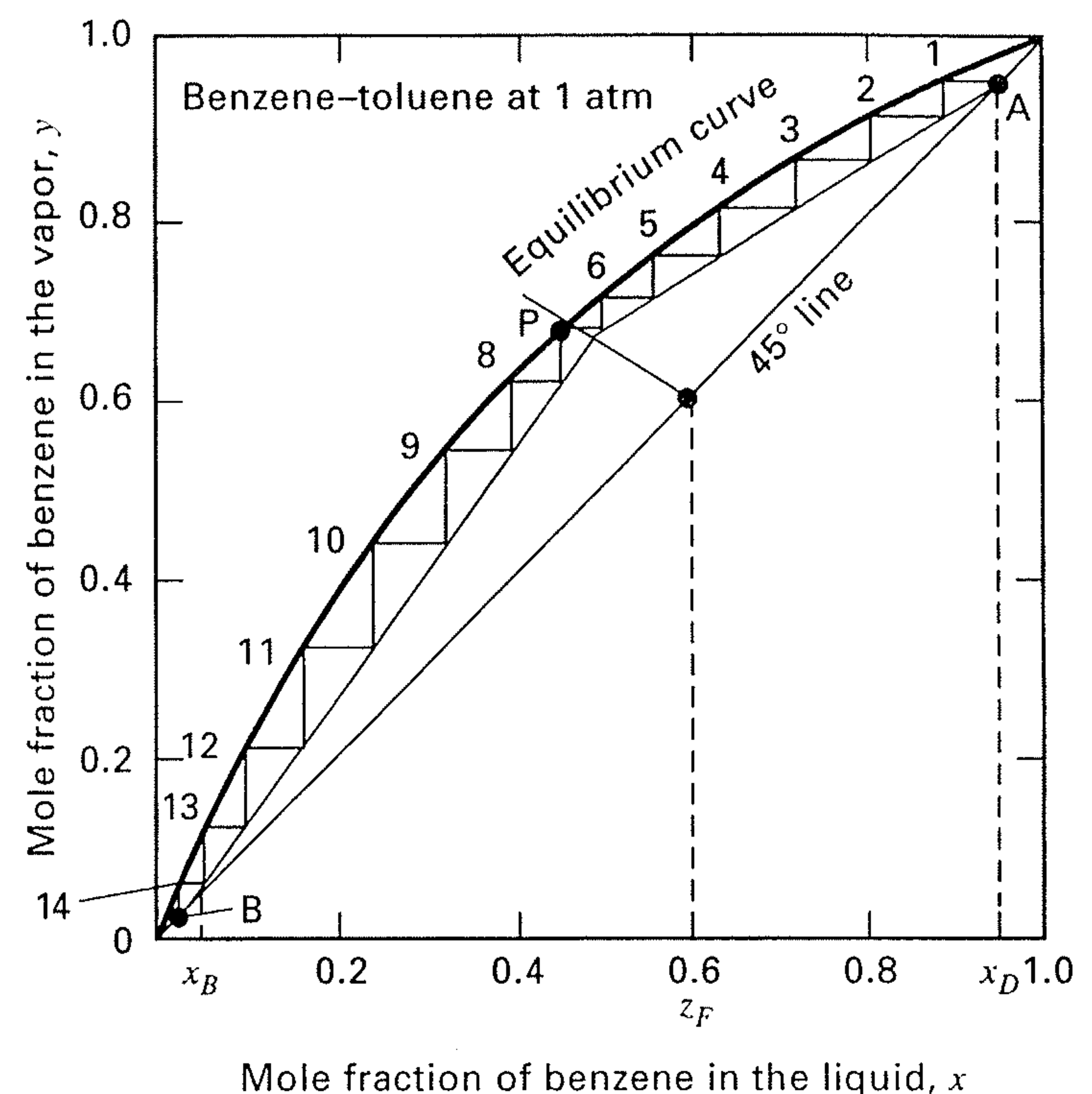


Figure 7.15 Determination of number of equilibrium stages and feed-stage location for Example 7.1.

stages, with stage 7 from the top being the feed stage. Thus, for this example,  $N/N_{\min} = 13.2/6.7 = 1.97$ . The bottom stage is the partial reboiler, leaving 12.2 equilibrium stages contained in the column. If the plate efficiency were 0.8, 16 trays would be needed.

### Column Operating Pressure and Condenser Type

For preliminary design, column operating pressure and condenser type are established by the procedure shown in Figure 7.16, which is formulated to achieve, if possible, a reflux-drum pressure,  $P_D$ , between 0 and 415 psia (2.86 MPa) at a minimum temperature of 120°F (49°C) (corresponding to the use of water as the coolant in the condenser). The pressure and temperature limits are representative only and depend on economic factors. Columns can operate at pressures higher than 415 psia if the critical or convergence

pressure of the mixture is not approached. A condenser pressure drop of 0 to 2 psi (0 to 14 kPa) and an overall, column pressure drop of 5 psi (35 kPa) may be assumed. However, when column tray requirements are known, more refined computations should result in approximately 0.1 psi/tray (0.7 kPa/tray) pressure drop for atmospheric and superatmospheric pressure operation and 0.05 psi/tray (0.35 kPa/tray) pressure drop for vacuum-column operation. Column bottom temperature must not result in bottoms decomposition or correspond to a near-critical condition. Therefore, after

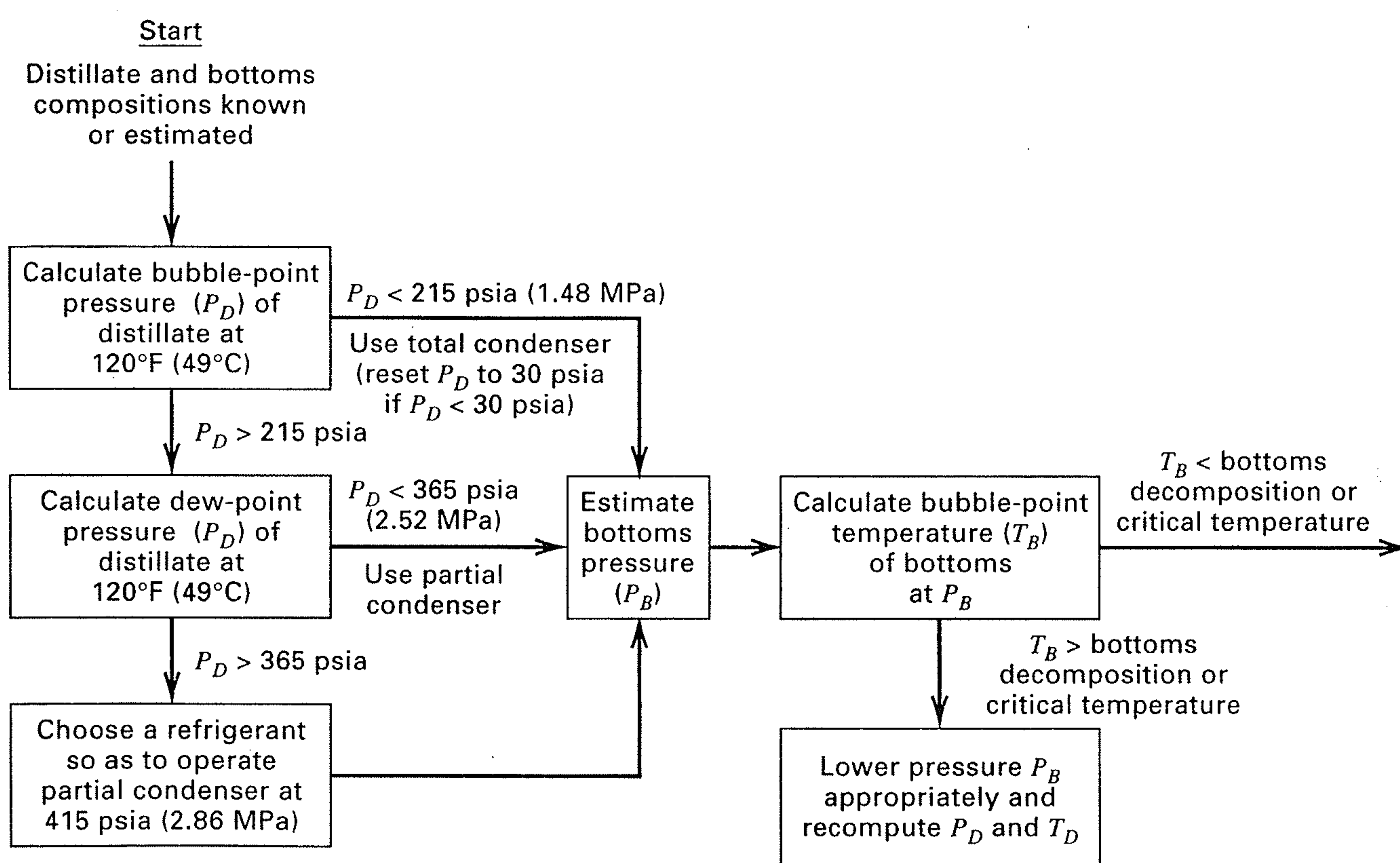
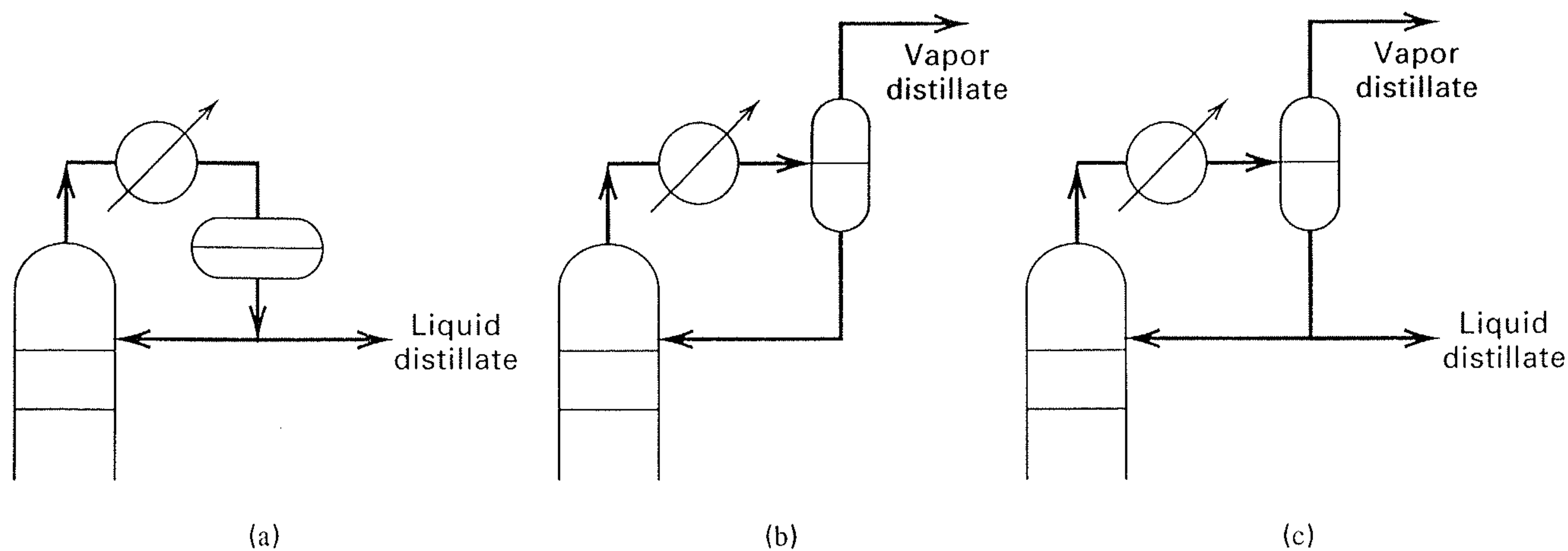


Figure 7.16 Algorithm for establishing distillation-column pressure and condenser type.



**Figure 7.17** Condenser types: (a) total condenser; (b) partial condenser; (c) mixed condenser.

the bottoms pressure is estimated from the pressure in the reflux drum, a bubble-point temperature of the bottoms is computed at the bottoms pressure. If that temperature exceeds the bottoms decomposition or critical temperature, then the bottoms pressure is recomputed at or below the bubble-point decomposition or critical temperature. The pressure in the reflux drum will then be lower and must be recomputed, together with the distillate temperature, from the assumed column and condenser pressure drops. This will result often in vacuum operation. If the recomputed distillate temperature is less than 120°F (49°C), a refrigerant, rather than cooling water, is used for the condenser.

A total condenser is recommended for reflux drum pressures to 215 psia (1.48 MPa). A partial condenser is appropriate from 215 psia to 365 psia (2.52 MPa). However, a partial condenser can be used below 215 psia when a vapor distillate is desired. A mixed condenser can provide both vapor and liquid distillates. The three types of condenser configurations are shown in Figure 7.17. A refrigerant is often used as condenser coolant if pressure tends to exceed 365 psia.

When a partial condenser is specified, the McCabe–Thiele staircase construction for the case of a total condenser must be modified, as will be illustrated in Example 7.2, to account for the fact that the first equilibrium stage, counted down from the top, is now the partial condenser. This is based on the assumption that the liquid reflux leaving the reflux drum is in equilibrium with the vapor distillate.

### Subcooled Reflux

Although most distillation columns are designed so that the reflux is a saturated (bubble-point) liquid, such is not always the case for operating columns. If the condenser type is partial or mixed, the reflux is a saturated liquid unless heat losses cause its temperature to decrease. For a total condenser, however, the operating reflux is often a subcooled liquid at column pressure, particularly if the condenser is not tightly designed and the distillate bubble-point temperature is significantly higher than the inlet cooling-water temperature. If the condenser outlet pressure is lower than the top-tray pressure of the column, the reflux is subcooled for any of the three types of condensers.

When subcooled reflux enters the top tray, its temperature rises and causes vapor entering the tray to condense. The

latent enthalpy of condensation of the vapor provides the sensible enthalpy to heat the subcooled reflux to the bubble point. In that event, the internal reflux ratio within the rectifying section of the column is higher than the external reflux ratio from the reflux drum. The McCabe–Thiele construction should be based on the internal reflux ratio, which can be estimated by the following equation derived from an approximate energy balance around the top tray:

$$R_{\text{internal}} = R \left( 1 + \frac{C_{P_L} \Delta T_{\text{subcooling}}}{\Delta H^{\text{vap}}} \right) \quad (7-30)$$

where  $C_{P_L}$  and  $\Delta H^{\text{vap}}$  are per mole and  $\Delta T_{\text{subcooling}}$  is the degrees of subcooling. The internal reflux ratio replaces  $R$ , the external reflux ratio, in (7-9). If a correction is not made for subcooled reflux, the calculated number of equilibrium stages is somewhat more than required.

### EXAMPLE 7.2

One thousand kmol/h of a feed containing 30 mol% *n*-hexane and 70% *n*-octane is to be distilled in a column consisting of a partial reboiler, one equilibrium (theoretical) plate, and a partial condenser, all operating at 1 atm (101.3 kPa). Thus, hexane is the light key and octane is the heavy key. The feed, a bubble-point liquid, is fed to the reboiler, from which a liquid bottoms product is continuously withdrawn. Bubble-point reflux is returned from the partial condenser to the plate. The vapor distillate, in equilibrium with the reflux, contains 80 mol% hexane, and the reflux ratio,  $L/D$ , is 2. Assume that the partial reboiler, plate, and partial condenser each function as equilibrium stages.

- Using the McCabe–Thiele method, calculate the bottoms composition and kmol/h of distillate produced.
- If the relative volatility  $\alpha$  is assumed constant at a value of 5 over the composition range (the relative volatility actually varies from approximately 4.3 at the reboiler to 6.0 at the condenser), calculate the bottoms composition analytically.

### SOLUTION

First determine whether the problem is completely specified. From Table 5.4c, we have  $N_D = C + 2N + 6$  degrees of freedom, where  $N$  includes the partial reboiler and the stages in the column, but not the partial condenser. With  $N = 2$  and  $C = 2$ ,  $N_D = 12$ . Specified in

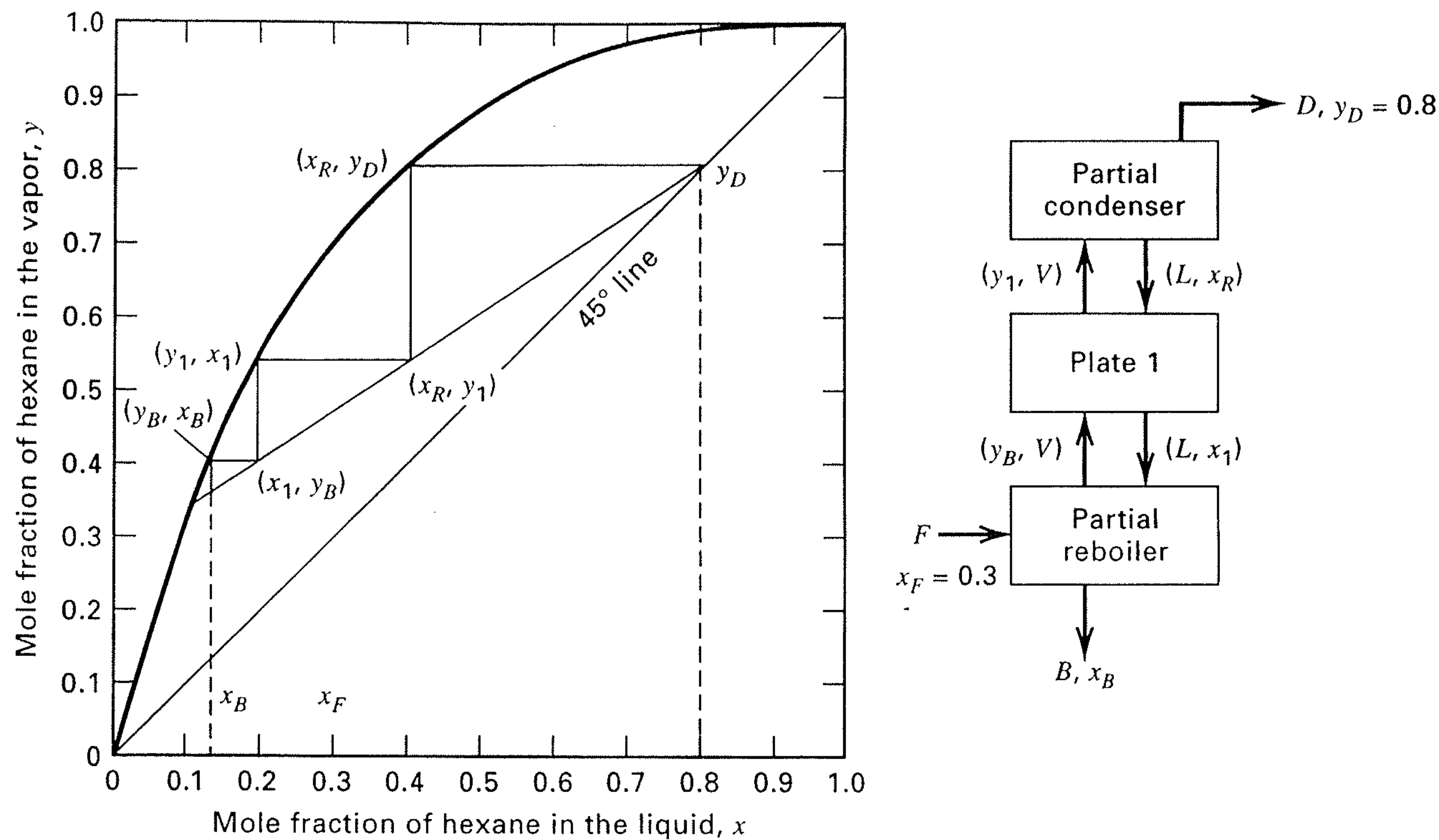


Figure 7.18 Solution to Example 7.2.

this problem are

Feed-stream variables	4
Plate and reboiler pressures	2
Condenser pressure	1
$Q (=0)$ for plate	1
Number of stages	1
Feed-stage location	1
Reflux ratio, $L/D$	1
Distillate composition	$\frac{1}{1}$
Total	12

Thus, the problem is fully specified and can be solved.

(a) *Graphical solution.* A diagram of the separator is given in Figure 7.18 as is the McCabe–Thiele graphical solution, which is constructed in the following manner.

1. The point  $y_D = 0.8$  at the partial condenser is located on the  $x = y$  line.
2. Conditions in the condenser are fixed because  $x_R$  (reflux composition) is in equilibrium with  $y_D$ . Hence, the point  $(x_R, y_D)$  is located on the equilibrium curve.
3. Noting that  $(L/V) = 1 - 1/[1 + (L/D)] = 2/3$ , the operating line with slope  $L/V = 2/3$  is drawn through the point  $y_D = 0.8$  on the  $45^\circ$  line until it intersects the equilibrium curve. Because the feed is introduced into the partial reboiler, there is no stripping section.
4. Three theoretical stages (partial condenser, plate 1, and partial reboiler) are stepped off and the bottoms composition  $x_B = 0.135$  is read.

The amount of distillate is determined from overall material balances. For hexane,  $z_F F = y_D D + x_B B$ . Therefore,  $(0.3)(1,000) = (0.8)D + (0.135)B$ . For the total flow,  $B = 1,000 - D$ . Solving these two equations simultaneously,  $D = 248$  kmol/h.

(b) *Analytical solution.* For constant  $\alpha$ , equilibrium liquid compositions for the light key, in terms of  $\alpha$  and  $y$  are given by a rearrangement of (7-3):

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

where  $\alpha$  is assumed constant at a value of 5.

The steps in the solution are as follows:

1. The liquid leaving the partial condenser at  $x_R$  is calculated from (1), for  $y = y_D = 0.8$ :

$$x_R = \frac{0.8}{0.8 + 5(1 - 0.8)} = 0.44$$

2. Then  $y_1$  is determined by a material balance about the partial condenser:

$$V y_1 = D y_D + L x_R \quad \text{with } D/V = 1/3 \quad \text{and } L/V = 2/3$$

$$y_1 = (1/3)(0.8) + (2/3)(0.44) = 0.56$$

3. From (1), for plate 1,  $x_1 = \frac{0.56}{0.56 + 5(1 - 0.56)} = 0.203$

4. By material balance around plate 1 and the partial condenser,

$$V y_B = D y_D + L x_1$$

$$\text{and } y_B = (1/3)(0.8) + (2/3)(0.203) = 0.402$$

5. From (1), for the partial reboiler,

$$x_B = \frac{0.402}{0.402 + 5(1 - 0.402)} = 0.119.$$

By approximating the equilibrium curve with  $\alpha = 5$ , an answer of 0.119 is obtained rather than 0.135 for  $x_B$  obtained in part (a). Note that for a larger number of theoretical plates, part (b) can be readily computed with a spreadsheet program.

### EXAMPLE 7.3

Consider Example 7.2. (a) Solve it graphically, assuming that the feed is introduced on plate 1, rather than into the reboiler. (b) Determine the minimum number of stages required to carry out the separation. (c) Determine the minimum reflux ratio.

### SOLUTION

(a) The flowsheet and solution given in Figure 7.19 are obtained as follows.

1. The point  $x_R, y_D$  is located on the equilibrium line.
2. The operating line for the enriching section is drawn through the point  $y = x = 0.8$ , with a slope of  $L/V = 2/3$ .



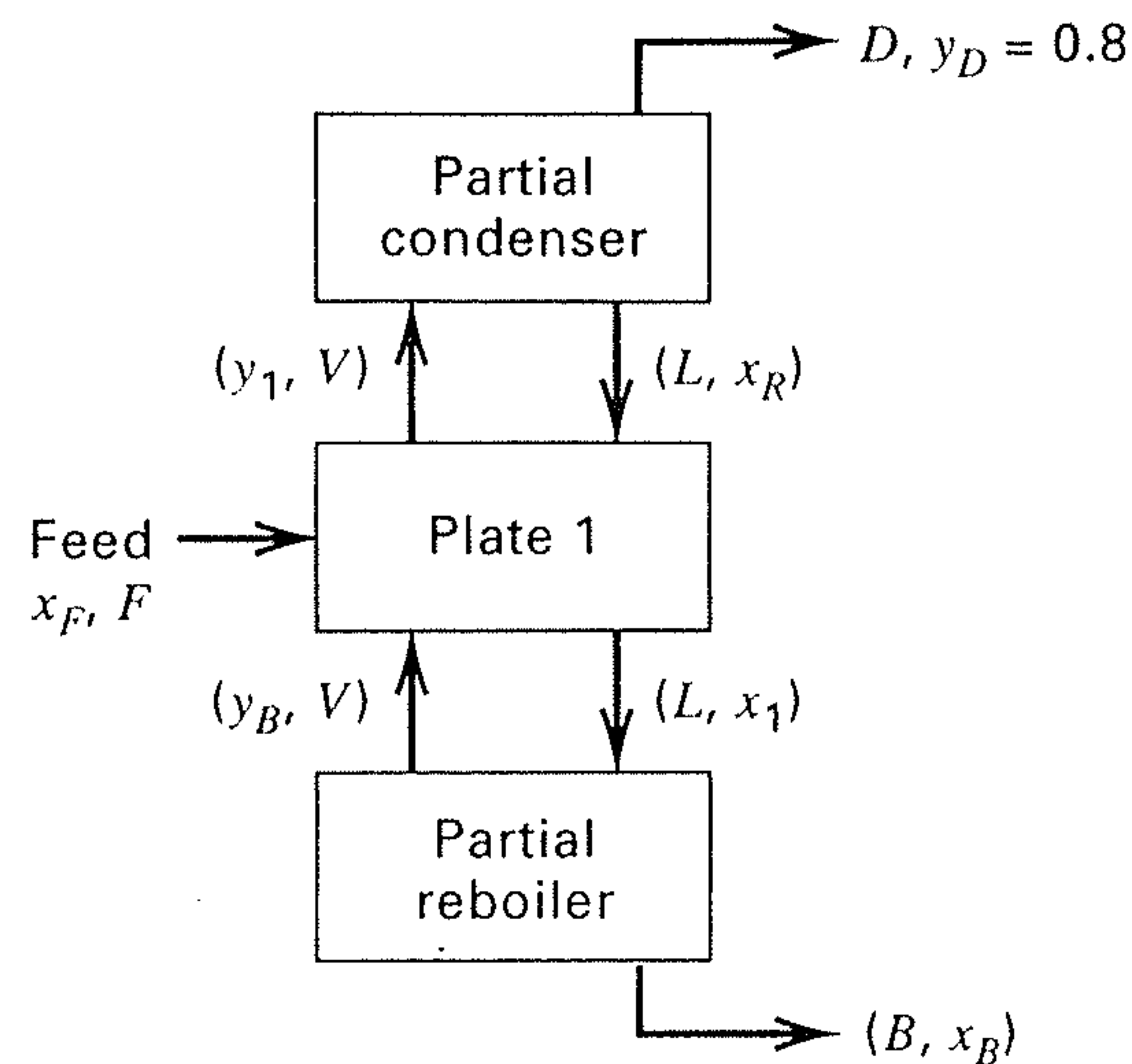
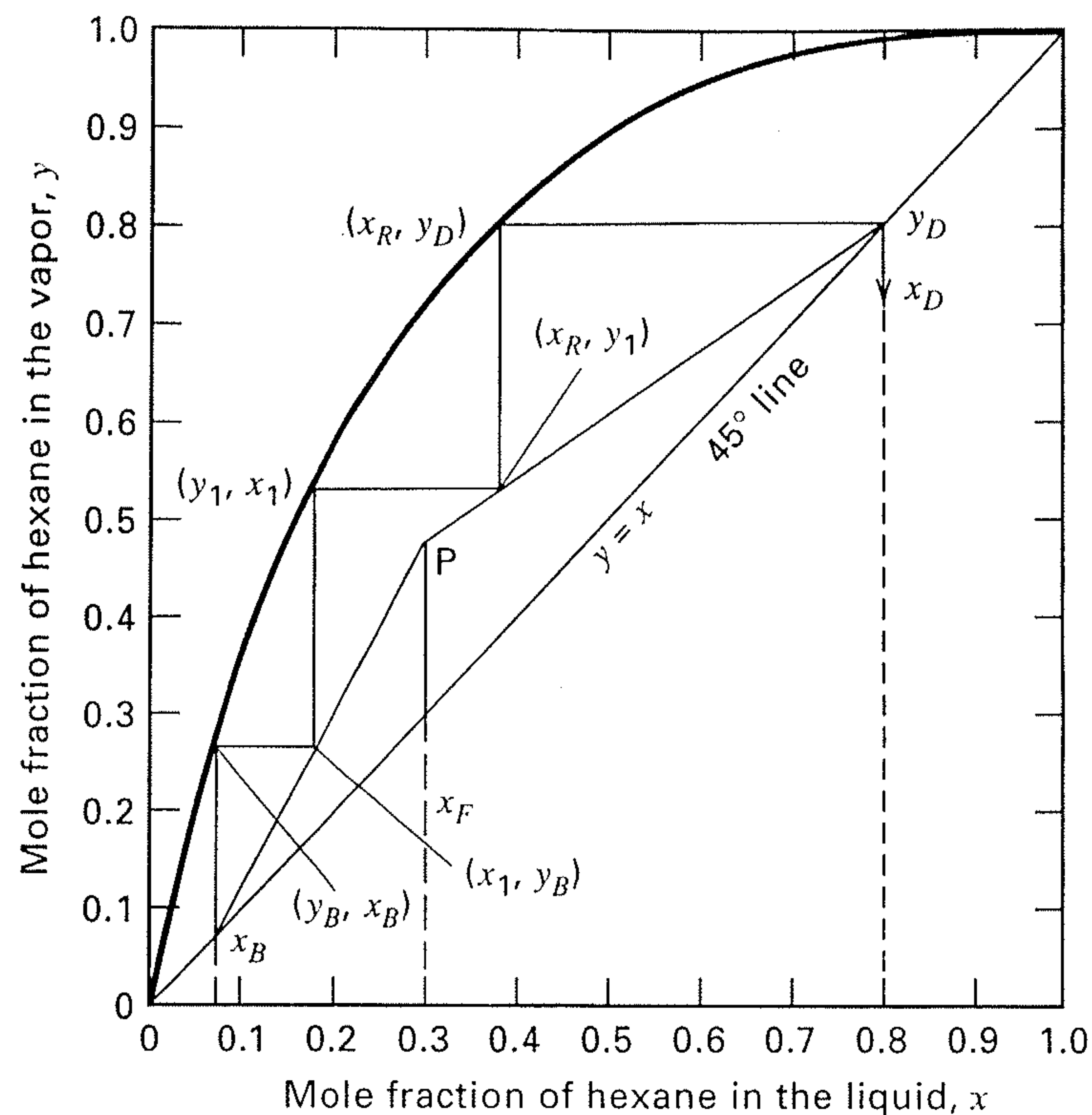


Figure 7.19 Solution to Example 7.3.

3. The intersection of the  $q$ -line,  $x_F = 0.3$  (which, for a saturated liquid, is a vertical line), with the enriching-section operating line is located at point P. The stripping-section operating line must also pass through this point, but its slope and the point  $x_B$  are not known initially.
4. The slope of the stripping-section operating line is found by trial and error to give three equilibrium contacts in the column, with the middle stage involved in the switch from one operating line to the other. If the middle stage is the optimal feed-stage location, the result is  $x_B = 0.07$ , as shown in Figure 7.19. The amount of distillate is obtained from the combined total and hexane overall material balances to give  $(0.3)(1,000) = (0.8D) + 0.07(1,000 - D)$ . Solving,  $D = 315$  kmol/h.

Comparing this result to that obtained in Example 7.2, we find that the bottoms purity and distillate yield are improved by introduction of the feed to plate 1, rather than to the reboiler. This improvement could have been anticipated if the  $q$ -line had been constructed in Figure 7.18. That is, the partial reboiler is not the optimal feed-stage location.

- (b) The construction corresponding to total reflux ( $L/V = 1$ , no products, no feed, minimum equilibrium stages) is shown

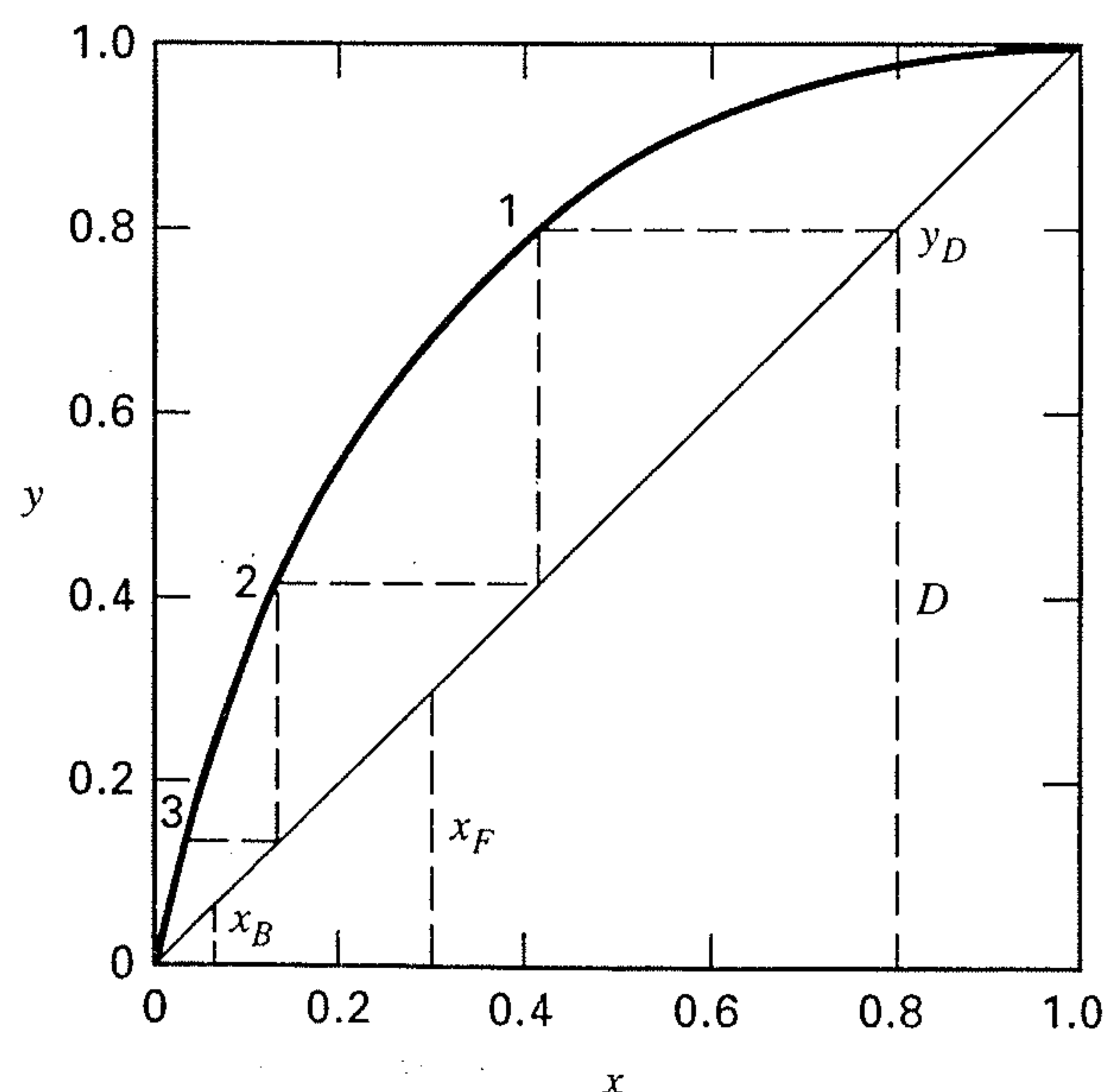


Figure 7.20 Solution for total reflux in Example 7.3.

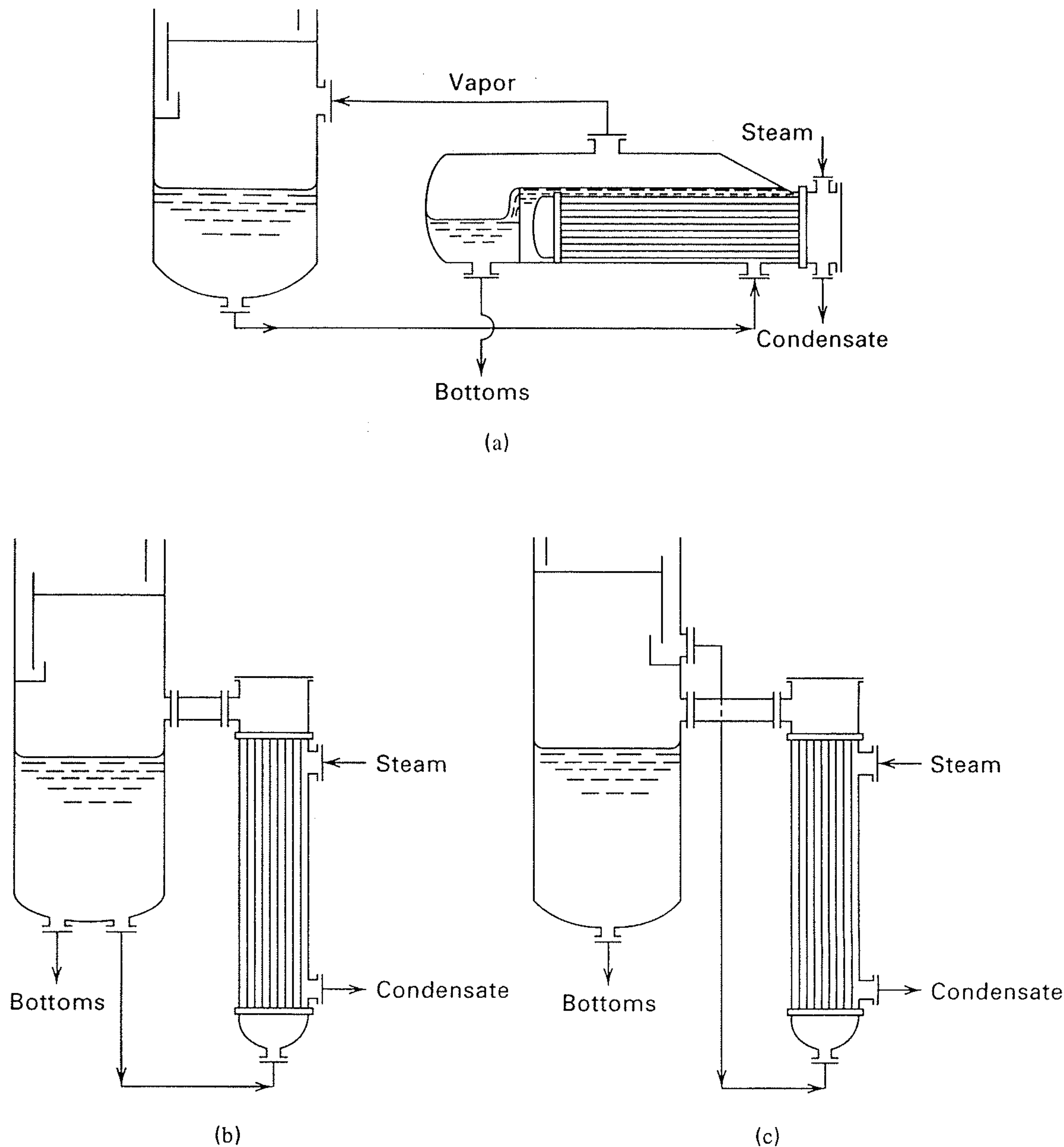
in Figure 7.20. Slightly more than two stages are required for an  $x_B$  of 0.07, compared to the three stages previously required.

- (c) To determine the minimum-reflux ratio, the vertical  $q$ -line in Figure 7.19 is extended from point P until the equilibrium curve is intersected, which is determined to be the point (0.71, 0.3). The slope,  $(L/V)_{\min}$  of the operating line for the rectifying section, which connects this point to the point (0.8, 0.8) on the 45° line is 0.18. Thus  $(L/D)_{\min} = (L/V_{\min}) / [1 - (L/V_{\min})] = 0.22$ . This is considerably less than the  $L/D = 2$  specified.

## Reboiler Type

Different types of reboilers are used to provide boilup vapor to the stripping section of a distillation column. For small laboratory and pilot-plant-size columns, the reboiler consists of a reservoir of liquid located just below the bottom plate to which heat is supplied from (1) a jacket or mantle that is heated by an electrical current or by condensing steam, or (2) tubes that pass through the liquid reservoir carrying condensing steam. Both of these types of reboilers have limited heat-transfer surface and are not suitable for industrial applications.

For plant-size distillation columns, the reboiler is usually an external heat exchanger, as shown in Figure 7.21, of either the kettle or vertical thermosyphon type. Both can provide the amount of heat-transfer surface required for large installations. In the former case, liquid leaving the sump (reservoir) at the bottom of the column enters the kettle, where it is partially vaporized by the transfer of heat from tubes carrying condensing steam or some other heating medium. The bottoms product liquid leaving the reboiler is assumed to be in equilibrium with the vapor returning to the bottom tray of the column. Thus the kettle reboiler is a partial reboiler equivalent to one equilibrium stage. The kettle



**Figure 7.21** Reboilers for plant-size distillation columns: (a) kettle-type reboiler; (b) vertical thermosyphon-type reboiler, reboiler liquid withdrawn from bottom sump; (c) vertical thermosyphon-type reboiler, reboiler liquid withdrawn from bottom-tray downcomer.

reboiler is sometimes located in the bottom of the column to avoid piping.

The vertical thermosyphon reboiler may be of the type shown in Figure 7.21b or 7.21c. In the former, both the bottoms product and the reboiler feed are withdrawn from the column bottom sump. Circulation through the tubes of the reboiler occurs because of the difference in static heads of the supply liquid and the column of partially vaporized fluid flowing through the reboiler tubes. The partial vaporization provides enrichment of the exiting vapor in the more volatile component. However, the exiting liquid is then mixed with liquid leaving the bottom tray, which contains a higher percentage of the more volatile component. The result is that this type of reboiler arrangement provides only a fraction of an equilibrium stage and it is best to take no credit for it.

A more complex and less-common vertical thermosyphon reboiler is that of Figure 7.21c, where the reboiler liquid is withdrawn from the downcomer of the bottom tray. Partially vaporized liquid is returned to the column, where the bottoms product from the bottom sump is withdrawn. This type of reboiler does function as an equilibrium stage.

Kettle reboilers are common, but thermosyphon reboilers are favored when (1) the bottoms product contains thermally sensitive compounds, (2) bottoms pressure is high, (3) only a small  $\Delta T$  is available for heat transfer, and (4) heavy

fouling occurs. Horizontal thermosyphon reboilers are sometimes used in place of the vertical types when only small static heads are needed for circulation, surface-area requirement is very large, and/or when frequent cleaning of the tubes is anticipated. A pump may be added for either thermosyphon type to improve circulation. Liquid residence time in the column bottom sump should be at least 1 minute and perhaps as much as 5 minutes or more.

### Condenser and Reboiler Duties

Following the determination of the feed condition, reflux ratio, and number of theoretical stages by the McCabe–Thiele method, estimates of the heat duties of the condenser and reboiler are made. An energy balance for the entire column gives

$$Fh_F + Q_R = Dh_D + Bh_B + Q_C + Q_{\text{loss}} \quad (7-31)$$

Except for small and/or uninsulated distillation equipment,  $Q_{\text{loss}}$  is negligible and can be ignored. We can approximate the energy balance of (7-31) by applying the assumptions of the McCabe–Thiele method. An energy balance for a total condenser is

$$Q_C = D(R + 1) \Delta H^{\text{vap}} \quad (7-32)$$

where  $\Delta H^{\text{vap}}$  = average molar heat of vaporization of the two components being separated. For a partial condenser,

$$Q_C = DR \Delta H^{\text{vap}} \quad (7-33)$$

For a partial reboiler,

$$Q_R = BV_B \Delta H^{\text{vap}} \quad (7-34)$$

When the feed is at the bubble point and a total condenser is used, (7-16) can be arranged to:

$$BV_B = L + D = D(R + 1) \quad (7-35)$$

Comparing this to (7-34) and (7-32), note that  $Q_R = Q_C$ . When the feed is partially vaporized and a total condenser is used, the heat required by the reboiler is less than the condenser duty and is given by

$$Q_R = Q_C \left[ 1 - \frac{V_F}{D(R + 1)} \right] \quad (7-36)$$

If saturated steam is the heating medium for the reboiler, the steam rate required is given by an energy balance:

$$m_s = \frac{M_s Q_R}{\Delta H_s^{\text{vap}}} \quad (7-37)$$

where

$m_s$  = mass flow rate of steam

$Q_R$  = reboiler duty (rate of heat transfer)

$M_s$  = molecular weight of steam

$\Delta H_s^{\text{vap}}$  = molar enthalpy of vaporization of steam

The cooling water rate for the condenser is

$$m_{\text{cw}} = \frac{Q_C}{C_{P_{\text{H}_2\text{O}}}(T_{\text{out}} - T_{\text{in}})} \quad (7-38)$$

where

$m_{\text{cw}}$  = mass flow rate of cooling water

$Q_C$  = condenser duty (rate of heat transfer)

$C_{P_{\text{H}_2\text{O}}}$  = specific heat of water

$T_{\text{out}}, T_{\text{in}}$  = temperature of cooling water out of and into the condenser, respectively

Because the annual cost of reboiler steam can be an order of magnitude higher than the annual cost of cooling water, the feed to a distillation column is frequently preheated and partially vaporized to reduce  $Q_R$ , in comparison to  $Q_C$ , as indicated by (7-36).

### Feed Preheat

The feed to a distillation column is usually a process feed, an effluent from a reactor, or a liquid product from another separator. The feed pressure must be greater than the pressure in the column at the feed-tray location. If so, any excess feed pressure is dropped across a valve, which may cause the feed to partially vaporize before entering the column; if not, additional pressure is added with a pump.

The temperature of the feed as it enters the column does not necessarily equal the temperature in the column at the feed-tray location. However, such equality will increase second-law efficiency. It is usually best to avoid a subcooled liquid or superheated vapor feed and supply a partially vaporized feed. This is achieved by preheating the feed in a heat exchanger with the bottoms product or some other process stream that possesses a suitably high temperature, to ensure a reasonable  $\Delta T$  driving force for heat transfer, and a sufficient available enthalpy.

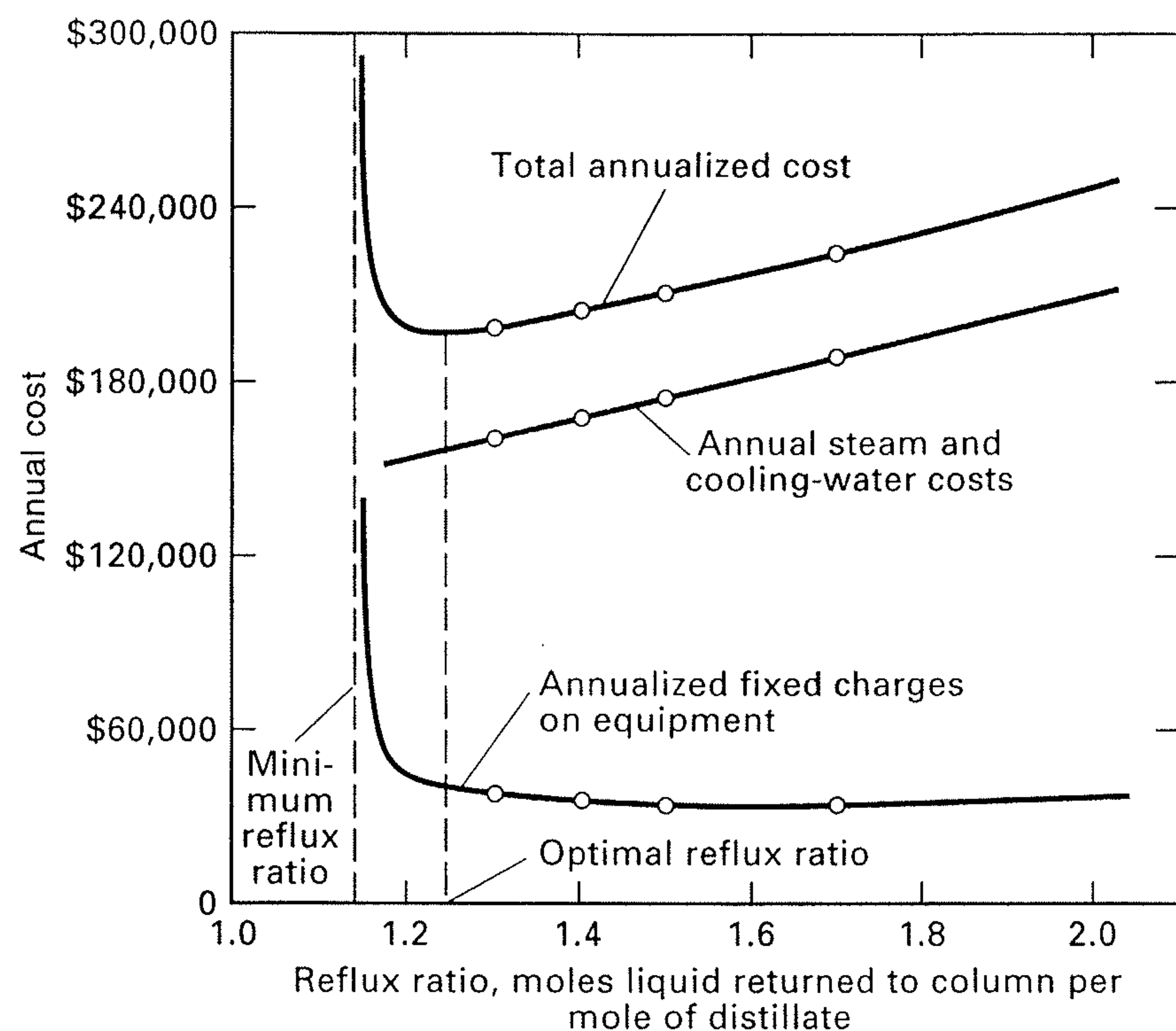
### Optimal Reflux Ratio

An industrial distillation column must be operated between the two limiting conditions of minimum reflux and total reflux. As shown in Table 7.3, for a typical case adapted from Peters and Timmerhaus [6], as the reflux ratio is increased from the minimum value, the number of plates decreases, the column diameter increases, and the reboiler steam and condenser cooling-water requirements increase. When the annualized fixed investment costs for the column, condenser, reflux drum, reflux pump, and reboiler are added to the annual cost of steam and cooling water, an optimal reflux ratio is established, as shown, for the conditions of Table 7.3, in Figure 7.22. For this example the optimal  $R/R_{\text{min}}$  is 1.1.

**Table 7.3** Effect of Reflux Ratio on Annualized Cost of a Distillation Operation

$R/R_{\text{min}}$	Actual $N$	Diam., ft	Reboiler Duty, Btu/h	Condenser Duty, Btu/h	Annualized Cost, \$/yr			Total Annualized Cost, \$/yr
					Equipment	Cooling Water	Steam	
1.00	Infinite	6.7	9,510,160	9,416,000	Infinite	17,340	132,900	Infinite
1.05	29	6.8	9,776,800	9,680,000	44,640	17,820	136,500	198,960
1.14	21	7.0	10,221,200	10,120,000	38,100	18,600	142,500	199,200
1.23	18	7.1	10,665,600	10,560,000	36,480	19,410	148,800	204,690
1.32	16	7.3	11,110,000	11,000,000	35,640	20,220	155,100	210,960
1.49	14	7.7	11,998,800	11,880,000	35,940	21,870	167,100	224,910
1.75	13	8.0	13,332,000	13,200,000	36,870	24,300	185,400	246,570

(Adapted from an example by Peters and Timmerhaus [6].)



**Figure 7.22** Optimal reflux ratio for a representative distillation operation.

[Adapted from M.S. Peters and K.D. Timmerhaus, *Plant Design and Economics for Chemical Engineers*, 4th ed., McGraw-Hill, New York (1991).]

The data in Table 7.3 show that although the condenser and reboiler duties are almost identical for a given reflux ratio, the annual cost of steam for the reboiler is almost eight times that of the cost of condenser cooling water. The total annual cost is dominated by the cost of steam except at the minimum-reflux condition. At the optimal reflux ratio, the cost of steam is 70% of the total annualized cost. Because the cost of steam is dominant, the optimal reflux ratio is sensitive to the steam cost. For example, at the extreme of zero cost for steam, the optimal  $R/R_{\min}$  for this example is shifted from 1.1 to 1.32. This example assumes that the heat removed by cooling water in the condenser has no value.

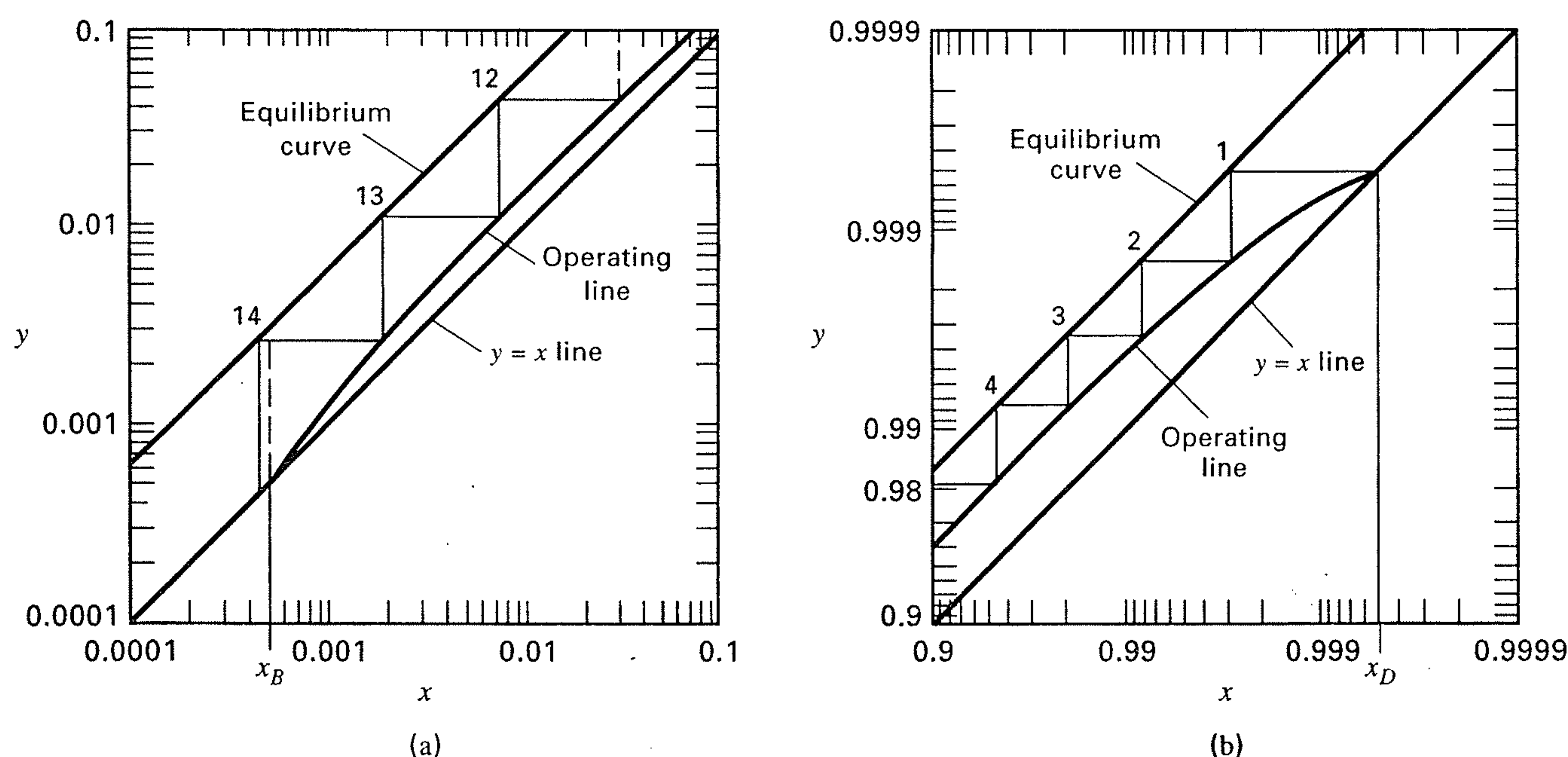
The range of optimal ratio of reflux to minimum reflux often is from 1.05 to 1.50, with the lower value applying to a difficult separation (e.g.,  $\alpha = 1.2$ ) and the higher value applying to an easy separation (e.g.,  $\alpha = 5$ ). However, as seen

in Figure 7.22, the optimal reflux ratio is not sharply defined. Accordingly, to achieve greater operating flexibility, columns are often designed for reflux ratios greater than the optimum.

### Large Number of Stages

The McCabe–Thiele graphical construction is difficult to apply when conditions of relative volatility and/or product purities are such that a large number of stages must be stepped off. In that event, one of the following techniques can be used to determine the stage requirements.

1. Separate plots of expanded scales and/or larger dimensions are used for stepping off stages at the ends of the  $y$ - $x$  diagram. For example, the additional plots might cover just the regions (1) 0.95 to 1.0 and (2) 0 to 0.05.
2. As described by Horvath and Schubert [7] and shown in Figure 7.23, a plot based on logarithmic coordinates is used for the low (bottoms) end of the  $y$ - $x$  diagram, while for the high (distillate) end, the log–log graph is turned upside down and rotated  $90^\circ$ . Unfortunately, as seen in Figure 7.23, the operating lines become curved, but they can be plotted from a few points computed from (7-9) and (7-14). The  $45^\circ$  line remains straight and the normally curved equilibrium curve becomes nearly straight at the two ends.
3. The stages at the two ends are computed algebraically in the manner of part (b) of Example 7.2. This is readily done with a spreadsheet computer program.
4. If the equilibrium data are given in analytical form, commercially available McCabe–Thiele computer programs can be used.
5. The stages are determined by combining the McCabe–Thiele graphical construction, for a suitable region in the middle, with the Kremser equations of Section 5.4 for the low and/or high ends, where absorption and stripping factors are almost constant. This technique, which is often preferred, is illustrated in the following example.



**Figure 7.23** Use of log–log coordinates for McCabe–Thiele construction: (a) bottoms end of column; (b) distillate end of column.

**EXAMPLE 7.4**

Repeat part (c) of Example 7.1 for benzene distillate and bottoms purities of 99.9 and 0.1 mol%, respectively, using a reflux ratio of 1.88, which is about 30% higher than the minimum reflux of 1.44 for these new purities. At the top of the column,  $\alpha = 2.52$ ; at the bottom,  $\alpha = 2.26$ .

**SOLUTION**

Figure 7.24 shows the McCabe–Thiele construction for the region of  $x$  from 0.028 to 0.956, where the stages have been stepped off in two directions, starting from the feed stage. In this middle region, seven stages are stepped off above the feed stage and eight below the feed stage, for a total of 16 stages, including the feed stage. The Kremser equations can now be applied to determine the remaining stages needed to achieve the desired high purities for the distillate and bottoms.

*Additional stages for the rectifying section.* With respect to Figure 5.8a, counting stages from the top down, from Figure 7.24:

From (7-3), for  $(x_N)_{\text{benzene}} = 0.956$ ,

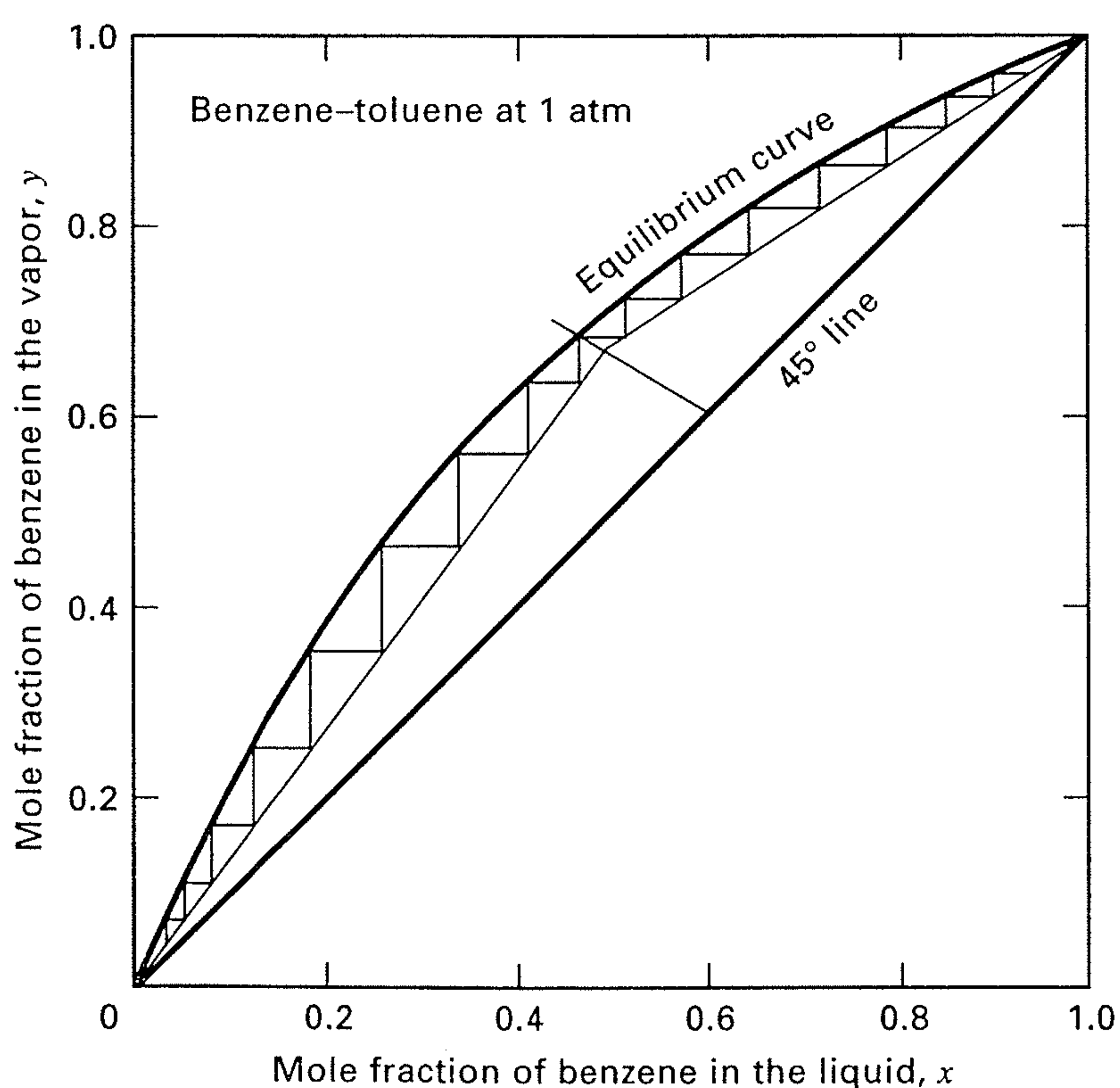
$$(y_{N+1})_{\text{benzene}} = 0.982 \quad \text{and} \quad (y_{N+1})_{\text{toluene}} = 0.018$$

Also,  $(x_0)_{\text{benzene}} = (y_1)_{\text{benzene}} = 0.999$  and  $(x_0)_{\text{toluene}} = (y_1)_{\text{toluene}} = 0.001$

Combining the Kremser equations (5.55), (5-34), (5-35), (5-48), and (5-50) and performing a number of algebraic manipulations:

$$N_R = \frac{\log \left[ \frac{1}{A} + \left( 1 - \frac{1}{A} \right) \left( \frac{y_{N+1} - x_0 K}{y_1 - x_0 K} \right) \right]}{\log A} \quad (7-39)$$

where  $N_R$  = additional equilibrium stages for the rectifying section. For that section, which is like an absorption section, it is best to apply (7-39) to toluene, the heavy key. Because  $\alpha = 2.52$  at the top of the column, where  $K_{\text{benzene}}$  is close to one, take  $K_{\text{toluene}} = 1/2.52 = 0.397$ . Since  $R = 1.88$ ,  $L/V = R/(R + 1) = 0.653$ .



**Figure 7.24** McCabe–Thiele construction for Example 7.4 from  $x = 0.028$  to  $x = 0.956$ .

Therefore, the absorption factor for toluene is  $A_{\text{toluene}} = L/(VK_{\text{toluene}}) = 0.653/0.397 = 1.64$  which is assumed to remain constant in the uppermost part of the rectifying section. Therefore, from (7-39) for toluene,

$$N_R = \frac{\log \left[ \frac{1}{1.64} + \left( 1 - \frac{1}{1.64} \right) \left( \frac{0.018 - 0.001(0.397)}{0.001 - 0.001(0.397)} \right) \right]}{\log 1.64} = 5.0$$

*Additional stages for the stripping section.* With respect to Figure 5.8b, counting stages from the bottom up, we have from Figure 7.24:  $(x_{N+1})_{\text{benzene}} = 0.048$ . Also,  $(x_1)_{\text{benzene}} = (x_B)_{\text{benzene}} = 0.001$ . Combining the Kremser equations for a stripping section gives

$$N_S = \frac{\log \left[ \bar{A} + (1 - \bar{A}) \left( \frac{x_{N+1} - x_1/K}{x_1 - x_1/K} \right) \right]}{\log(1/\bar{A})} \quad (7-40)$$

where

$N_S$  = additional equilibrium stages for the stripping section

$\bar{A}$  = absorption factor in the stripping section =  $\bar{L}/K\bar{V}$

Because benzene is being stripped in the stripping section, it is best to apply (7-40) to the benzene. At the bottom of the column, where  $K_{\text{toluene}}$  is approximately 1.0,  $\alpha = 2.26$ , and therefore  $K_{\text{benzene}} = 2.26$ . By material balance, with flows in lbmol/h,  $D = 270.1$ . For  $R = 1.88$ ,  $L = 507.8$ , and  $V = 270.1 + 507.8 = 777.9$ . From Example 7.1,  $V_F = D = 270.1$  and  $L_F = 450 - 270.1 = 179.9$ . Therefore,  $\bar{L} = L + L_F = 507.8 + 179.9 = 687.7$  lbmol/h and  $\bar{V} = V - V_F = 777.9 - 270.1 = 507.8$  lbmol/h.

$$\bar{L}/\bar{V} = 687.7/507.8 = 1.354;$$

$$\bar{A}_{\text{benzene}} = \bar{L}/K\bar{V} = 1.354/2.26 = 0.599$$

Substitution into (7-40) gives

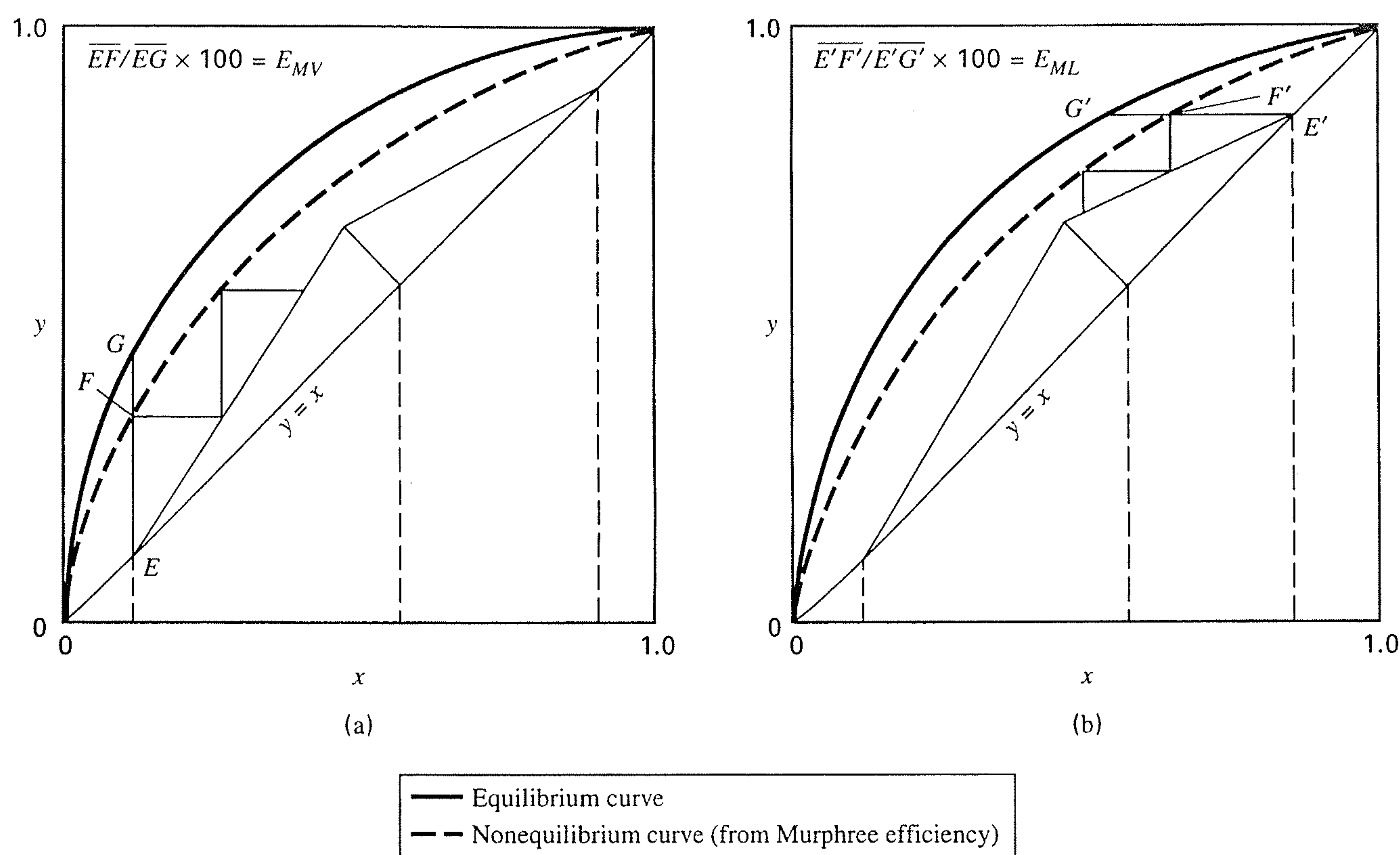
$$N_S = \frac{\log \left[ 0.599 + (1 - 0.599) \left( \frac{0.028 - 0.001/2.26}{0.001 - 0.001/2.26} \right) \right]}{\log(1/0.599)} = 5.9$$

This value includes the partial reboiler. Accordingly, the total number of equilibrium stages starting from the bottom is: partial reboiler + 5.9 + 8 + feed stage + 7 + 5.0 = 26.9.

**Use of Murphree Efficiency**

The McCabe–Thiele method assumes that the two phases leaving each stage are in thermodynamic equilibrium. In industrial, countercurrent, multistage equipment, it is not always practical to provide the combination of residence time and intimacy of contact required to approach equilibrium closely. Hence, concentration changes for a given stage are usually less than predicted by equilibrium.

As discussed in Section 6.5, a stage efficiency frequently used to describe individual tray performance for individual components is the Murphree plate efficiency. This efficiency can be defined on the basis of either phase and, for a given component, is equal to the change in actual composition in



**Figure 7.25** Use of Murphree plate efficiencies in McCabe–Thiele construction.

the phase, divided by the change predicted by equilibrium. This definition applied to the vapor phase can be expressed in a manner similar to (6-28):

$$E_{MV} = \frac{y_n - y_{n+1}}{y_n^* - y_{n+1}} \quad (7-41)$$

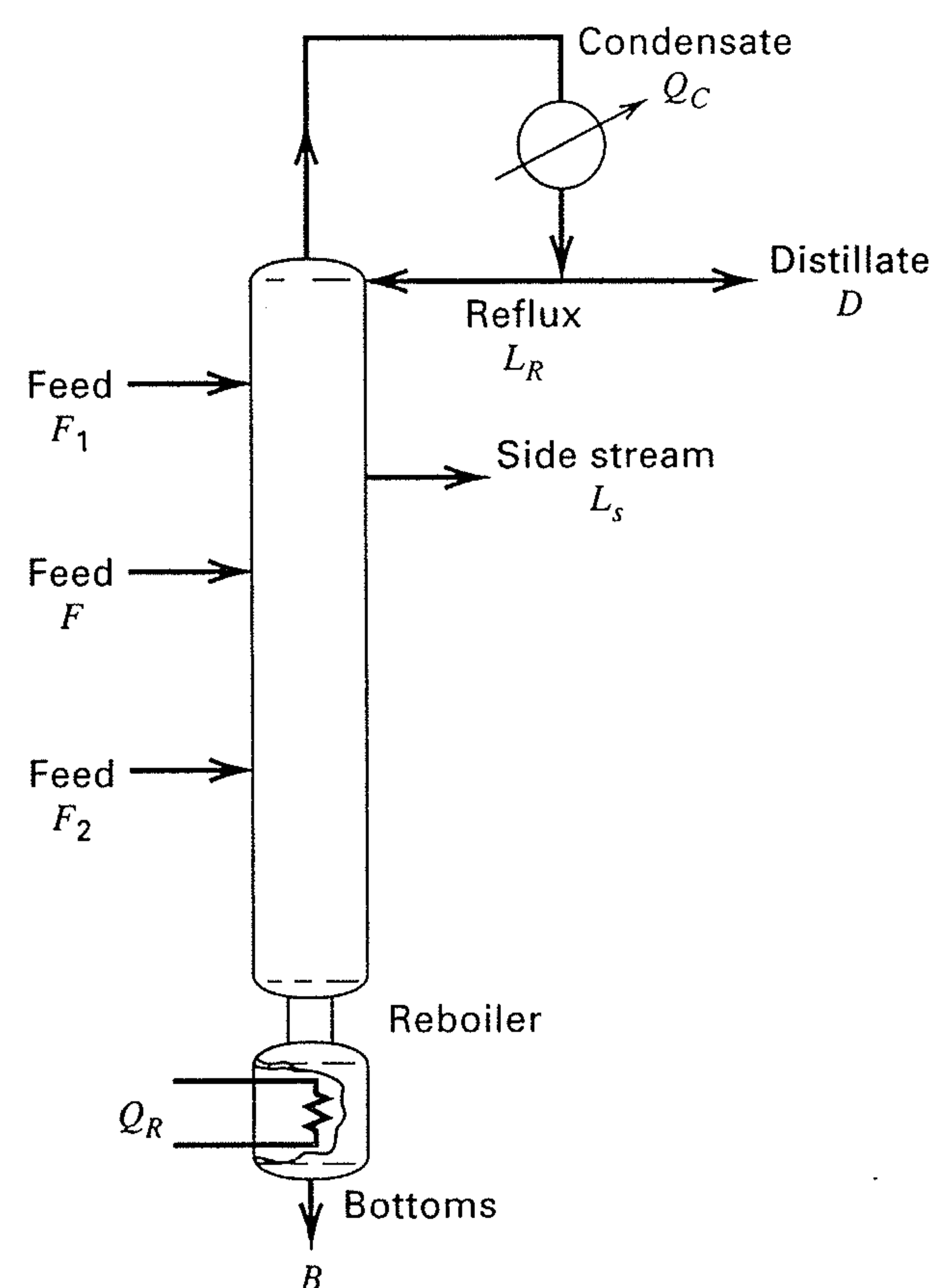
Where  $E_{MV}$  is the Murphree vapor efficiency for stage  $n$ , where  $n + 1$  is the stage below and  $y_n^*$  is the composition in the hypothetical vapor phase in equilibrium with the liquid composition leaving stage  $n$ . Values of  $E_{MV}$  can be less than or somewhat more than 100%. The component subscript in (7-41) is dropped because values of  $E_{MV}$  are equal for the two components of a binary mixture.

In stepping off stages, the Murphree vapor efficiency, if known, can be used to dictate the percentage of the distance taken from the operating line to the equilibrium line; only  $E_{MV}$  of the total vertical path is traveled. This is shown in Figure 7.25a for the case of Murphree efficiencies based on the vapor phase. Figure 7.25b shows the case when the Murphree tray efficiency is based on the liquid. In effect, the dashed curve for actual exit-phase composition replaces the thermodynamic equilibrium curve for a particular set of operating lines. In Figure 7.25a,  $E_{MV} = \overline{EF}/\overline{EG} = 0.7$  for the bottom stage.

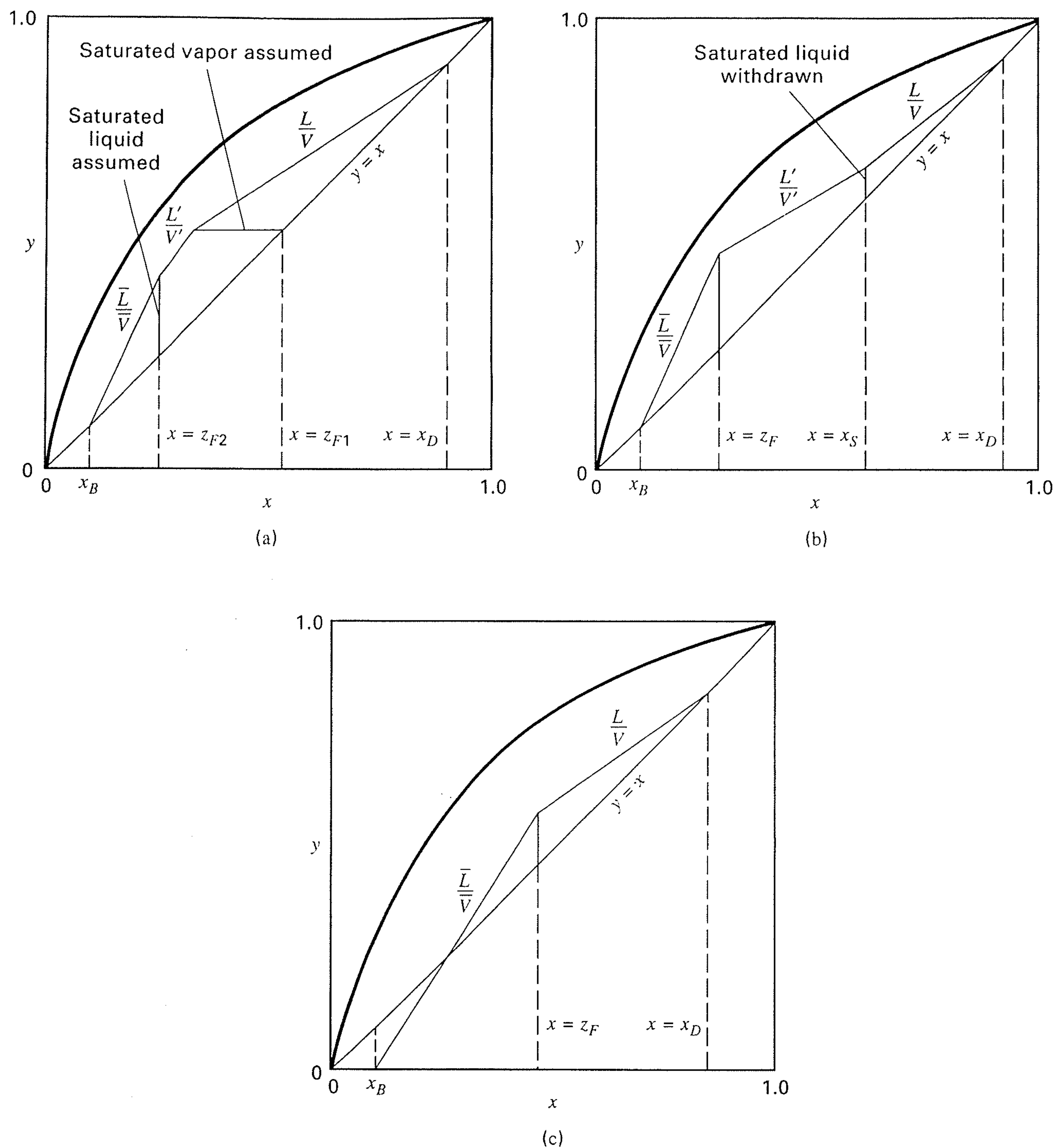
### Multiple Feeds, Side Streams, and Open Steam

The McCabe–Thiele method for a single feed and two products is readily extended to the case of multiple feeds and/or side streams by adding one additional operating line for each additional feed or side stream. A multiple-feed arrangement is shown in Figure 7.26. In the absence of side stream  $L_S$ , this arrangement has no effect on the material balance associated with the rectifying section of the column above the upper-feed point,  $F_1$ . The section of column between the upper-feed point

and the lower-feed point  $F_2$  (in the absence of feed  $F$ ) is represented by an operating line of slope  $L'/V'$ , this line intersecting the rectifying-section operating line. A similar argument holds for the stripping section of the column. Hence it is possible to apply the McCabe–Thiele graphical construction shown in Figure 7.27a, where feed  $F_1$  is a dew-point vapor, while feed  $F_2$  is a bubble-point liquid. Feed  $F$  and side stream  $L_S$  of Figure 7.26 are not present. Thus, between the two feed points for this example, the molar vapor flow rate is  $V' = V - F_1$  and  $\bar{L} = L' + F_2 = L + F_2$ . For given  $x_B, z_{F_2}$ ,



**Figure 7.26** Complex distillation column with multiple feeds and side stream.



**Figure 7.27** McCabe–Thiele construction for complex columns: (a) two feeds (saturated liquid and saturated vapor); (b) one feed, one side stream (saturated liquid); (c) use of open steam.

$z_{F1}$ ,  $x_D$ , and  $L/D$ , the three operating lines in Figure 7.27a are readily constructed.

A side stream may be withdrawn from the rectifying section, the stripping section, or between multiple feed points, as a saturated vapor or saturated liquid. Within material-balance constraints,  $L_S$  and  $x_S$  can both be specified. In Figure 7.27b, a saturated-liquid side stream of composition  $x_S$  and molar flow rate  $L_S$  is withdrawn from the rectifying section above feed  $F$ . In the section of stages between the side stream-withdrawal stage and the feed stage,  $L' = L - L_S$ , while  $V' = V$ . The McCabe–Thiele constructions determine the location of the side stream stage. However, if it is not located directly above  $x_S$ , the reflux ratio must be varied until it does.

For certain types of distillation, an inert hot gas is introduced directly into the base of the column. Open steam, for example, can be used if one of the components in the mixture is water, or if water can form a second liquid phase, thereby reducing the boiling point, as in the steam distillation of fats, where heat is supplied by live, superheated steam and no reboiler is used. Most commonly, the feed contains water,

which is removed as bottoms. In that application,  $Q_R$  of Figure 7.26 is replaced by a stream of composition  $y = 0$  (pure steam) which, with  $x = x_B$ , becomes a point on the operating line, since the passing streams at this point actually exist at the end of the column. With open steam, the bottoms flow rate is increased by the flow rate of the open steam. The use of open steam rather than a reboiler for the operating condition  $F_1 = F_2 = L_S = 0$  is represented graphically in Figure 7.27c.

#### EXAMPLE 7.5

A complex distillation column, equipped with a partial reboiler and total condenser, and operating at steady state with a saturated-liquid feed, has a liquid side stream draw-off in the enriching (rectifying) section. Making the usual simplifying assumptions of the McCabe–Thiele method: (a) Derive an equation for the two operating lines in the enriching section. (b) Find the point of intersection of these operating lines. (c) Find the intersection of the operating line between  $F$  and  $L_S$  with the diagonal. (d) Show the construction on a  $y-x$  diagram.

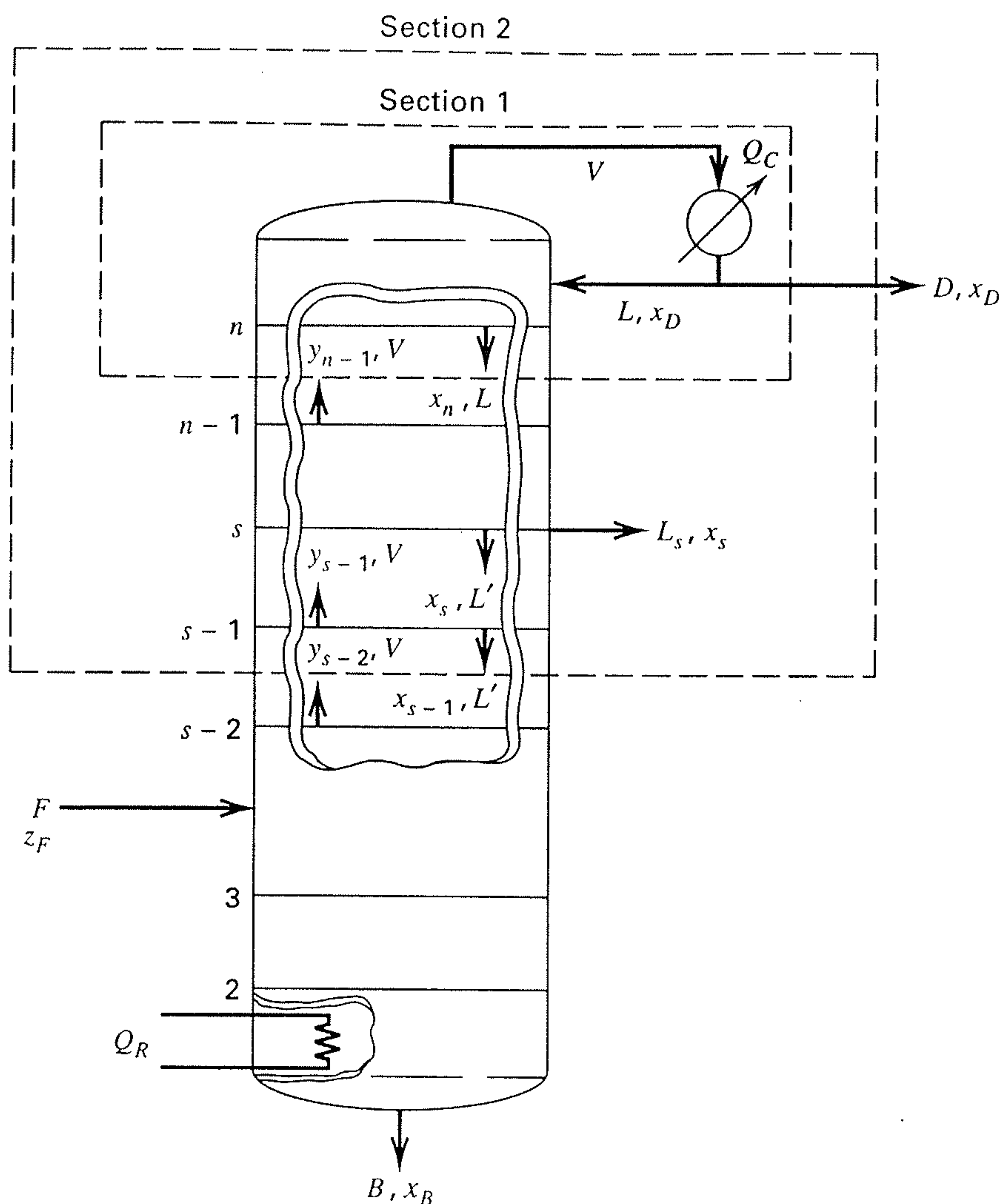


Figure 7.28 Distillation column with side stream for Example 7.5.

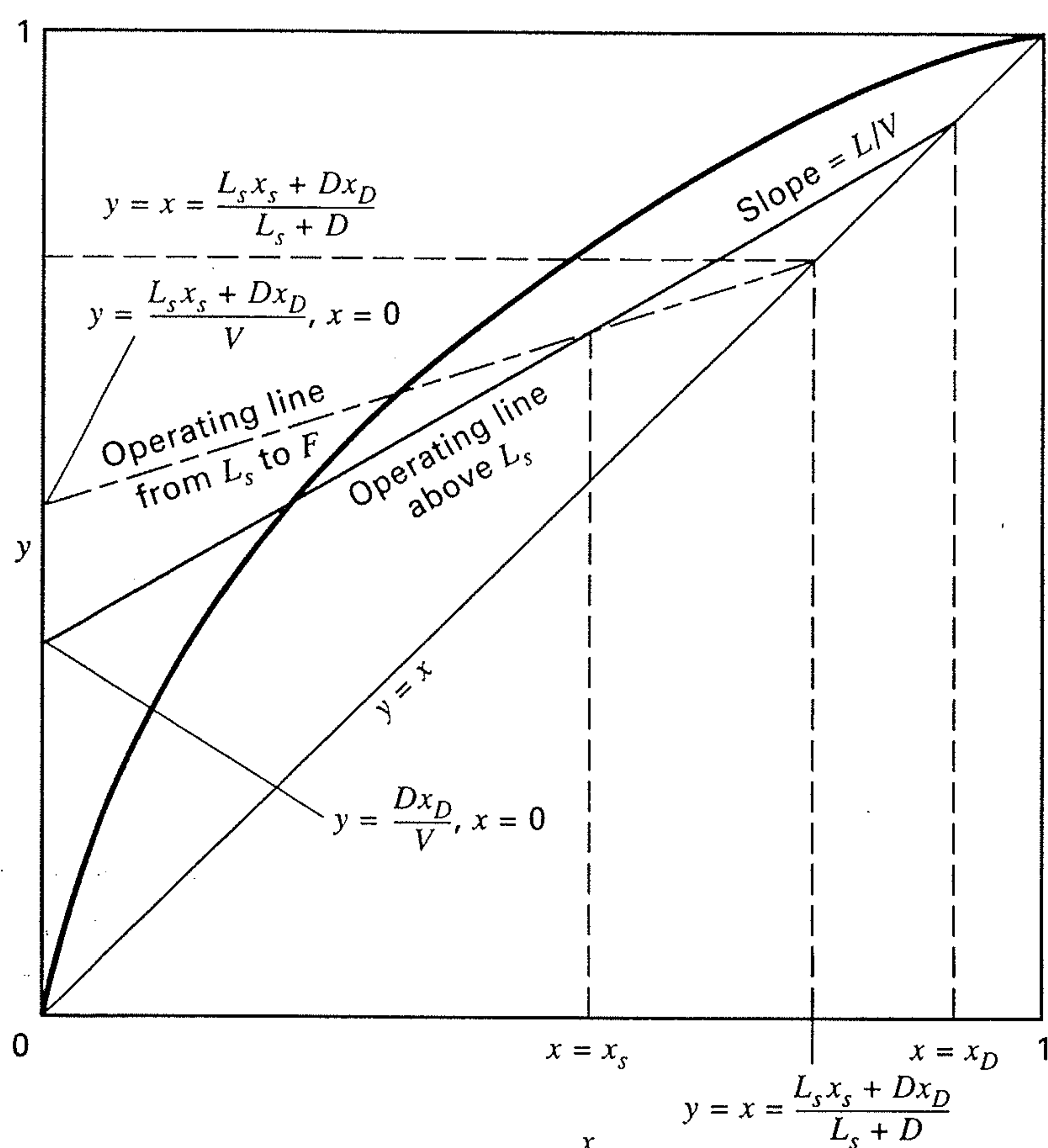


Figure 7.29 McCabe-Thiele diagram for Example 7.5.

### SOLUTION

- (a) By material balance over section 1 in Figure 7.28,  $V_{n-1}y_{n-1} = L_n x_n + D x_D$ . About section 2,  $V_{s-2}y_{s-2} = L'_{s-1}x_{s-1} + L_s x_s + D x_D$ . The two operating lines for conditions of

constant molar overflow become:

$$y = \frac{L}{V}x + \frac{D}{V}x_D \quad \text{and} \quad y = \frac{L'}{V}x + \frac{L_s x_s + D x_D}{V}$$

- (b) Equating the two operating lines, the intersection occurs at  $(L - L')x = L_s x_s$  and since  $L - L' = L_s$ , the point of intersection becomes  $x = x_s$ .

- (c) The intersection of the lines

$$y = \frac{L'}{V}x + \frac{L_s x_s + D x_D}{V}$$

and  $y = x$  occurs at  $x = \frac{L_s x_s + D x_D}{L_s + D}$

- (d) The  $y$ - $x$  diagram is shown in Figure 7.29.

### 7.3 ESTIMATION OF STAGE EFFICIENCY

Methods for estimating the stage efficiency for binary distillation are analogous to those for absorption and stripping, presented in Section 6.5. The efficiency is a complex function of tray design, fluid properties, and flow patterns. However, in hydrocarbon absorption and stripping, the liquid phase is often rich in heavy components so that liquid viscosity is high and mass-transfer rates are relatively low. This leads to low stage efficiencies, usually less than 50%. In contrast, for binary distillation, particularly of close-boiling mixtures, liquid viscosity is low, with the result that stage efficiencies, for well-designed trays and optimal operating conditions, are often higher than 70% and can be even higher than 100% for large-diameter columns where a cross-flow effect is present.

#### Performance Data

As discussed in *AIChE Equipment Testing Procedure* [8], performance data for an industrial distillation column are best obtained at conditions of total reflux (no feed or products) so as to avoid possible column-feed fluctuations, simplify location of the operating line, and avoid discrepancies between feed and feed-tray compositions. However, as shown by Williams, Stigger, and Nichols [9], efficiency measured at total reflux can differ markedly from that at design reflux ratio. Ideally, the column is operated in the range of 50% to 85% of flooding. If liquid samples are taken from the top and bottom of the column, the overall plate efficiency,  $E_o$ , can be determined from (6-21), where the number of theoretical stages required is determined by applying the McCabe-Thiele method at total reflux, as in Figure 7.11. If liquid samples are taken from the downcomers of intermediate trays, Murphree vapor efficiencies,  $E_{MV}$ , can be determined using (6-28). If liquid samples are withdrawn from different points on one tray, (6-30) can be applied to obtain point efficiencies,  $E_{OV}$ . Reliable values for these efficiencies require the availability of accurate vapor-liquid equilibrium data. For that reason, efficiency data for binary mixtures that form ideal solutions are preferred.



**Table 7.4** Performance Data for the Distillation of a Mixture of Methylene Chloride and Ethylene Chloride

Company	Eastman Kodak
Location	Rochester, New York
Column diameter	5.5 ft (65.5 in. I.D.)
No. of trays	60
Tray spacing	18 in.
Type tray	10 rows of 3-in.-diameter bubble caps on 4-7/8-in. triangular centers. 115 caps/tray
Bubbling area	20 ft <sup>2</sup>
Length of liquid travel	49 in.
Outlet-weir height	2.25 in.
Downcomer clearance	1.5 in.
Liquid rate	24.5 gal/min-ft = 1,115.9 lb/min
Vapor $F$ -factor	1.31 ft/s (lb/ft <sup>3</sup> ) <sup>0.5</sup>
Percent of flooding	85
Pressure, top tray	33.8 psia
Pressure, bottom tray	42.0 psia
Liquid composition, mole % methylene chloride:	
From tray 33	89.8
From tray 32	72.6
From tray 29	4.64

Source: J.A. Gerster, A.B. Hill, N.H. Hochgrof, and D.B. Robinson, *Tray Efficiencies in Distillation Columns, Final Report from the University of Delaware, AIChE, New York (1958)*.

Table 7.4, from Gerster et al. [10], lists plant data, obtained from Eastman Kodak Company in Rochester, New York, for the distillation at total reflux of a methylene chloride (MC)–ethylene chloride (EC) mixture in a 5.5-ft-diameter column containing 60 bubble-cap trays on 18-in. tray spacing and operating at 85% of flooding at total reflux. MC is the light key.

### EXAMPLE 7.6

Using the performance data of Table 7.4, estimate: (a) the overall tray efficiency for the section of trays from 35 to 29 and (b) the Murphree vapor efficiency for tray 32. Assume the following values for relative volatility:

$x_{MC}$	$\alpha_{MC,EC}$	$y_{MC}$ from (7-3)
0.00	3.55	0.00
0.10	3.61	0.286
0.20	3.70	0.481
0.30	3.76	0.617
0.40	3.83	0.719
0.50	3.91	0.796
0.60	4.00	0.857
0.70	4.03	0.904
0.80	4.09	0.942
0.90	4.17	0.974
1.00	4.25	1.00

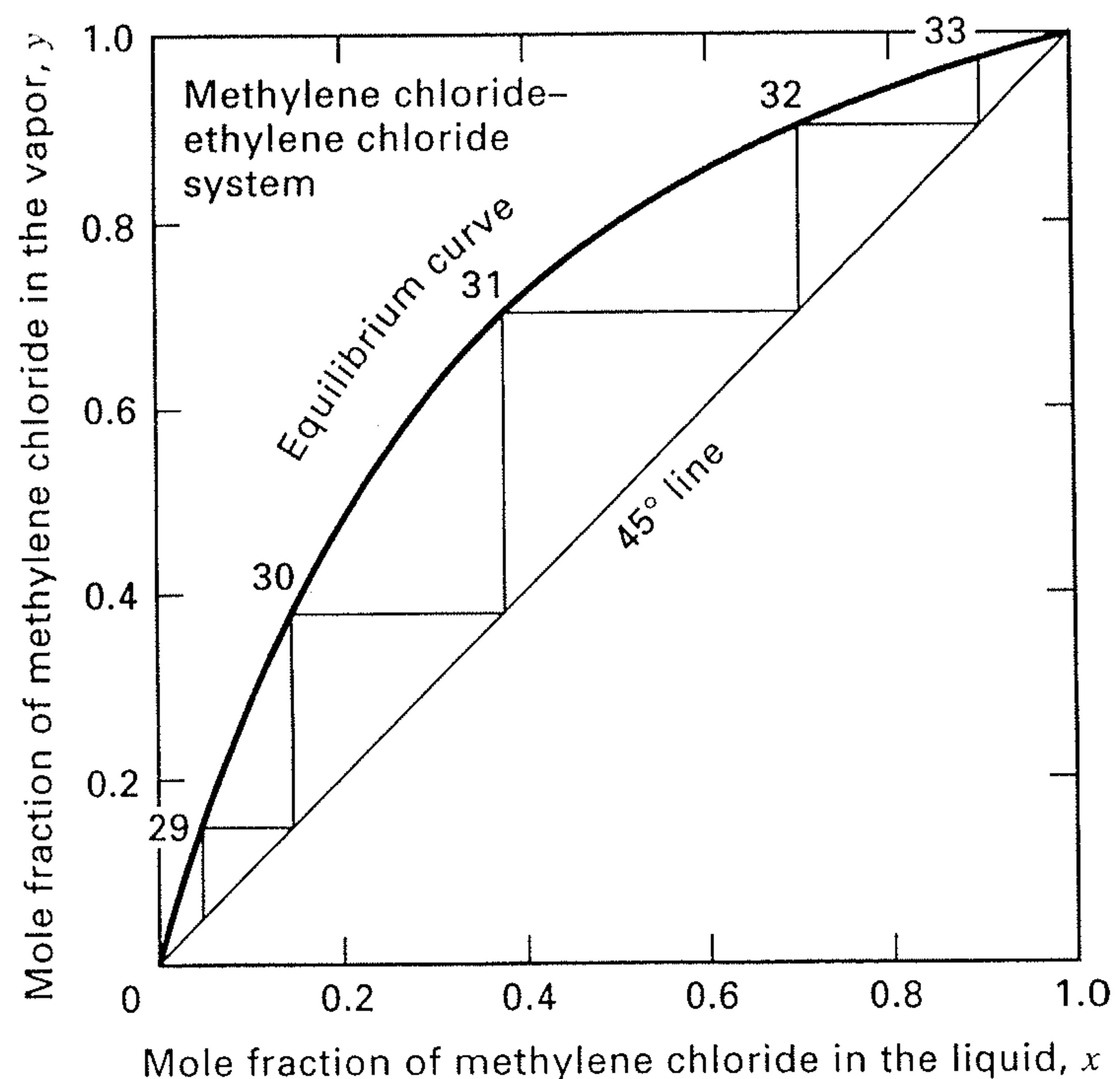


Figure 7.30 McCabe–Thiele diagram for Example 7.6.

### SOLUTION

- (a) The above  $x$ – $y$  data are plotted in Figure 7.30. Four theoretical stages are stepped off from  $x_{33} = 0.898$  to  $x_{29} = 0.0464$  for total reflux. Since the actual number of stages is also 4, the overall stage efficiency from (6-21) is 100%.
- (b) At total reflux conditions, passing vapor and liquid streams have the same composition. That is, the operating line is the 45° line. Using this together with the above performance data and the equilibrium curve in Figure 7.30, we obtain for methylene chloride, with trays counted from the bottom up:

$$y_{32} = x_{33} = 0.898 \quad \text{and} \quad y_{31} = x_{32} = 0.726$$

From (6-28),

$$(E_{MV})_{32} = \frac{y_{32} - y_{31}}{y_{32}^* - y_{31}}$$

From Figure 7.30, for  $x_{32} = 0.726$ ,  $y_{32}^* = 0.917$ ,

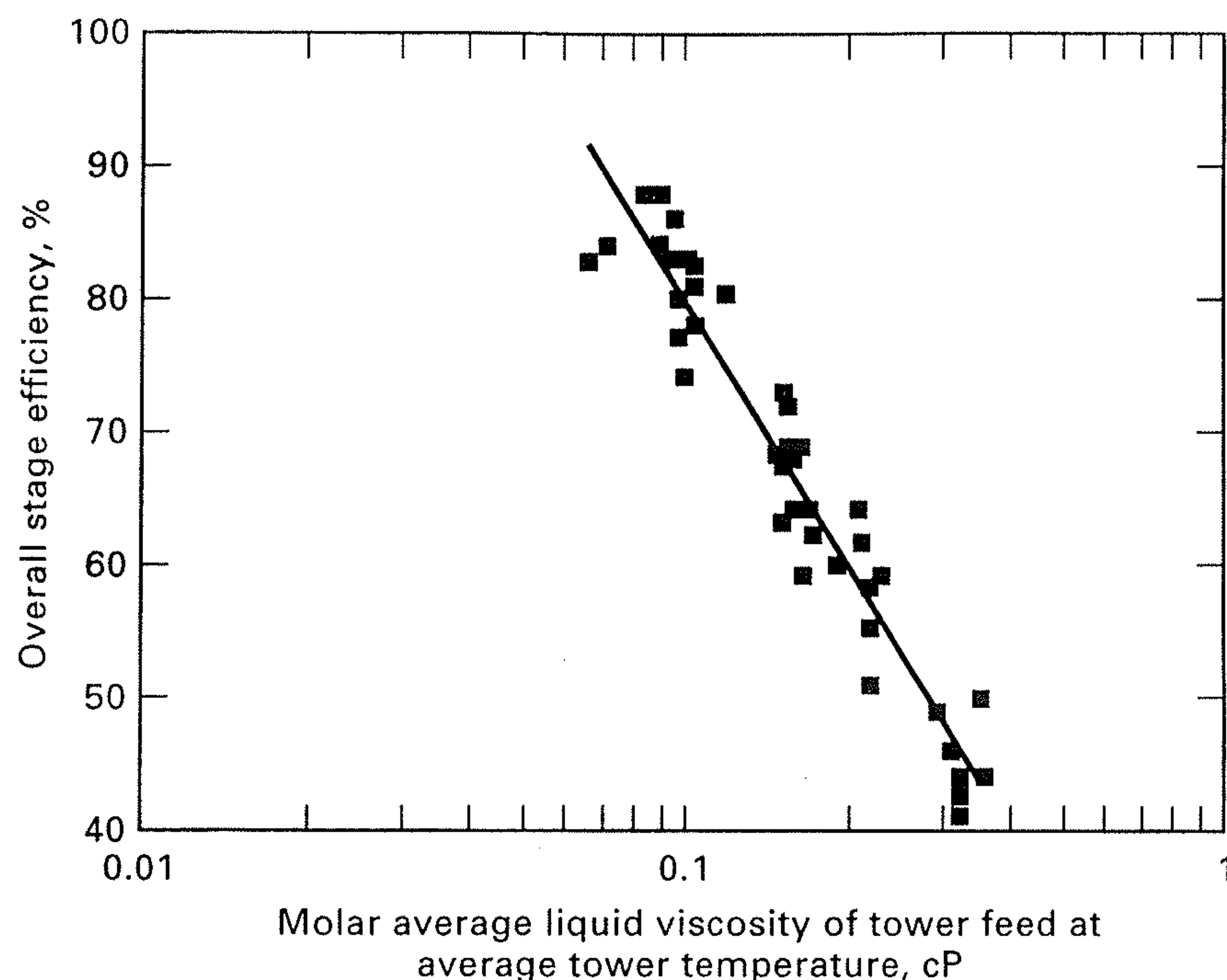
$$(E_{MV})_{32} = \frac{0.898 - 0.726}{0.917 - 0.726} = 0.90 \quad \text{or} \quad 90\%$$

### Empirical Correlations

Based on 41 sets of performance data for bubble-cap-tray and sieve-tray columns, distilling mainly hydrocarbon mixtures and a few water and miscible organic mixtures, Drickamer and Bradford [11] correlated the overall stage efficiency for the separation of the two key components in terms of the molar-average liquid viscosity of the tower feed at the average tower temperature. The data covered average temperatures from 157 to 420°F, pressures from 14.7 to 366 psia, feed liquid viscosities from 0.066 to 0.355 cP, and overall tray efficiencies from 41% to 88%. The empirical equation

$$E_o = 13.3 - 66.8 \log \mu \quad (7-42)$$

where  $E_o$  is in percent and  $\mu$  is in centipoise, fits the data with average and maximum percent deviations of 5.0% and 13.0%, respectively. A plot of the Drickamer and Bradford



**Figure 7.31** Drickamer and Bradford correlation for plate efficiency of distillation columns.

correlation, compared to performance data for distillation, is given in Figure 7.31. Equation (7-42) is restricted to the range of the data and is intended mainly for hydrocarbon mixtures.

Mass-transfer theory, discussed in Section 6.5, indicates that, when the relative volatility covers a wide range, the relative importance of liquid-phase and gas-phase mass-transfer resistances can shift. Thus, as might be expected, O'Connell [12] found that the Drickamer-Bradford correlation correlates data inadequately for fractionators operating on key components with large relative volatilities. Separate correlations in terms of a viscosity-volatility product were developed for fractionators and for absorbers and strippers by O'Connell. However, as shown in Figure 7.32, Lockhart and Leggett [13] were able to obtain a single correlation by using the product of liquid viscosity and an appropriate volatility as the correlating variable. For fractionators, the relative volatility of the key components was used; for hydrocarbon absorbers, the volatility was taken as 10 times the  $K$ -value of a selected key component, which must be one

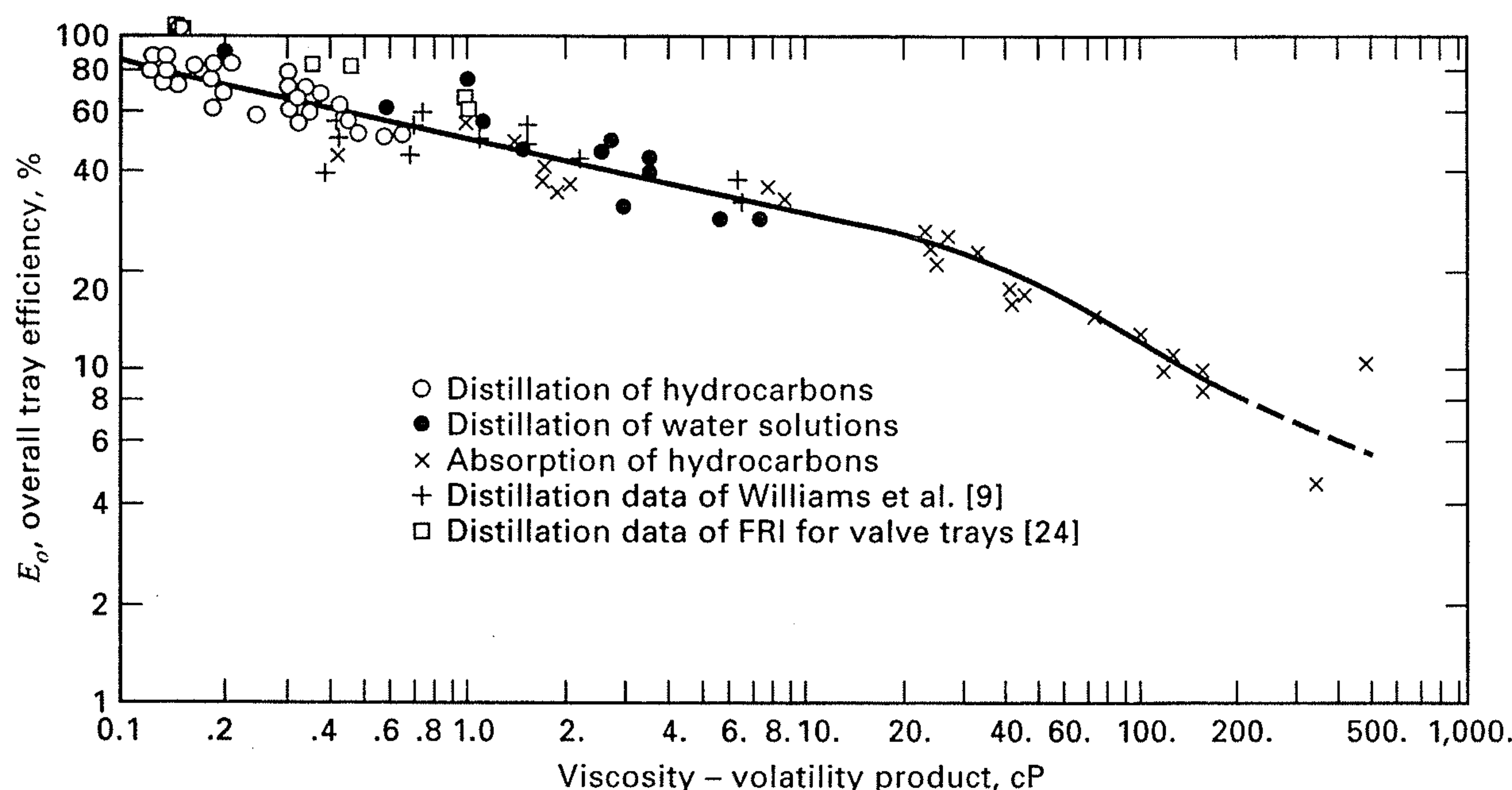
that is reasonably distributed between top and bottom products. The data used by O'Connell cover a range of relative volatility from 1.16 to 20.5. A comprehensive study of the effect on  $E_o$  of the ratio of liquid-to-vapor molar flow rates,  $L/V$ , for eight different binary systems in a 10-in.-diameter column with bubble-cap trays was reported by Williams, et al. [9]. The systems included water, hydrocarbons, and other organic compounds. While  $L/V$  did have an effect, it could not be correlated. For fractionation with  $L/V$  nearly equal to 1.0 (i.e., total reflux), their distillation data, which are included in Figure 7.32, are in reasonable agreement with the O'Connell correlation. For the distillation of hydrocarbons in a column having a diameter of 0.45 m, Zuiderweg, Verburg, and Gilissen [14] found differences in  $E_o$  among bubble-cap, sieve, and valve trays to be insignificant at 85% of flooding. Accordingly, Figure 7.32 is assumed to be applicable to all three tray types, but may be somewhat conservative for well-designed trays. For example, data of Fractionation Research Incorporated (FRI) for valve trays operating with the cyclohexane/ $n$ -hexane and isobutane/ $n$ -butane systems are also included in Figure 7.32 and show efficiencies 10% to 20% higher than the correlation.

For the distillation data plotted in Figure 7.32, which cover a viscosity-relative volatility range for distillation of from 0.1 to 10 cP, the O'Connell correlation fits the empirical equation

$$E_o = 50.3(\alpha\mu)^{-0.226} \quad (7-43)$$

where  $E_o$  is in percent and  $\mu$  is in centipoise. The relative volatility is determined for the two key components at average column conditions.

Most of the data for developing the correlation of Figure 7.32 are for columns having a liquid flow path across the active tray area of from 2 to 3 ft. Gautreaux and O'Connell [15], using theory and experimental data, showed that higher efficiencies are achieved for longer flow paths. For short liquid flow paths, the liquid flowing across the tray is usually mixed completely. For longer flow paths, the equivalent of two or more completely mixed, successive liquid zones may



**Figure 7.32** Lockhart and Leggett version of the O'Connell correlation for overall tray efficiency of fractionators, absorbers, and strippers.

[Adapted from F.J. Lockhart and C.W. Leggett, in *Advances in Petroleum Chemistry and Refining*, Vol. 1, Eds., K.A. Kobe and John J. McKetta, Jr., Interscience, New York, pp. 323-326 (1958).]

**Table 7.5** Correction to Overall Tray Efficiency for Length of Liquid Flow Path ( $0.1 \leq \mu\alpha \leq 1.0$ )

Length of Liquid Flow Path, ft	Value to Be Added to $E_o$ from Figure 7.32, %
3	0
4	10
5	15
6	20
8	23
10	25
15	27

Source: F.J. Lockhart and C.W. Leggett, in K.A. Kobe and J.J. McKetta, Jr., Eds., *Advances in Petroleum Chemistry and Refining*, Vol. 1, Interscience, New York, pp. 323–326 (1958).

be present. The result is a greater average driving force for mass transfer, and, thus, a higher efficiency—sometimes even greater than 100%. Provided that the viscosity–volatility product lies between 0.1 and 1.0, Lockhart and Leggett [13] recommend addition of the increments in Table 7.5 to the value of  $E_o$  from Figure 7.32 when the liquid flow path is greater than 3 ft. However, at large liquid rates, long liquid-path lengths are undesirable because they lead to excessive liquid gradients, causing maldistribution of vapor flow. The use of multipass trays, shown in Figure 6.16, to prevent excessive liquid gradients is discussed in Section 6.5.

### EXAMPLE 7.7

For the benzene–toluene distillation of Figure 7.1, use the Drickamer–Bradford and O’Connell correlations to estimate the overall stage efficiency and number of actual plates required. Calculate the height of the tower assuming 24-in. tray spacing, with 4 ft above the top tray for removal of entrained liquid and 10 ft below the bottom tray for bottoms surge capacity. The separation requires 20 equilibrium stages plus a partial reboiler that acts as an equilibrium stage.

### SOLUTION

For estimating overall stage efficiency, the liquid viscosity is determined at the feed-stage condition of 220°F, assuming a liquid composition of 50 mol% benzene.

$$\begin{aligned} \mu \text{ of benzene} &= 0.10 \text{ cP}; & \mu \text{ of toluene} &= 0.12 \text{ cP}; \\ \text{Average } \mu &= 0.11 \text{ cP}. \end{aligned}$$

From Figure 7.3, take the average relative volatility as

$$\text{Average } \alpha = \frac{\alpha_{\text{top}} + \alpha_{\text{bottom}}}{2} = \frac{2.52 + 2.26}{2} = 2.39$$

From the Drickamer–Bradford correlation (7-42),  $E_o = 13.3 - 66.8 \log(0.11) = 77\%$

This is close to the value given in the description of this problem. Therefore, 26 actual trays are required and column height =  $4 + 2(26 - 1) + 10 = 64$  ft.

From the O’Connell correlation (7-43),

$$E_o = 50.3[(2.39)(0.11)]^{-0.226} = 68\%.$$

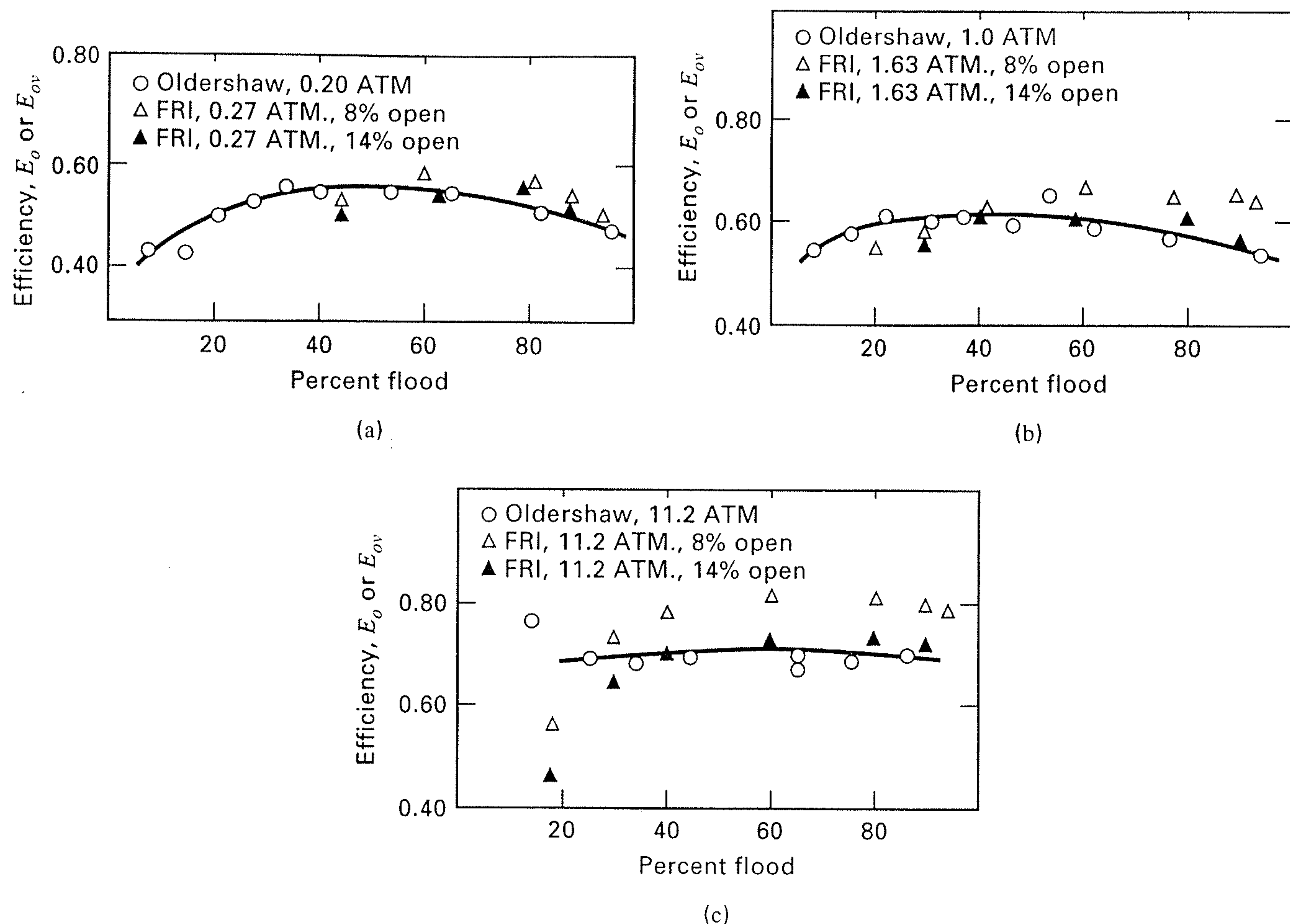
For a 5-ft-diameter column, the length of the liquid flow path is about 3 ft for a single-pass tray and even less for a two-pass tray. From Table 7.5, the efficiency correction is zero. Therefore, the actual number of trays required is  $20/0.68 = 29.4$ , or call it 30 trays. Column height =  $4 + 2(30 - 1) + 10 = 72$  ft.

### Semi-Theoretical Models

In Section 6.5, semi-theoretical tray models based on the Murphree vapor efficiency and the Murphree vapor-point efficiency are applied to absorption and stripping. These same relationships are valid for distillation. However, because the equilibrium line is curved for distillation,  $\lambda$  must be taken as  $mV/L$  (not  $KV/L = 1/A$ ), where  $m$  = local slope of the equilibrium curve =  $dy/dx$ . In Section 6.6, the method of Chan and Fair [16] is used for estimating the Murphree vapor-point efficiency from mass-transfer considerations. The Murphree vapor efficiency can then be estimated. The Chan and Fair correlation is specifically applicable to binary distillation because it was developed from experimental data that includes six different binary systems.

### Scale-up from Laboratory Data

When binary mixtures form ideal or nearly ideal solutions, it is rarely necessary to obtain laboratory distillation data. Where nonideal solutions are formed and/or the possibility of azeotrope formation exists, use of a small laboratory Oldershaw column, of the type discussed in Section 6.5, should be used to verify the desired degree of separation and to obtain an estimate of the Murphree vapor-point efficiency. The ability to predict the efficiency of an industrial-size sieve-tray column from measurements with 1-in. glass and 2-in. metal diameter Oldershaw columns is shown in Figure 7.33, from the work of Fair, Null, and Bolles [17]. The measurements were made for the cyclohexane/*n*-heptane system at vacuum conditions (Figure 7.33a) and at near-atmospheric conditions (Figure 7.33b) and for the isobutane/*n*-butane system at 11.2 atm (Figure 7.33c). The Oldershaw data are correlated by the solid lines. Data for the 4-ft-diameter column with sieve trays of 8.3% and 13.7% open area were obtained by Sakata and Yanagi [18] and Yanagi and Sakata [19], respectively, of FRI. The Oldershaw column is assumed to measure point efficiency. The FRI column measured overall efficiency, but the relations of Section 6.5 were used to convert the FRI data to the point efficiencies shown in Figure 7.33. The data cover a percent of flooding ranging from about 10% to 95%. Data from the Oldershaw column are in reasonable agreement with the FRI data for 14% open area, except at the lower part of the flooding range. In Figures 7.33b and 7.33c, the FRI data for 8% open area show efficiencies as much as 10 percentage points higher.



**Figure 7.33** Comparison of Oldershaw column efficiency with point-efficiency in 4-ft-diameter FRI column with sieve trays: (a) cychlohexane/*n*-heptane system; (b) cyclohexane/*n*-heptane systems; (c) isobutane/*n*-butane system.

## 7.4 DIAMETER OF TRAYED TOWERS AND REFLUX DRUMS

In Section 6.6, methods for estimating tray diameter and pressure drop for absorbers and strippers are presented. These same methods apply to distillation columns. Calculations of column diameter are usually made for conditions at the top and bottom trays of the tower. If the diameters differ by 1 ft or less, the larger diameter is used for the entire column. If the diameters differ by more than 1 ft, it is often more economical to swage the column, using the different diameters computed for the sections above and below the feed.

### Reflux Drums

Almost all commercial towers are provided with a cylindrical reflux drum, as shown in Figure 7.1. This drum is usually located near ground level, necessitating a pump to lift the reflux to the top of the column. If a partial condenser is used, the drum is often oriented vertically to facilitate the separation of vapor from liquid—in effect, acting as a flash drum. Vertical reflux and flash drums are sized by calculating a minimum drum diameter,  $D_T$ , to prevent liquid carryover by entrainment, using (6-44) in conjunction with the curve for 24-in. tray spacing in Figure 6.24 and a value of  $F_{HA} = 1.0$  in (6-42). Also,  $f = 0.85$  and  $A_d = 0$  are used. To absorb process upsets and fluctuations, and otherwise facilitate control, vessel volume,  $V_V$ , is determined on the basis of liquid residence time,  $t$ , which should be at least 5 min, with the

vessel half full of liquid [20]:

$$V_V = \frac{2LM_L t}{\rho_L} \quad (7-44)$$

where  $L$  is the molar liquid flow rate leaving the vessel. Assuming a vertical, cylindrical vessel and neglecting the volume associated with the heads, the height  $H$  of the vessel is

$$H = \frac{4V_V}{\pi D_T^2} \quad (7-45)$$

However, if  $H > 4D_T$ , it is generally preferable to increase  $D_T$  and decrease  $H$  to give  $H = 4D_T$ . Then

$$D_T = \frac{H}{4} = \left( \frac{V_V}{\pi} \right)^{1/3} \quad (7-46)$$

A height above the liquid level of at least 4 ft is necessary for feed entry and disengagement of liquid droplets from the vapor. Within this space, it is common to install a wire mesh pad, which serves as a mist eliminator.

When vapor is totally condensed, a cylindrical, horizontal reflux drum is commonly employed to receive the condensate. Equations (7-44) and (7-46) permit estimates of the drum diameter,  $D_T$ , and length,  $H$ , by assuming a near-optimal value for  $H/D_T$  of 4, and the same liquid residence time suggested for a vertical drum. A horizontal drum is also used following a partial condenser when the liquid flow rate is appreciably greater than the vapor flow rate.

**EXAMPLE 7.8**

Equilibrium vapor and liquid streams leaving a flash drum, supplied by a partial condenser, are as follows:

Component	Vapor	Liquid
Pound-moles per hour:		
HCl	49.2	0.8
Benzene	118.5	81.4
Monochlorobenzene	71.5	178.5
Total	239.2	260.7
Pounds per hour	19,110	26,480
$T$ , °F	270	270
$P$ , psia	35	35
Density, lb/ft <sup>3</sup>	0.371	57.08

Determine the dimensions of the flash drum.

**SOLUTION**

Using Figure 6.24,

$$F_{LV} = \frac{26,480}{19,110} \left( \frac{0.371}{57.08} \right)^{0.5} = 0.112$$

$C_F$  at a 24-in. tray spacing is 0.34. Assume, in (6-24), that  $C = C_F$ . From (6-40),

$$U_f = 0.34 \left( \frac{57.08 - 0.371}{0.371} \right)^{0.5} = 4.2 \text{ ft/s} = 15,120 \text{ ft/h}$$

From (6-44) with  $A_d/A = 0$ ,

$$D_T = \left[ \frac{(4)(19,110)}{(0.85)(15,120)(3.14)(1)(0.371)} \right]^{0.5} = 2.26 \text{ ft}$$

From (7-44), with  $t = 5 \text{ min} = 0.0833 \text{ h}$ ,

$$V_V = \frac{(2)(26,480)(0.0833)}{(57.08)} = 77.3 \text{ ft}^3$$

From (7-43),

$$H = \frac{(4)(77.3)}{(3.14)(2.26)^2} = 19.3 \text{ ft}$$

However,  $H/D_T = 19.3/2.26 = 8.54 > 4$ . Therefore, redimension  $V_V$  for  $H/D_T = 4$ .

From (7-46),

$$D_T = \left( \frac{77.3}{3.14} \right)^{1/3} = 2.91 \text{ ft and } H = 4D_T = (4)(2.91) = 11.64 \text{ ft}$$

Height above the liquid level is  $11.64/2 = 5.82 \text{ ft}$ , which is adequate.

Alternatively, with a height of twice the minimum disengagement height,  $H = 8 \text{ ft}$  and  $D_T = 3.5 \text{ ft}$ .

## 7.5 RATE-BASED METHOD FOR PACKED COLUMNS

With the availability of more efficient liquid distributors and economical and efficient packings, packed towers are finding increasing use in new distillation processes and for

**Table 7.6** Modified Efficiency and Mass-Transfer Equations for Binary Distillation

$$\lambda = mV/L \quad (7-47)$$

$$m = dy/dx = \text{local slope of equilibrium curve}$$

Efficiency:

Equations (6.31) to (6.37) hold if  $\lambda$  is defined by (7.47)

Mass transfer:

$$\frac{1}{N_{OG}} = \frac{1}{N_G} + \frac{\lambda}{N_L} \quad (7-48)$$

$$\frac{1}{K_{OG}} = \frac{1}{k_G a} + \frac{m P M_L / \rho_L}{k_L a} \quad (7-49)$$

$$\frac{1}{K_y a} = \frac{1}{k_y a} + \frac{m}{k_x a} \quad (7-50)$$

$$\frac{1}{K_x a} = \frac{1}{k_x a} + \frac{1}{m k_y a} \quad (7-51)$$

$$H_{OG} = H_G + \lambda H_L \quad (7-52)$$

$$\text{HETP} = H_{OG} \ln \lambda / (\lambda - 1) \quad (7-53)$$

retrofitting existing trayed towers. Methods in Section 6.8 for estimating packed-column efficiency, diameter, and pressure drop for absorbers are applicable to distillation. Methods for determining packed height are similar to those presented in Section 6.7 and are extended here for use in conjunction with the McCabe–Thiele diagram. Both the HETP and the HTU methods are discussed and illustrated. Unlike the case of absorption or stripping of dilute solutions, where values of HETP and HTU may be constant throughout the packed height, values of HETP and HTU can vary over the packed height of a distillation column, especially across the feed entry, where appreciable changes in vapor and liquid traffic occur. Also, because the equilibrium line for distillation is curved rather than straight, the mass-transfer equations of Section 6.8 must be modified by replacing  $\lambda = KV/L = 1/A$  with

$$\lambda = \frac{mV}{L} = \frac{\text{slope of equilibrium curve}}{\text{slope of operating line}}$$

where  $m = dy/dx$  varies with location in the tower. The modified efficiency and mass-transfer relationships are summarized in Table 7.6.

### HETP Method

In the HETP method, the equilibrium stages are first stepped off on a McCabe–Thiele diagram. The case of equimolar counterdiffusion (EMD) applies to distillation. At each stage, the temperature, pressure, phase-flow ratio, and phase compositions are noted. A suitable packing material is selected and the column diameter is estimated for operation at, say, 70% of flooding by one of the methods of Section 6.8. Mass-transfer coefficients for the individual phases are estimated for the conditions at each stage from correlations also

discussed in Section 6.8. From these coefficients, values of  $H_{OG}$  and HETP are estimated for each stage. The latter values are then summed to obtain the separate packed heights of the rectifying and stripping sections. If experimental values of HETP are available, they are used directly. In computing values of  $H_{OG}$  from  $H_G$  and  $H_L$ , or  $K_y$  from  $k_y$  and  $k_x$ , (6-92) and (6-80) must be modified because for binary distillation where the mole fraction of the light key may range from almost 0 at the bottom of the column to almost 1 at the top of the column, the ratio  $(y_1 - y^*)/(x_1 - x)$  in (6-76) is no longer a constant equal to the  $K$ -value, but is  $dy/dx$  equal to the slope,  $m$ , of the equilibrium curve. The modified equations are included in Table 7.6.

### EXAMPLE 7.9

For the benzene–toluene distillation of Example 7.1, determine packed heights of the rectifying and stripping sections based on a column diameter and packing material with the following values for the individual HTUs. Included are the  $L/V$  values for each section from Example 7.1.

	$H_G$ , ft	$H_L$ , ft	$L/V$
Rectifying section	1.16	0.48	0.62
Stripping section	0.90	0.53	1.40

### SOLUTION

Slopes  $dy/dx$  of the equilibrium curve are obtained from Figure 7.15 and values of  $\lambda$  from (7-47).  $H_{OG}$  for each stage is determined from (7-52) in Table 7.6. HETP for each stage is determined from (7-53) in Table 7.6. The results are given in Table 7.7, where only 0.2 of stage 13 is needed and stage 14 is the partial reboiler.

Based on the results in Table 7.7, 10 ft of packing should be used in each of the two sections.

**Table 7.7** Results for Example 7.9

Stage	$m$	$\lambda = \frac{mV}{L}$ or $m\frac{\bar{V}}{\bar{L}}$	$H_{OG}$ , ft	HETP, ft
1	0.47	0.76	1.52	1.74
2	0.53	0.85	1.56	1.70
3	0.61	0.98	1.62	1.64
4	0.67	1.08	1.68	1.62
5	0.72	1.16	1.71	1.59
6	0.80	1.29	1.77	1.56
Total for rectifying section:				9.85
7	0.90	0.64	1.32	1.64
8	0.98	0.70	1.28	1.52
9	1.15	0.82	1.34	1.47
10	1.40	1.00	1.43	1.43
11	1.70	1.21	1.53	1.40
12	1.90	1.36	1.62	1.38
13	2.20	1.57	1.73	$1.37(0.2) = 0.27$
Total for stripping section:				9.11
Total packed height:				18.96

### HTU Method

In the HTU method, equilibrium stages are not stepped off on a McCabe–Thiele diagram. Instead, the diagram provides data to perform an integration over the packed height of each section using either mass-transfer coefficients or transfer units.

Consider the schematic diagram of a packed distillation column and its accompanying McCabe–Thiele diagram in Figure 7.34. Assume that  $V$ ,  $L$ ,  $\bar{V}$ , and  $\bar{L}$  are constant in their respective sections. For equimolar countercurrent diffusion (EMD), the rate of mass transfer of the light-key component from the liquid phase to the vapor phase is

$$n = k_x a(x - x_1) = k_y a(y_1 - y) \quad (7-54)$$

Rearranging:

$$-\frac{k_x a}{k_y a} = \frac{y_1 - y}{x_1 - x} \quad (7-55)$$

Thus, as shown in Figure 7.34b, for any point  $(x, y)$  on the operating line, the corresponding interfacial point  $(x_1, y_1)$  on the equilibrium curve is obtained by drawing a line of slope  $(-k_x a/k_y a)$  from the point  $(x, y)$  to the point where it intersects the equilibrium curve.

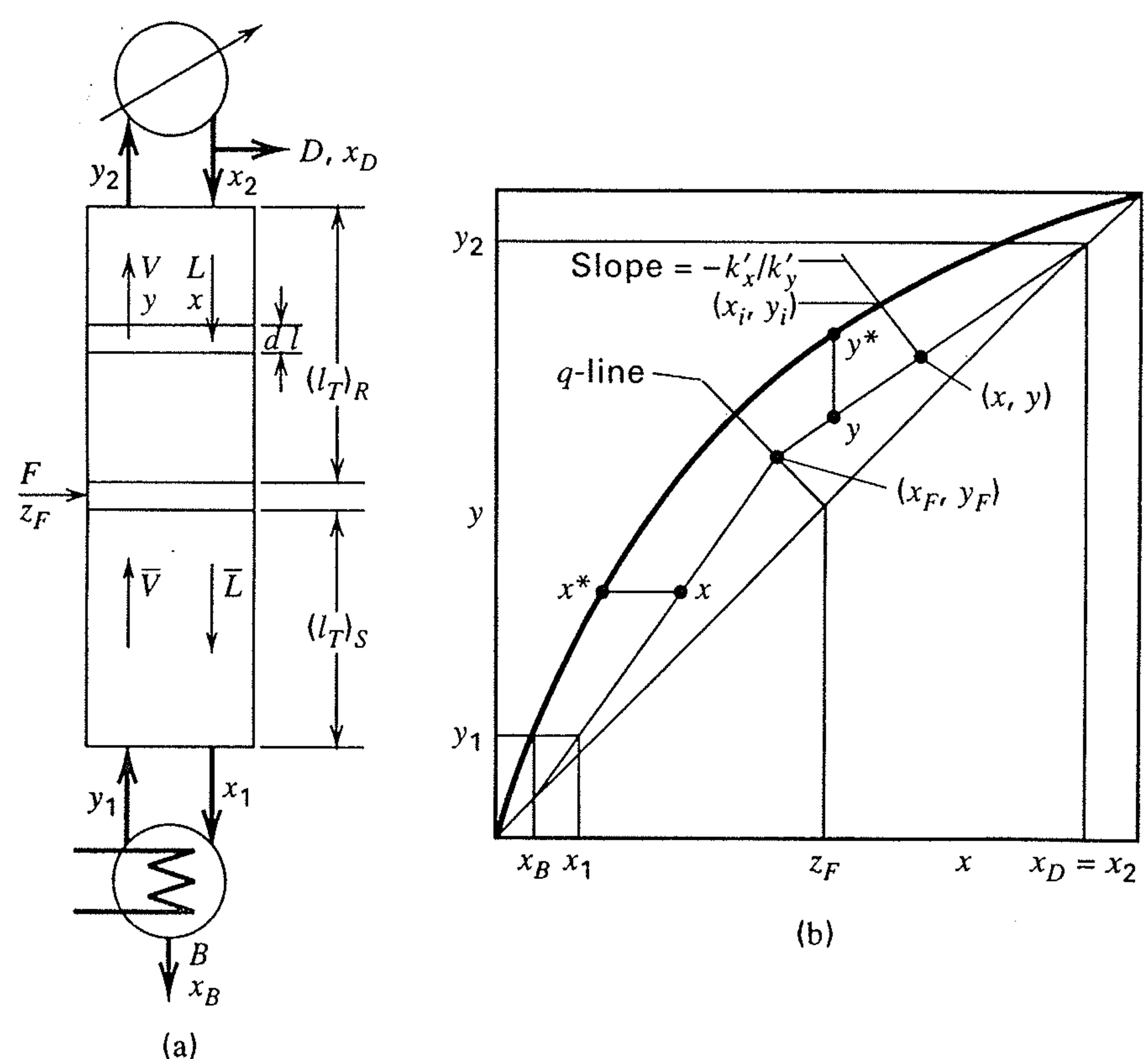
By material balance over an incremental section of packed height, assuming constant molar overflow,

$$V dy = k_y a(y_1 - y) S dl \quad (7-56)$$

$$L dx = k_x a(x - x_1) S dl \quad (7-57)$$

where  $S$  is the cross-sectional area of the packed section. Integrating over the rectifying section,

$$(l_T)_R = \int_0^{(l_T)_R} dl = \int_{y_F}^{y_2} \frac{V dy}{k_y a S (y_1 - y)} = \int_{x_F}^{x_D} \frac{L dx}{k_x a S (x - x_1)} \quad (7-58)$$



**Figure 7.34** Distillation in a packed column.

or

$$(l_T)_R = \int_{y_F}^{y_2} \frac{H_G dy}{(y_1 - y)} = \int_{x_F}^{x_D} \frac{H_L dx}{(x - x_1)} \quad (7-59)$$

Integrating over the stripping section,

$$(l_T)_S = \int_0^{(l_T)_S} dl = \int_{y_1}^{y_F} \frac{V dy}{k_y a S (y_1 - y)} = \frac{L dx}{k_x a S (x - x_1)} \quad (7-60)$$

or

$$(l_T)_S = \int_{y_1}^{y_F} \frac{H_G dy}{(y_1 - y)} = \int_{x_1}^{x_F} \frac{H_L dx}{(x - x_1)} \quad (7-61)$$

In general, values of  $k_y$  and  $k_x$  vary over the packed height, causing the slope ( $-k_x a / k_y a$ ) to vary. If  $k_x a > k_y a$ , the main resistance to mass transfer resides in the vapor and it is most accurate to evaluate the integrals in  $y$ . For  $k_y a > k_x a$ , the integrals in  $x$  are used. Usually, it is sufficient to evaluate  $k_y$  and  $k_x$  at just three points in each section, from which their variation with  $x$  can be determined. Then by computing and plotting their ratios from (7-55), a locus of points P can be found, from which values of  $(y_1 - y)$  for any value of  $y$ , or  $(x - x_1)$  for any value of  $x$  can be read for use in integrals (7-58) to (7-61). These integrals can be evaluated either graphically or numerically to determine the packed heights.

### EXAMPLE 7.10

Suppose that 250 kmol/h of a mixture of 40 mol% isopropyl ether in isopropanol is distilled in a packed column operating at 1 atm to obtain a distillate of 75 mol% isopropyl ether and a bottoms of 95 mol% isopropanol. At the feed entry, the mixture is a saturated liquid. A reflux ratio of 1.5 times minimum is used and the column is equipped with a total condenser and a partial reboiler. For the packing and column diameter, mass-transfer coefficients given below have been estimated from empirical correlations of the type discussed in Section 6.8. Compute the required packed heights of the rectifying and stripping sections.

### SOLUTION

The distillate and bottoms rates are computed by an overall material balance on isopropyl ether:

$$0.40(250) = 0.75D + 0.05(250 - D)$$

$$\text{Solving, } D = 125 \text{ kmol/h and } B = 250 - 125 = 125 \text{ kmol/h}$$

The equilibrium curve for this mixture at 1 atm is shown in Figure 7.35, where it is noted that isopropyl ether is the light key and an azeotrope is formed at 78 mol% isopropyl ether. The distillate composition of 75 mol% is safely below the azeotropic composition. Also shown in Figure 7.35 are the  $q$ -line and the rectification-section operating line for the condition of minimum reflux. The slope of the latter line is measured to be  $(L/V)_{\min} = 0.39$ . From (7-27),

$$R_{\min} = 0.39 / (1 - 0.39) = 0.64 \quad \text{and} \quad R = 1.5 R_{\min} = 0.96$$

$$L = RD = 0.96(125) = 120 \text{ kmol/h}$$

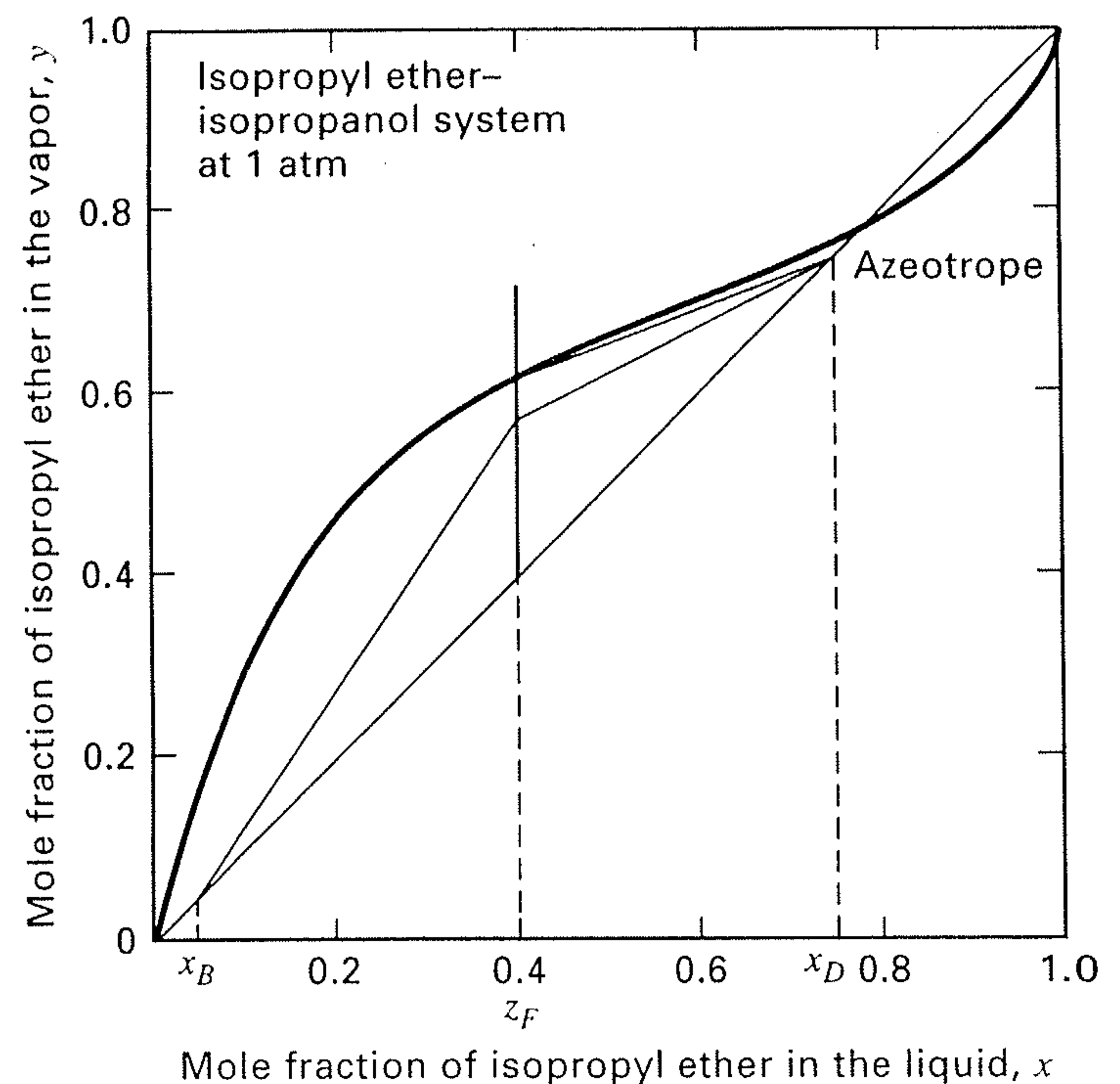


Figure 7.35 Operating lines and minimum-reflux line for Example 7.10.

and  $V = L + D = 120 + 125 = 245 \text{ kmol/h}$

$$\bar{L} = L + L_F = 120 + 250 = 370 \text{ kmol/h}$$

and  $\bar{V} = V - V_F = 245 - 0 = 245 \text{ kmol/h}$

Slope of rectification-section operating line  $= L/V = 120/245 = 0.49$

This line and the stripping-section operating line are plotted in Figure 7.35. The partial reboiler, R, is stepped off in Figure 7.36 to give the following end points for determining the packed heights of the two sections, where the symbols refer to Figure 7.34a:

	Stripping Section	Rectifying Section
Top	$(x_F = 0.40, y_F = 0.577)$	$(x_2 = 0.75, y_2 = 0.75)$
Bottom	$(x_1 = 0.135, y_1 = 0.18)$	$(x_F = 0.40, y_F = 0.577)$

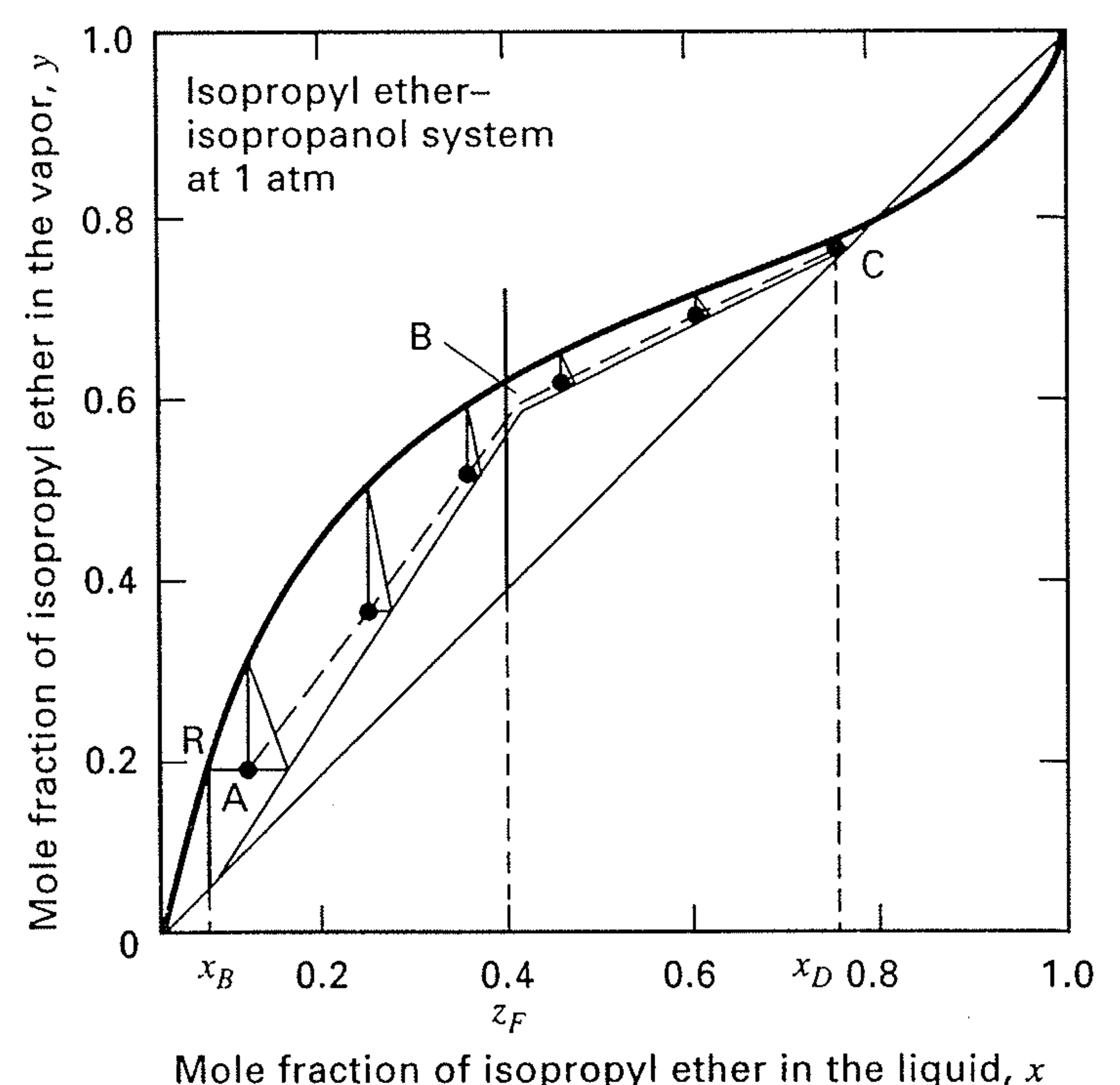


Figure 7.36 Mass-transfer driving forces for Example 7.10.

Mass-transfer coefficients at three values of  $x$  in each section are as follows:

$x$	$k_y a$ kmol/m <sup>3</sup> -h-(mole fraction)	$k_x a$ kmol/m <sup>3</sup> -h-(mole fraction)
Stripping section:		
0.15	305	1,680
0.25	300	1,760
0.35	335	1,960
Rectifying section:		
0.45	185	610
0.60	180	670
0.75	165	765

Slopes of the above mass-transfer coefficients are computed, for each point,  $x$ , on the operating line using (7-55), and drawn from the operating line to the equilibrium line, as shown in Figure 7.36. These lines are often referred to as tie lines because they tie the operating line to the equilibrium line. Using the tie lines as hypotenuses, right triangles are drawn, as shown in Figure 7.36. Dashed locus lines, AB and BC, are then drawn through the points at the 90° corners of the triangles. Using these locus lines, additional tie lines can quickly be added to the three plotted in each section, as needed, to give sufficient accuracy. From the tie lines, values of  $(y_1 - y)$  can be tabulated for values of  $y$  on the operating lines. Since the diameter of the column is not given, the packed volumes are determined from the following rearrangements of (7-58) and (7-60), where  $V = Sl_T$ :

$$V_R = \int_{y_F}^{y_2} \frac{V dy}{k_y a (y_1 - y)} \quad (7-62)$$

$$V_S = \int_{y_1}^{y_F} \frac{V dy}{k_y a (y_1 - y)} \quad (7-63)$$

Values of  $k_y a$  are interpolated as necessary. Results are given in the following table.

$y$	$(y_1 - y)$	$k_y a$	$\frac{V(\text{or } \bar{V})}{k_y a (y_1 - y)}, \text{m}^3$
Stripping section:			
0.18	0.145	307	5.5
0.25	0.150	303	5.4
0.35	0.143	300	5.7
0.45	0.103	320	7.4
0.577	0.030	350	23.3
Rectifying section:			
0.577	0.030	187	43.7
0.60	0.033	185	40.1
0.65	0.027	182	49.9
0.70	0.017	175	82.3
0.75	0.010	165	148.5

By numerical integration,  $V_S = 3.6 \text{ m}^3$  and  $V_R = 12.3 \text{ m}^3$ .

## 7.6 PONCHON–SAVARIT GRAPHICAL EQUILIBRIUM-STAGE METHOD FOR TRAYED TOWERS

The McCabe–Thiele method, in Section 7.2 for binary distillation, assumes that molar vapor and liquid flow rates are constant in each section of the column. This assumption

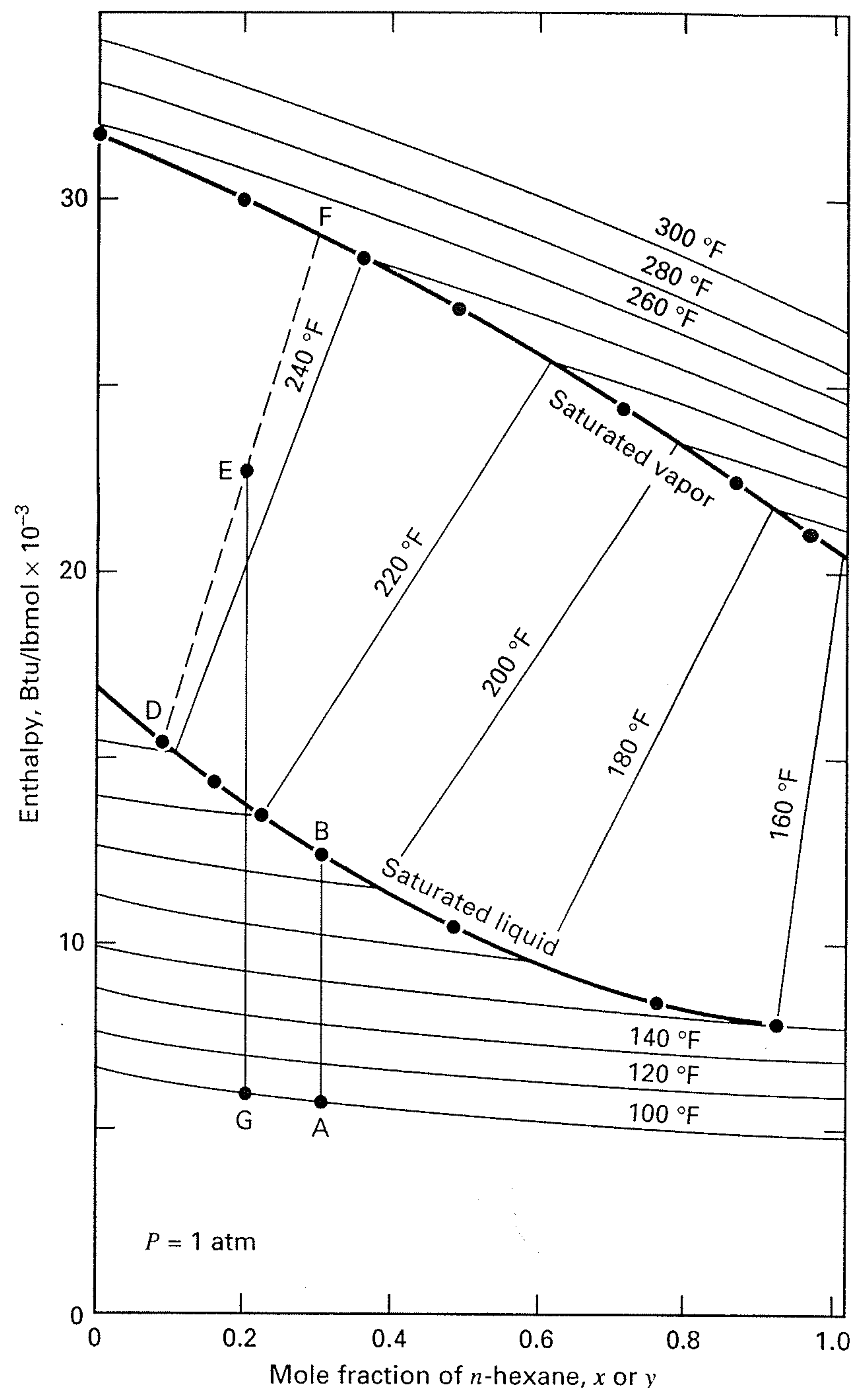


Figure 7.37 Enthalpy–concentration diagram for  $n$ -hexane/ $n$ -octane.

(constant molar overflow) eliminates the need to make an energy balance around each stage. For nonideal binary mixtures, such an assumption may not be valid and the McCabe–Thiele method may not be accurate. A graphical method that includes energy balances as well as material balances and phase equilibrium relations is the Ponchon–Savarit method [21, 22], which utilizes an enthalpy–composition diagram of the type shown in Figure 7.37 for the  $n$ -hexane/ $n$ -octane system at 1 atm. This diagram includes curves for the enthalpies of saturated vapor and liquid mixtures. Terminal points of tie lines connecting these two curves represent the equilibrium vapor and liquid compositions, together with vapor and liquid enthalpies, for the given temperature. Isotherms above the saturated vapor curve represent enthalpies of the superheated vapor, while isotherms below the saturated liquid curve represent the subcooled liquid. In Figure 7.37, a mixture of 30 mol% hexane and 70 mol% octane at 100°F (Point A) is a subcooled liquid. By heating it to Point B at 204°F, it becomes a liquid at its bubble point (Point B). When a mixture of 20 mol% hexane and 80 mol% octane at 100°F (Point G) is heated to 243°F (Point E), at equilibrium, it splits into a vapor phase at Point F and a liquid phase at Point D. The



liquid phase contains 7 mol% hexane, while the vapor contains 29 mol% hexane.

The application of the enthalpy–concentration diagram to equilibrium-stage calculations may be illustrated by considering a single equilibrium stage,  $n - 1$ , where vapor from stage  $n - 2$  below is mixed adiabatically with liquid from stage  $n$  above to give an overall mixture, denoted by mole-fraction  $z$ , and then brought to equilibrium. The process is represented schematically in two steps, mixing followed by equilibration, at the top of Figure 7.38. The energy-balance equations for stage  $n - 1$  are

Mixing:

$$V_{n-2}H_{n-2} + L_n h_n = (V_{n-2} + L_n)h_z \quad (7-64)$$

Equilibration:

$$(V_{n-2} + L_n)h_z = V_{n-1}H_{n-1} + L_{n-1}h_{n-1} \quad (7-65)$$

where  $H$  and  $h$  are vapor and liquid molar enthalpies, respectively. The governing material-balance equations for the light component are

Mixing:

$$y_{n-2}V_{n-2} + x_n L_n = z(V_{n-2} + L_n) \quad (7-66)$$

Equilibration:

$$z(V_{n-2} + L_n) = y_{n-1}V_{n-1} + x_{n-1}L_{n-1} \quad (7-67)$$

Simultaneous solution of (7-64) and (7-66) gives

$$\frac{H_{n-2} - h_z}{y_{n-2} - z} = \frac{h_z - h_n}{z - x_n} \quad (7-68)$$

which is the three-point form of a straight line plotted in Figure 7.38. Similarly, the simultaneous solution of (7-65) and (7-67) gives

$$\frac{H_{n-1} - h_z}{y_{n-1} - z} = \frac{h_z - h_{n-1}}{z - x_{n-1}} \quad (7-69)$$

which is also the equation for a straight line. However, in this case  $y_{n-1}$  and  $x_{n-1}$  are in equilibrium and, therefore, the points  $(H_{n-1}, y_{n-1})$  and  $(h_{n-1}, x_{n-1})$  must lie on the

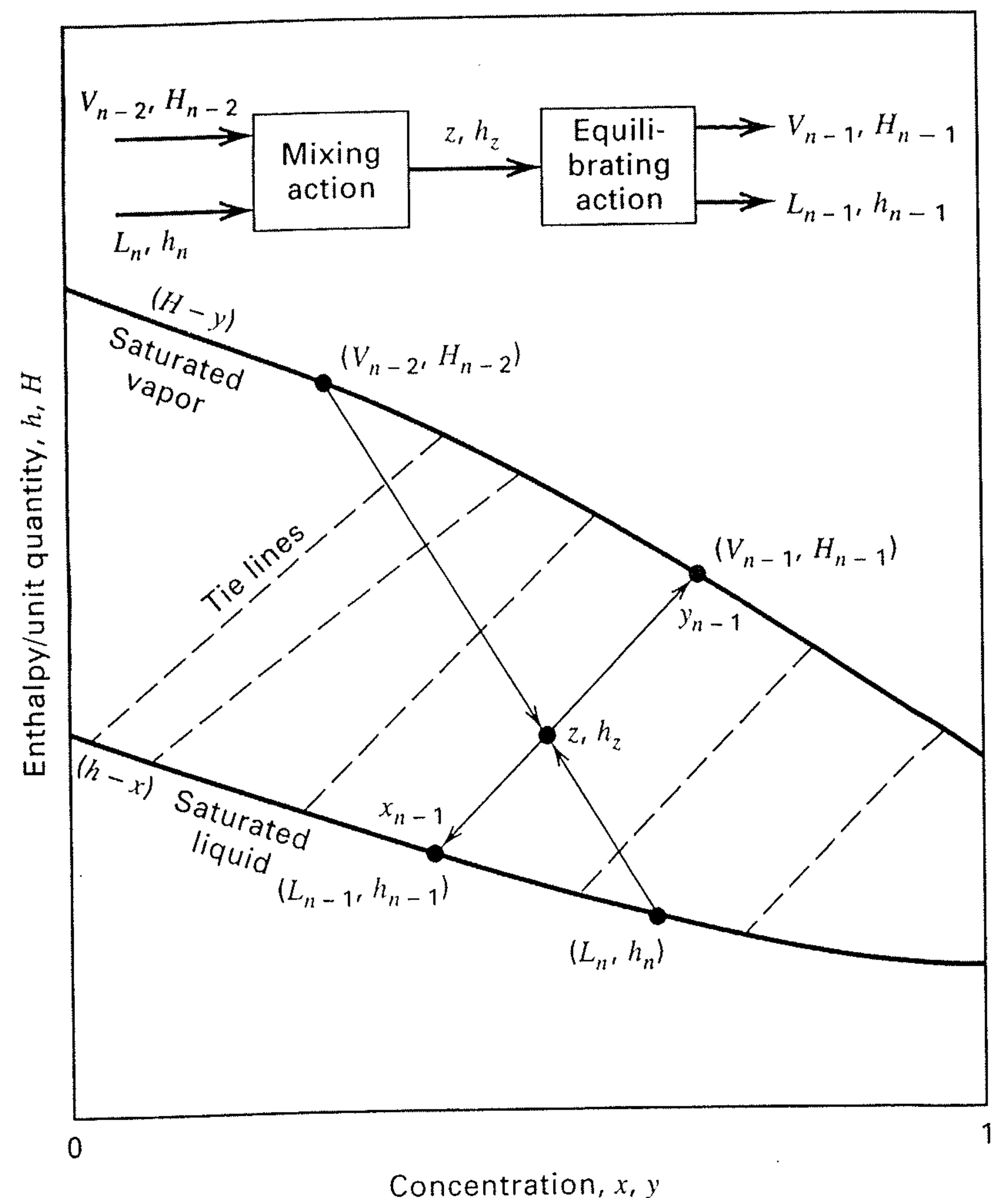


Figure 7.38 Two-phase mixing and equilibration on an enthalpy–concentration diagram.

opposite ends of the tie line that passes through the mixing point  $(h_z, z)$ , as shown in Figure 7.38.

The Ponchon–Savarit method for binary distillation is an extension of the construction in Figure 7.38 to countercurrent cascades above and below the feed stage, with consideration of the condenser and reboiler. A detailed description of the method is not given here because the method has been largely superseded by the rigorous computer-aided calculation procedures, discussed in Chapter 10, which include energy balances and can be applied to multicomponent as well as binary mixtures. A detailed presentation of the Ponchon–Savarit method for binary distillation is given by Henley and Seader [23].

## SUMMARY

1. A binary-liquid and/or binary-vapor mixture can be separated economically into two nearly pure products (distillate and bottoms) by distillation, provided that the value of the relative volatility of the two components is high enough, usually greater than 1.05.
2. Distillation is the most mature and widely used separation operation, with design procedures and operation practices well established.
3. The purities of the products from distillation depend on the number of equilibrium stages in the rectifying section above the feed entry and in the stripping section below the feed entry, and on the reflux ratio. Both the number of stages and the reflux ratio must be greater than the minimum values corresponding to total reflux and infinite stages, respectively. The optimal reflux-to-minimum-reflux ratio is usually in the range of 1.10 to 1.50.

4. Distillation is most commonly conducted in trayed towers equipped with sieve or valve trays, or in columns packed with random or structured packings. Many older towers are equipped with bubble-cap trays.
5. Most distillation towers are equipped with a condenser, cooled with cooling water, to provide reflux, and a reboiler, heated with steam, to provide boilup.
6. When the assumption of constant molar overflow is valid in each of the two sections of the distillation tower, the McCabe–Thiele graphical method is convenient for determining stage and reflux requirements. This method facilitates the visualization of many aspects of distillation and provides a procedure for locating the optimal feed-stage location.

7. Miscellaneous considerations involved in the design of a distillation tower include selection of operating pressure, type of condenser, degree of reflux subcooling, type of reboiler, and extent of feed preheat.

8. The McCabe–Thiele method can be extended to handle Murphree stage efficiency, multiple feeds, side streams, open steam, and use of interreboilers and intercondensers.

9. Rough estimates of overall stage efficiency, defined by (6-21), can be made with the Drickamer and Bradford, (7-42), or O'Connell, (7-43), correlations. More accurate and reliable procedures use data from a small Oldershaw column or the same semi-theoretical equations for mass transfer in Chapter 6 that are used for absorption and stripping.

10. Tray diameter, pressure drop, weeping, entrainment, and downcomer backup can all be estimated by the procedures in Chapter 6.

11. Reflux and flash drums are sized by a procedure based on avoidance of entrainment and provision for adequate liquid residence time.

12. Packed-column diameter and pressure drop are determined by the same procedures presented in Chapter 6 for absorption and stripping.

13. The height of a packed column may be determined by the HETP method, or preferably from the HTU method. Application of the latter method is similar to that of Chapter 6 for absorbers and strippers, but differs in the manner in which the curved equilibrium line must be handled, as given by (7-47).

14. The Ponchon–Savarit graphical method removes the assumption of constant molar overflow in the McCabe–Thiele method by employing energy balances with an enthalpy-concentration diagram. However, use of the Ponchon–Savarit method has largely been supplanted by rigorous computer-aided methods.

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## EXERCISES

Unless otherwise stated, the usual simplifying assumptions of saturated-liquid reflux, optimal feed-stage location, no heat losses, steady state, and constant molar liquid and vapor flows apply to each of the following problems.

### Section 7.1

7.1 List as many differences between absorption and distillation as you can. List as many differences between stripping and distillation as you can.

7.2 Prior to the 1980s, packed columns were rarely used for distillation unless column diameter was less than 2.5 ft. Explain why, in recent years, some existing trayed towers are being retrofitted with packing and some new large-diameter columns are being designed for packing rather than trays.

7.3 A mixture of methane and ethane is to be separated by distillation. Explain why water cannot be used as the coolant in the condenser. What would you choose as the coolant?

7.4 A mixture of ethylene and ethane is to be separated by distillation. Determine the maximum operating pressure of the column. What operating pressure would you suggest? Why?

7.5 Under what circumstances would it be advisable to conduct laboratory or pilot-plant tests of a proposed distillation separation?

7.6 Explain why an economic tradeoff exists between the number of trays and the reflux ratio.

### Section 7.2

7.7 Following the development by Sorel in 1894 of a mathematical model for continuous, steady-state, equilibrium-stage

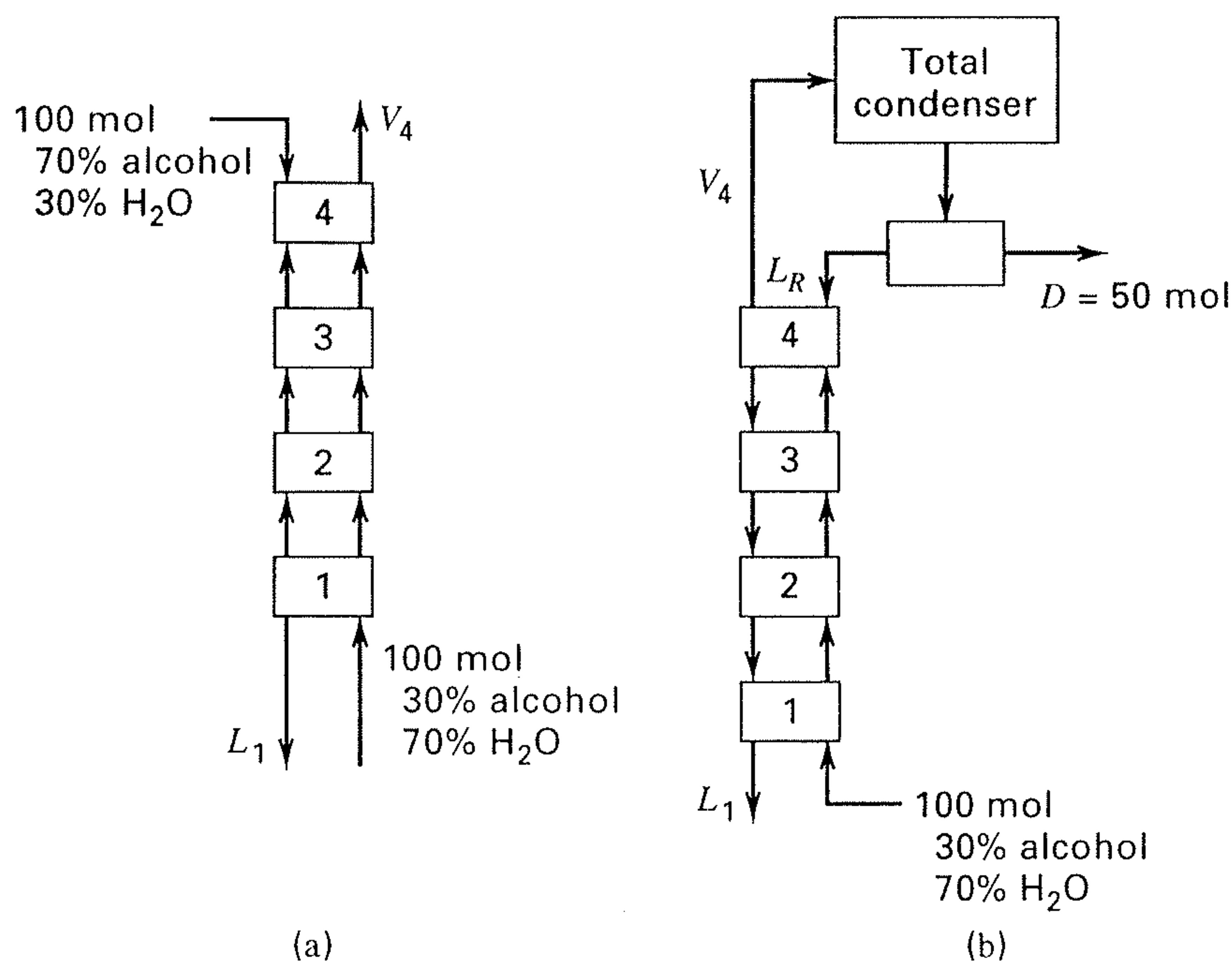


Figure 7.39 Data for Exercise 7.8.

distillation, a number of methods were proposed for solving the equations graphically or algebraically during an 18-year period from 1920 to 1938, prior to the availability of digital computers. Today, the only method from that era that remains in widespread use is the McCabe–Thiele method. What are the attributes of this method that are responsible for its continuing popularity?

**7.8** (a) For the cascade shown in Figure 7.39a, calculate the compositions of streams  $V_4$  and  $L_1$ . Assume atmospheric pressure, saturated liquid and vapor feeds, and the vapor–liquid equilibrium data given below. Compositions are in mole percent.

(b) Given the feed compositions in cascade (a), how many equilibrium stages are required to produce a  $V_4$  containing 85 mol% alcohol?

(c) For the cascade configuration shown in Figure 7.39b, with  $D = 50$  mol, what are the compositions of  $D$  and  $L_1$ ?

(d) For the configuration of cascade (b), how many equilibrium stages are required to produce a  $D$  of 50 mol% alcohol?

#### EQUILIBRIUM DATA, MOLE-FRACTION ALCOHOL

$x$	0.1	0.3	0.5	0.7	0.9
$y$	0.2	0.5	0.68	0.82	0.94

**7.9** Liquid air is fed to the top of a perforated-tray reboiled stripper operated at substantially atmospheric pressure. Sixty percent of the oxygen in the feed is to be drawn off in the bottoms vapor product from the still. This product is to contain 0.2 mol% nitrogen. Based on the assumptions and data given below, calculate:

- The mole percent of nitrogen in the vapor leaving the top plate.
- The moles of vapor generated in the still per 100 mol of feed.
- The number of theoretical plates required.

Notes: To simplify the problem, assume constant molar overflow equal to the moles of feed. Liquid air contains 20.9 mol% of oxygen and 79.1 mol% of nitrogen. The equilibrium data [*Chem. Met. Eng.*, **35**, 622 (1928)] at atmospheric pressure are

Temperature, K	Mole-Percent N <sub>2</sub> in Liquid	Mole-Percent N <sub>2</sub> in Vapor
77.35	100.00	100.00
77.98	90.00	97.17
78.73	79.00	93.62

Temperature, K	Mole-Percent N <sub>2</sub> in Liquid	Mole-Percent N <sub>2</sub> in Vapor
79.44	70.00	90.31
80.33	60.00	85.91
81.35	50.00	80.46
82.54	40.00	73.50
83.94	30.00	64.05
85.62	20.00	50.81
87.67	10.00	31.00
90.17	0.00	0.00

**7.10** A mixture of A (more volatile) and B is being separated in a plate distillation column. In two separate tests run with a saturated-liquid feed of 40 mol% A, the following compositions, in mol% A, were obtained for samples of liquid and vapor streams from three consecutive stages between the feed and total condenser at the top:

Stage	Mol% A			
	Test 1		Test 2	
	Vapor	Liquid	Vapor	Liquid
$M + 2$	79.5	68.0	75.0	68.0
$M + 1$	74.0	60.0	68.0	60.5
$M$	67.9	51.0	60.5	53.0

Determine the reflux ratio and overhead composition in each case, assuming that the column has more than three stages.

**7.11** A saturated-liquid mixture containing 70 mol% benzene and 30 mol% toluene is to be distilled at atmospheric pressure to produce a distillate of 80 mol% benzene. Five procedures, described below, are under consideration. For each of the procedures, calculate and tabulate:

- Moles of distillate per 100 moles of feed,
- Moles of total vapor generated per mole of distillate,
- Mole percent benzene in the residue, and
- For each part, construct a  $y$ - $x$  diagram. On this, indicate the compositions of the overhead product, the reflux, and the composition of the residue.
- If the objective is to maximize total benzene recovery, which, if any, of these procedures is preferred?

Note: Assume that the relative volatility equals 2.5.

The procedures are as follows:

- Continuous distillation followed by partial condensation. The feed is sent to the direct-heated still pot, from which the residue is continuously withdrawn. The vapors enter the top of a helically coiled partial condenser that discharges into a trap. The liquid is returned (refluxed) to the still, while the residual vapor is condensed as a product containing 80 mol% benzene. The molar ratio of reflux to product is 0.5.
- Continuous distillation in a column containing one equilibrium plate. The feed is sent to the direct-heated still, from which residue is withdrawn continuously. The vapors from the plate enter the top of a helically coiled partial condenser that discharges into a trap. The liquid from the trap is returned to the plate, while the uncondensed vapor is condensed to form a distillate containing 80 mol% benzene. The molar ratio of reflux to product is 0.5.
- Continuous distillation in a column containing the equivalent of two equilibrium plates. The feed is sent to the direct-heated still, from which residue is withdrawn continuously. The

vapors from the top plate enter the top of a helically coiled partial condenser that discharges into a trap. The liquid from the trap is returned to the top plate (refluxed) while the uncondensed vapor is condensed to form a distillate containing 80 mol% benzene. The molar ratio of reflux to product is 0.5.

4. The operation is the same as that described for Procedure 3 with the exception that the liquid from the trap is returned to the bottom plate.
5. Continuous distillation in a column containing the equivalent of one equilibrium plate. The feed at its boiling point is introduced on the plate. The residue is withdrawn continuously from the direct-heated still pot. The vapors from the plate enter the top of a helically coiled partial condenser that discharges into a trap. The liquid from the trap is returned to the plate while the uncondensed vapor is condensed to form a distillate containing 80 mol% benzene. The molar ratio of reflux to product is 0.5.

**7.12** A saturated-liquid mixture of benzene and toluene containing 50 mol% benzene is distilled in an apparatus consisting of a still pot, one theoretical plate, and a total condenser. The still pot is equivalent to one equilibrium stage, and the pressure is 101 kPa.

The still is supposed to produce a distillate containing 75 mol% benzene. For each of the following procedures, calculate, if possible, the number of moles of distillate per 100 moles of feed. Assume a relative volatility of 2.5.

- (a) No reflux with feed to the still pot.
- (b) Feed to the still pot, reflux ratio  $L/D = 3$ .
- (c) Feed to the plate with a reflux ratio of 3.
- (d) Feed to the plate with a reflux ratio of 3. However, in this case, a partial condenser is employed.
- (e) Part (b) using minimum reflux.
- (f) Part (b) using total reflux.

**7.13** A fractionation column operating at 101 kPa is to separate 30 kg/h of a solution of benzene and toluene containing 0.6 mass-fraction toluene into an overhead product containing 0.97 mass-fraction benzene and a bottoms product containing 0.98 mass-fraction toluene. A reflux ratio of 3.5 is to be used. The feed is liquid at its boiling point, feed is to the optimal tray, and the reflux is at saturation temperature.

- (a) Determine the quantity of top and bottom products.
- (b) Determine the number of stages required.

#### EQUILIBRIUM DATA IN MOLE-FRACTION BENZENE, 101 KPA

y	0.21	0.37	0.51	0.64	0.72	0.79	0.86	0.91	0.96	0.98
x	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	0.95

**7.14** A mixture of 54.5 mol% benzene in chlorobenzene at its bubble point is fed continuously to the bottom plate of a column containing two theoretical plates. The column is equipped with a partial reboiler and a total condenser. Sufficient heat is supplied to the reboiler to give  $\bar{V}/F = 0.855$ , and the reflux ratio  $L/V$  in the top of the column is kept constant at 0.50. Under these conditions, what quality of product and bottoms ( $x_D, x_B$ ) can be expected?

#### EQUILIBRIUM DATA AT COLUMN PRESSURE, MOLE-FRACTION BENZENE

x	0.100	0.200	0.300	0.400	0.500	0.600	0.700	0.800
y	0.314	0.508	0.640	0.734	0.806	0.862	0.905	0.943

**7.15** A continuous distillation operation with a reflux ratio ( $L/D$ ) of 3.5 yields a distillate containing 97 wt% B (benzene) and bottoms containing 98 wt% T (toluene). Due to weld failures, the 10 plates in the bottom section of the column are ruined, but the 14 upper plates are intact. It is suggested that the column still be used, with the feed ( $F$ ) as saturated vapor at the dew point, with  $F = 13,600$  kg/h containing 40 wt% B and 60 wt% T. Assuming that the plate efficiency remains unchanged at 50%: (a) Can this column still yield a distillate containing 97 wt% B, (b) How much distillate can we get, and (c) What will the composition of the residue be in mole percent?

For vapor-liquid equilibrium data, see Exercise 7.13.

**7.16** A distillation column having eight theoretical stages (seven in the column + partial reboiler + total condenser) is being used to separate 100 kmol/h of a saturated-liquid feed containing 50 mol% A into a product stream containing 90 mol% A. The liquid-to-vapor molar ratio at the top plate is 0.75. The saturated-liquid feed is introduced on plate 5 from the top. Determine: (a) The composition of the bottoms, (b) The  $\bar{L}/\bar{V}$  ratio in the stripping section, and (c) The moles of bottoms per hour.

Unbeknown to the operators, the bolts holding plates 5, 6, and 7 rust through, and the plates fall into the still pot. If no adjustments are made, what is the new bottoms composition?

It is suggested that, instead of returning reflux to the top plate, an equivalent amount of liquid product from another column be used as reflux. If this product contains 80 mol% A, what now is the composition of: (a) The distillate, and (b) The bottoms.

#### EQUILIBRIUM DATA, MOLE FRACTION OF A

y	0.19	0.37	0.5	0.62	0.71	0.78	0.84	0.9	0.96
x	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9

**7.17** A distillation unit consists of a partial reboiler, a column with seven equilibrium plates, and a total condenser. The feed consists of a 50 mol% mixture of benzene in toluene. It is desired to produce a distillate containing 96 mol% benzene, when operating at 101 kPa.

(a) With saturated-liquid feed fed to the fifth plate from the top, calculate: (1) Minimum reflux ratio  $(L_R/D)_{\min}$ , (2) The bottoms composition, using a reflux ratio  $(L_R/D)$  of twice the minimum, and (3) Moles of product per 100 moles of feed.

(b) Repeat part (a) for a saturated-vapor feed fed to the fifth plate from the top.

(c) With saturated-vapor feed fed to the reboiler and a reflux ratio  $(L/V)$  of 0.9, calculate: (1) Bottoms composition, (2) Moles of product per 100 mole of feed.

Equilibrium data are given in Exercise 7.13.

**7.18** A valve-tray fractionating column containing eight theoretical plates, a partial reboiler equivalent to one theoretical plate, and a total condenser is in operation separating a benzene-toluene mixture containing 36 mol% benzene at 101 kPa. Under normal operating conditions, the reboiler generates 100 kmol of vapor per hour. A request has been made for very pure toluene, and it is proposed to operate this column as a stripper, introducing the feed on the top plate as a saturated liquid, employing the same boilup at the still, and returning no reflux to the column. Equilibrium data are given in Exercise 7.13.

(a) What is the minimum feed rate under the proposed conditions, and what is the corresponding composition of the liquid in the reboiler at the minimum feed?

(b) At a feed rate 25% above the minimum, what is the rate of production of toluene, and what are the compositions in mole percent of the product and distillate?

**7.19** A solution of methanol and water at 101 kPa containing 50 mol% methanol is continuously rectified in a seven-theoretical-plate, perforated-tray column, equipped with a total condenser and a partial reboiler heated by steam.

During normal operation, 100 kmol/h of feed is introduced on the third plate from the bottom. The overhead product contains 90 mol% methanol, and the bottoms product contains 5 mol% methanol. One mole of liquid reflux is returned to the column for each mole of overhead product.

Recently it has been impossible to maintain the product purity in spite of an increase in the reflux ratio. The following test data were obtained:

Stream	kmol/h	mol% alcohol
Feed	100	51
Waste	62	12
Product	53	80
Reflux	94	—

What is the most probable cause of this poor performance? What further tests would you make to establish definitely the reason for the trouble? Could some 90% product be obtained by further increasing the reflux ratio, while keeping the vapor rate constant?

Vapor-liquid equilibrium data at 1 atm [*Chem. Eng. Prog.* 48, 192 (1952)] in mole-fraction methanol are

$x$	0.0321	0.0523	0.075	0.154	0.225	0.349	0.813	0.918
$y$	0.1900	0.2940	0.352	0.516	0.593	0.703	0.918	0.963

**7.20** A fractionating column equipped with a partial reboiler heated with steam, as shown in Figure 7.40, and with a total condenser, is operated continuously to separate a mixture of 50 mol% A and 50 mol% B into an overhead product containing 90 mol% A and a bottoms product containing 20 mol% A. The column has three theoretical plates, and the reboiler is equivalent to one theoretical plate. When the system is operated at an  $L/V = 0.75$  with

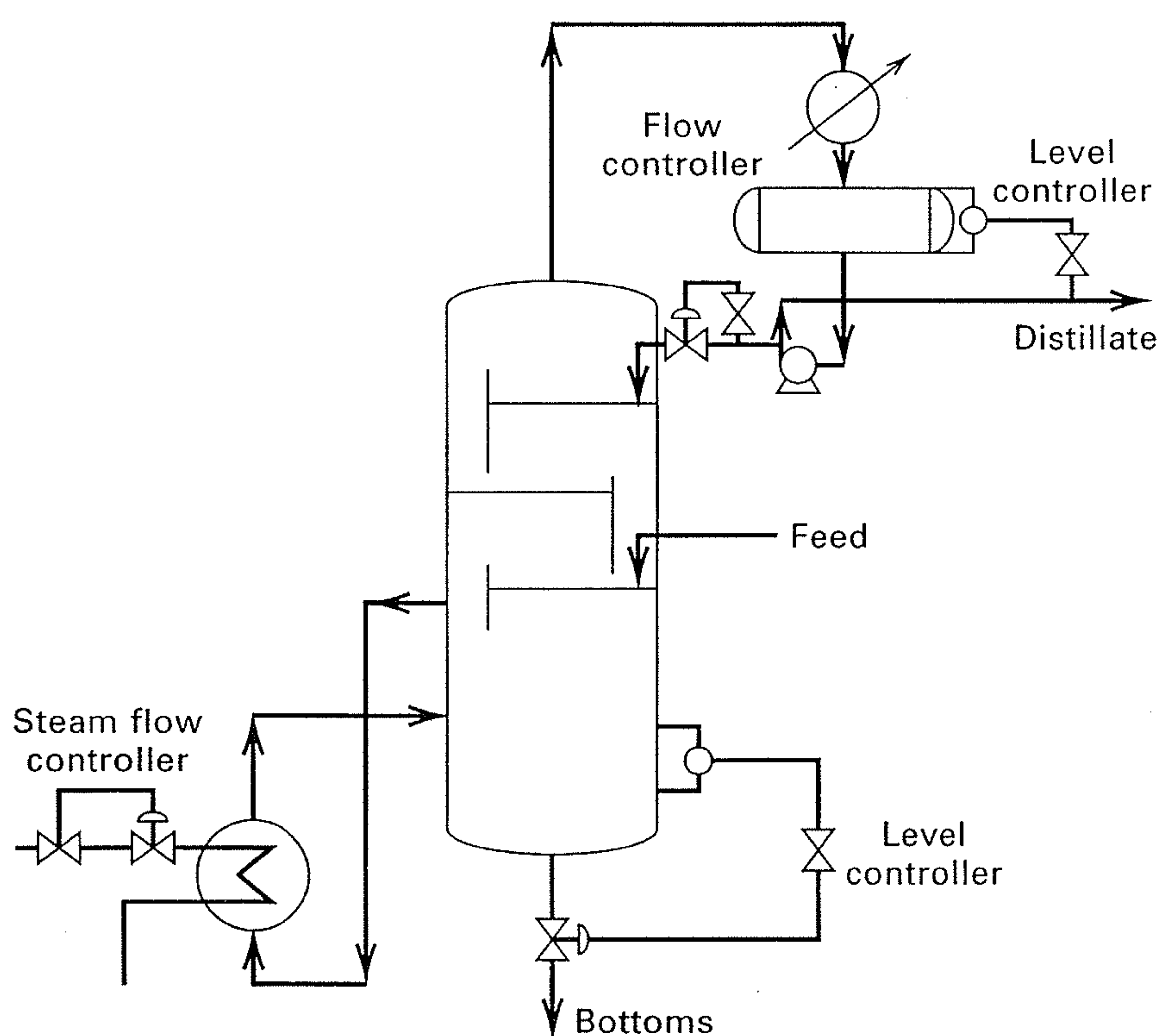


Figure 7.40 Data for Exercise 7.20.

the feed as a saturated liquid to the bottom plate of the column, the desired products can be obtained. The system is instrumented as shown. The steam to the reboiler is controlled by a flow controller so that it remains constant. The reflux to the column is also on a flow controller so that the quantity of reflux is constant. The feed to the column is normally 100 kmol/h, but it was inadvertently cut back to 25 kmol/h. What would be the composition of the reflux, and what would be the composition of the vapor leaving the reboiler under these new conditions? Assume that the vapor leaving the reboiler is not superheated. Relative volatility for the system is 3.0.

**7.21** A saturated-vapor mixture of maleic anhydride and benzoic acid containing 10 mol% acid is a by-product of the manufacture of phthalic anhydride. This mixture is distilled continuously at 13.3 kPa to give a product of 99.5 mol% maleic anhydride and a bottoms of 0.5 mol% anhydride. Using the data below, calculate the number of theoretical plates needed using an  $L/D$  of 1.6 times the minimum.

#### VAPOR PRESSURE, TORR:

Temperature, °C:	10	50	100	200	400
Maleic anhydride	78.7	116.8	135.8	155.9	179.5
Benzoic acid	131.6	167.8	185.0	205.8	227

**7.22** A bubble-point binary mixture containing 5 mol% A in B is to be distilled to give a distillate containing 35 mol% A and a bottoms product containing 0.2 mol% A. If the relative volatility is constant at a value of 6, calculate the following algebraically, assuming that the column will be equipped with a partial reboiler and a partial condenser.

- The minimum number of equilibrium stages
- The minimum boilup ratio  $\bar{V}/B$  leaving the reboiler
- The actual number of equilibrium stages for a boilup ratio equal to 1.2 times the minimum value

**7.23** Methanol (M) is to be separated from water (W) by distillation as shown in Figure 7.41. The feed is subcooled such that  $q = 1.12$ . Determine the feed-stage location and the number of theoretical stages required. Vapor-liquid equilibrium data are given in Exercise 7.19.

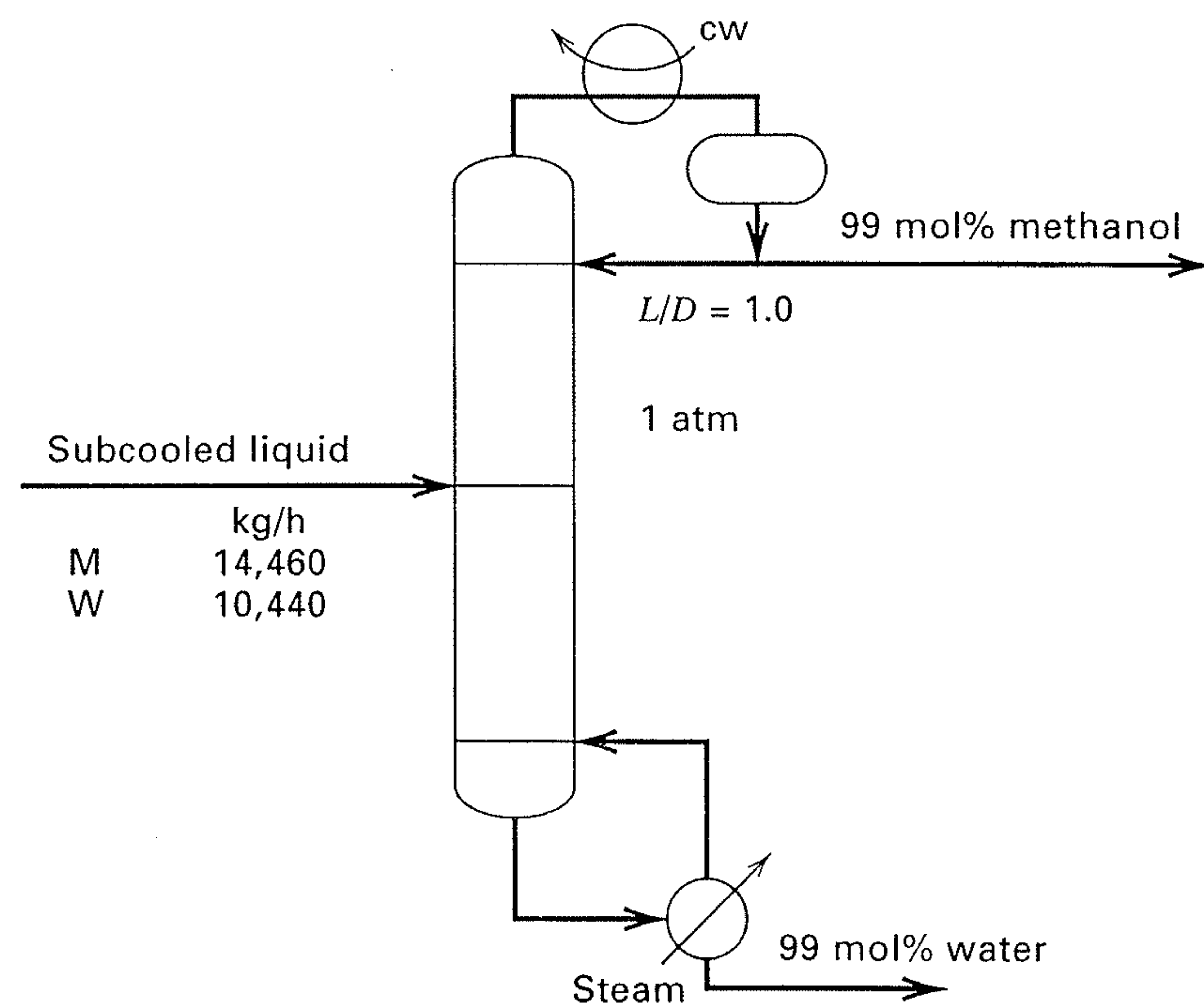


Figure 7.41 Data for Exercise 7.23.

**7.24** A saturated-liquid mixture of 69.4 mol% benzene (B) in toluene (T) is to be continuously distilled at atmospheric pressure to produce a distillate containing 90 mol% benzene, with a yield of 25 moles of distillate per 100 moles of feed. The feed is sent to a steam-heated still (reboiler), where residue is to be withdrawn continuously. The vapors from the still pass directly to a partial condenser. From a liquid separator following the condenser, reflux is returned to the still. Vapors from the separator, which are in equilibrium with the liquid reflux, are sent to a total condenser and are continuously withdrawn as distillate. At equilibrium the mole ratio of B to T in the vapor may be taken as 2.5 times the mole ratio of B to T in the liquid. Calculate analytically and graphically the total moles of vapor generated in the still per 100 mol of feed.

**7.25** A plant has a batch of 100 kmol of a liquid mixture containing 20 mol% benzene and 80 mol% chlorobenzene. It is desired to rectify this mixture at 1 atm to obtain bottoms containing only 0.1 mol% benzene. The relative volatility may be assumed constant at 4.13. There are available a suitable still to vaporize the feed, a column containing the equivalent of four theoretical plates, a total condenser, and a reflux drum to collect the condensed overhead. The run is to be made at total reflux. While the steady state is being approached, a finite amount of distillate is held in a reflux trap. When the steady state is reached, the bottoms contain 0.1 mol% benzene. With this apparatus, what yield of bottoms can be obtained? The liquid holdup in the column is negligible compared to that in the still and in the reflux drum.

**7.26** A mixture of acetone and isopropanol containing 50 mol% acetone is to be distilled continuously to produce an overhead product containing 80 mol% acetone and a bottoms containing 25 mol% acetone. If a saturated-liquid feed is employed, if the column is operated with a reflux ratio of 0.5, and if the Murphree vapor efficiency is 50%, how many trays will be required? Assume a total condenser, partial reboiler, saturated-liquid reflux, and optimal feed stage. The vapor–liquid equilibrium data for this system are

#### EQUILIBRIUM DATA, MOLE-PERCENT ACETONE

Liquid	0	2.6	5.4	11.7	20.7	29.7	34.1	44.0	52.0
Vapor	0	8.9	17.4	31.5	45.6	55.7	60.1	68.7	74.3
Liquid	63.9	74.6	80.3	86.5	90.2	92.5	95.7	100.0	
Vapor	81.5	87.0	89.4	92.3	94.2	95.5	97.4	100.0	

**7.27** A mixture of 40 mol% carbon disulfide (CS<sub>2</sub>) in carbon tetrachloride (CCl<sub>4</sub>) is continuously distilled. The feed is 50% vaporized ( $q = 0.5$ ). The top product from a total condenser is 95 mol% CS<sub>2</sub>, and the bottoms product from a partial reboiler is a liquid of 5 mol% CS<sub>2</sub>.

The column operates with a reflux ratio,  $L/D$ , of 4 to 1. The Murphree vapor efficiency is 80%.

(a) Calculate graphically the minimum reflux, the minimum boilup ratio from the reboiler,  $\bar{V}/B$ , and the minimum number of stages (including reboiler).

(b) How many trays are required for the actual column at 80% efficiency by the McCabe–Thiele method.

The vapor–liquid equilibrium data at column pressure for this mixture in terms of CS<sub>2</sub> mole fraction are

$x$	0.05	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
$y$	0.135	0.245	0.42	0.545	0.64	0.725	0.79	0.85	0.905	0.955

**7.28** A distillation unit consists of a partial reboiler, a bubble-cap column, and a total condenser. The overall plate efficiency is 65%.

The feed is a liquid mixture, at its bubble point, consisting of 50 mol% benzene in toluene. This liquid is fed to the optimal plate. The column is to produce a distillate containing 95 mol% benzene and a bottoms of 95 mol% toluene. Calculate for an operating pressure of 1 atm: (a) Minimum reflux ratio  $(L/D)_{\min}$ , (b) Minimum number of actual plates to carry out the desired separation, (c) Using a reflux ratio  $(L/D)$  of 50% more than the minimum, the number of actual plates needed, (d) The kilograms per hour of product and residue, if the feed is 907.3 kg/h, (e) The saturated steam at 273.7 kPa required in kilograms per hour for heat to the reboiler using enthalpy data below and any assumptions necessary, and (f) A rigorous enthalpy balance on the reboiler, using the enthalpy data, tabulated below and assuming ideal solutions. Enthalpies in Btu/lbmol at reboiler temperature:

	$h_L$	$h_V$
benzene	4,900	18,130
toluene	8,080	21,830

Vapor–liquid equilibrium data are given in Exercise 7.13.

**7.29** A continuous distillation unit, consisting of a perforated-tray column together with a partial reboiler and a total condenser, is to be designed to operate at atmospheric pressure to separate ethanol and water. The feed, which is introduced into the column as liquid at its bubble point, contains 20 mol% alcohol. The distillate is to contain 85 mol% alcohol, and the alcohol recovery is to be 97%.

(a) What is the molar concentration of the bottoms?

(b) What is the minimum value of:

(1) The reflux ratio  $L/V$ ?

(2) The reflux ratio  $L/D$ ?

(3) The boilup ratio  $\bar{V}/B$  from the reboiler?

(c) What is the minimum number of theoretical stages and the corresponding number of actual plates if the overall plate efficiency is 55%?

(d) If the reflux ratio  $L/V$  used is 0.80, how many actual plates will be required?

Vapor–liquid equilibrium for ethanol–water at 1 atm in terms of mole fractions of ethanol are [*Ind. Eng. Chem.*, **24**, 881 (1932)]:

$x$	$y$	$T, ^\circ\text{C}$	$x$	$y$	$T, ^\circ\text{C}$
0.0190	0.1700	95.50	0.3273	0.5826	81.50
0.0721	0.3891	89.00	0.3965	0.6122	80.70
0.0966	0.4375	86.70	0.5079	0.6564	79.80
0.1238	0.4704	85.30	0.5198	0.6599	79.70
0.1661	0.5089	84.10	0.5732	0.6841	79.30
0.2337	0.5445	82.70	0.6763	0.7385	78.74
0.2608	0.5580	82.30	0.7472	0.7815	78.41
			0.8943	0.8943	78.15

**7.30** A solvent A is to be recovered by distillation from its water solution. It is necessary to produce an overhead product containing 95 mol% A and to recover 95% of the A in the feed. The feed is available at the plant site in two streams, one containing 40 mol% A and the other 60 mol% A. Each stream will provide 50 kmol/h of component A, and each will be fed into the column as saturated liquid. Since the less volatile component is water, it has been proposed to supply the necessary heat in the form of open steam. For the preliminary design, it has been suggested that the operating reflux ratio,  $L/D$ , be 1.33 times the minimum value. A total condenser will be employed. For this system, it is estimated that the overall plate

efficiency will be 70%. How many plates will be required, and what will be the bottoms composition? The relative volatility may be assumed to be constant at 3.0. Determine analytically the points necessary to locate the operating lines. Each feed should enter the column at its optimal location.

**7.31** A saturated-liquid feed stream containing 40 mol% *n*-hexane (H) and 60 mol% *n*-octane is fed to a plate column. A reflux ratio  $L/D$  equal to 0.5 is maintained at the top of the column. An overhead product of 0.95 mole fraction H is required, and the column bottoms is to be 0.05 mole fraction H. A cooling coil submerged in the liquid of the second plate from the top removes sufficient heat to condense 50 mol% of the vapor rising from the third plate down from the top.

(a) Derive the equations needed to locate the operating line.

(b) Locate the operating lines and determine the required number of theoretical plates if the optimal feed plate location is used.

**7.32** One hundred kilogram-moles per hour of a saturated liquid mixture of 12 mol% ethyl alcohol in water is distilled continuously by direct steam at 1 atm introduced directly to the bottom plate. The distillate required is 85 mol% alcohol, representing 90% recovery of the alcohol in the feed. The reflux is saturated liquid with  $L/D = 3$ . Feed is on the optimal stage. Vapor-liquid equilibrium data are given in Exercise 7.29. Calculate:

(a) Steam requirement, kmol/h

(b) Number of theoretical stages

(c) The feed stage (optimal)

(d) Minimum reflux ratio,  $(L/D)_{\min}$

**7.33** A water-isopropanol mixture at its bubble point containing 10 mol% isopropanol is to be continuously rectified at atmospheric pressure to produce a distillate containing 67.5 mol% isopropanol. Ninety-eight percent of the isopropanol in the feed must be recovered. If a reflux ratio  $L/D$  of 1.5 times the minimum is used, how many theoretical stages will be required: (a) If a partial reboiler is used? (b) If no reboiler is used and saturated steam at 101 kPa is introduced below the bottom plate? (c) How many stages are required at total reflux?

Vapor-liquid equilibrium data in mole fraction of isopropanol at 101 kPa are

$T, ^\circ\text{C}$	93.00	84.02	82.12	81.25	80.62	80.16	80.28	81.51
$y$	0.2195	0.4620	0.5242	0.5686	0.5926	0.6821	0.7421	0.9160
$x$	0.0118	0.0841	0.1978	0.3496	0.4525	0.6794	0.7693	0.9442

Notes: Composition of the azeotrope is  $x = y = 0.6854$ . Boiling point of azeotrope = 80.22°C

**7.34** An aqueous solution containing 10 mol% isopropanol is fed at its bubble point to the top of a continuous stripping column, operated at atmospheric pressure, to produce a vapor containing 40 mol% isopropanol. Two procedures are under consideration, both involving the same heat expenditure with  $V/F$  (moles of vapor generated/mole of feed) = 0.246 in each case. Scheme 1 uses a partial reboiler at the bottom of a plate-type stripping column, generating vapor by the use of steam condensing inside a closed coil. In Scheme 2, the reboiler is omitted and live steam is injected directly below the bottom plate. Determine the number of stages required in each case.

Equilibrium data for the system isopropanol-water are given in Exercise 7.33. The usual simplifying assumptions may be made.

**7.35** Determine the optimal-stage location for each feed and the number of theoretical stages required for the distillation separation

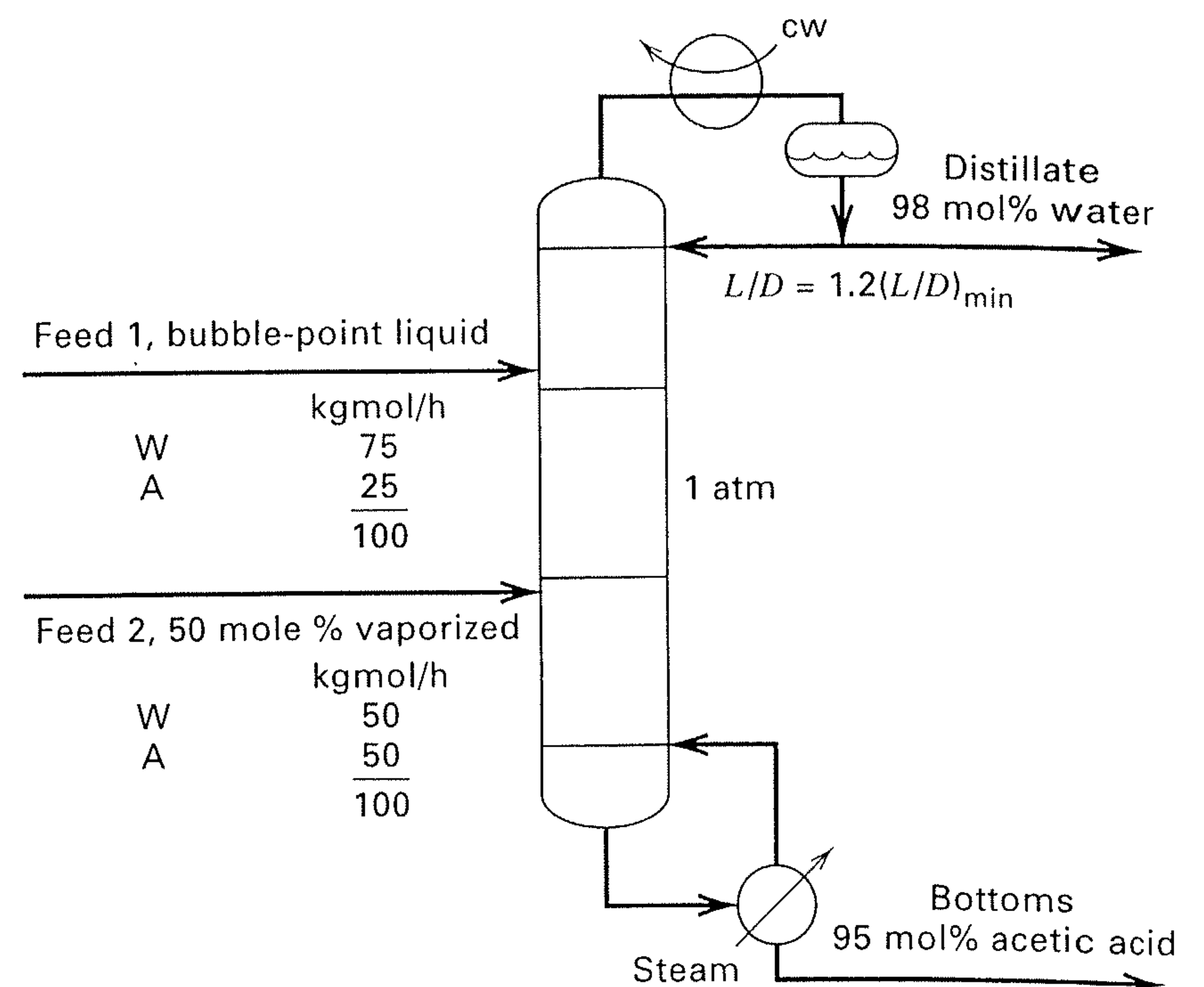


Figure 7.42 Data for Exercise 7.35.

shown in Figure 7.42 using the following equilibrium data in mole fractions.

#### WATER (W)/ACETIC ACID (A), 1 ATM

$x_W$	0.0055	0.053	0.125	0.206	0.297	0.510	0.649	0.803	0.9594
$y_W$	0.0112	0.133	0.240	0.338	0.437	0.630	0.751	0.866	0.9725

**7.36** Determine the number of theoretical stages required and the optimal-stage locations for the feed and liquid side stream for the distillation process shown in Figure 7.43 assuming that methanol (M) and ethanol (E) form an ideal solution.

**7.37** A mixture of *n*-heptane (H) and toluene (T) is separated by extractive distillation with phenol (P). Distillation is then used to recover the phenol for recycle as shown in Figure 7.44a, where the small amount of *n*-heptane in the feed is ignored. For the conditions shown in Figure 7.44a, determine the number of theoretical stages

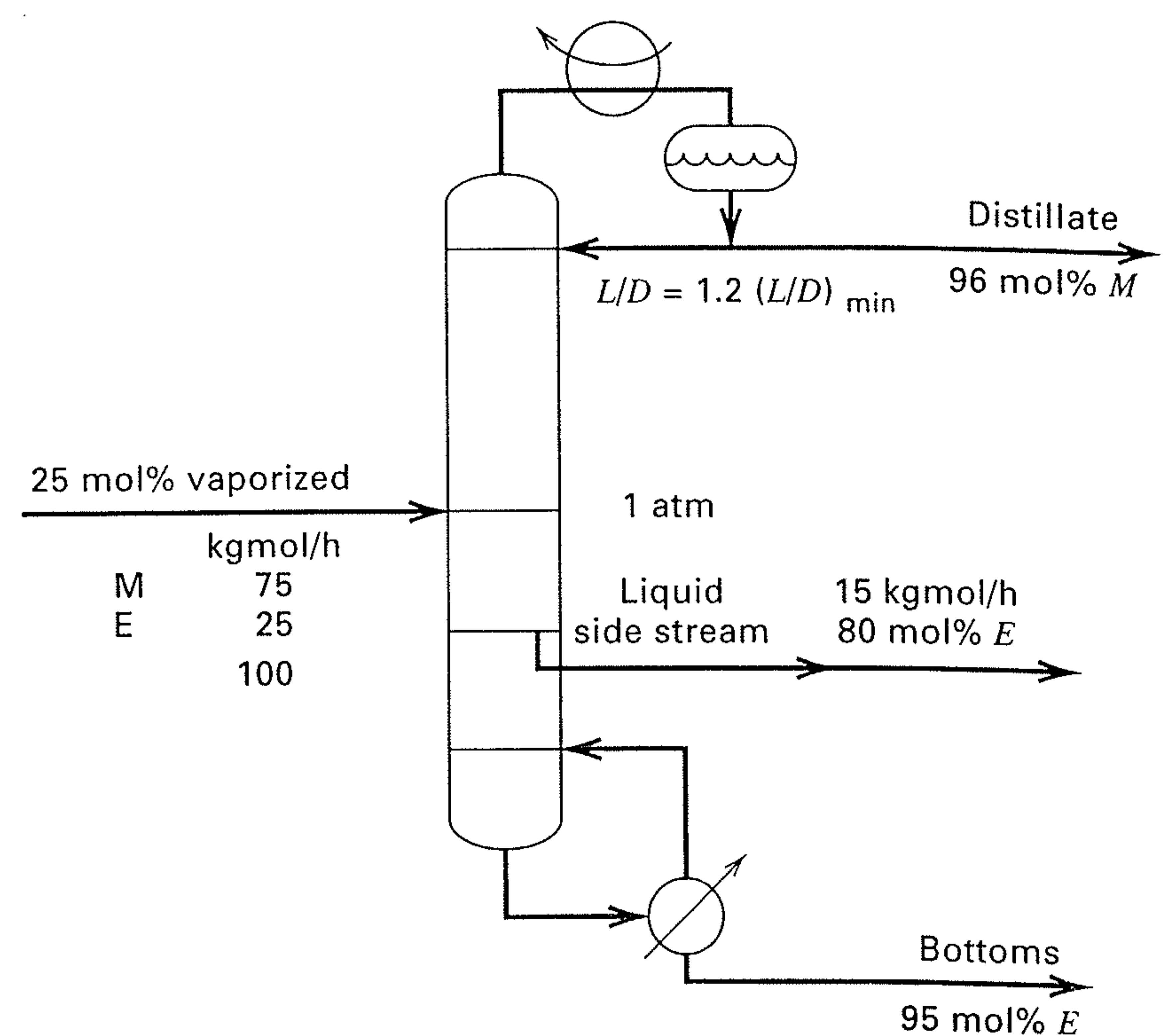


Figure 7.43 Data for Exercise 7.36.

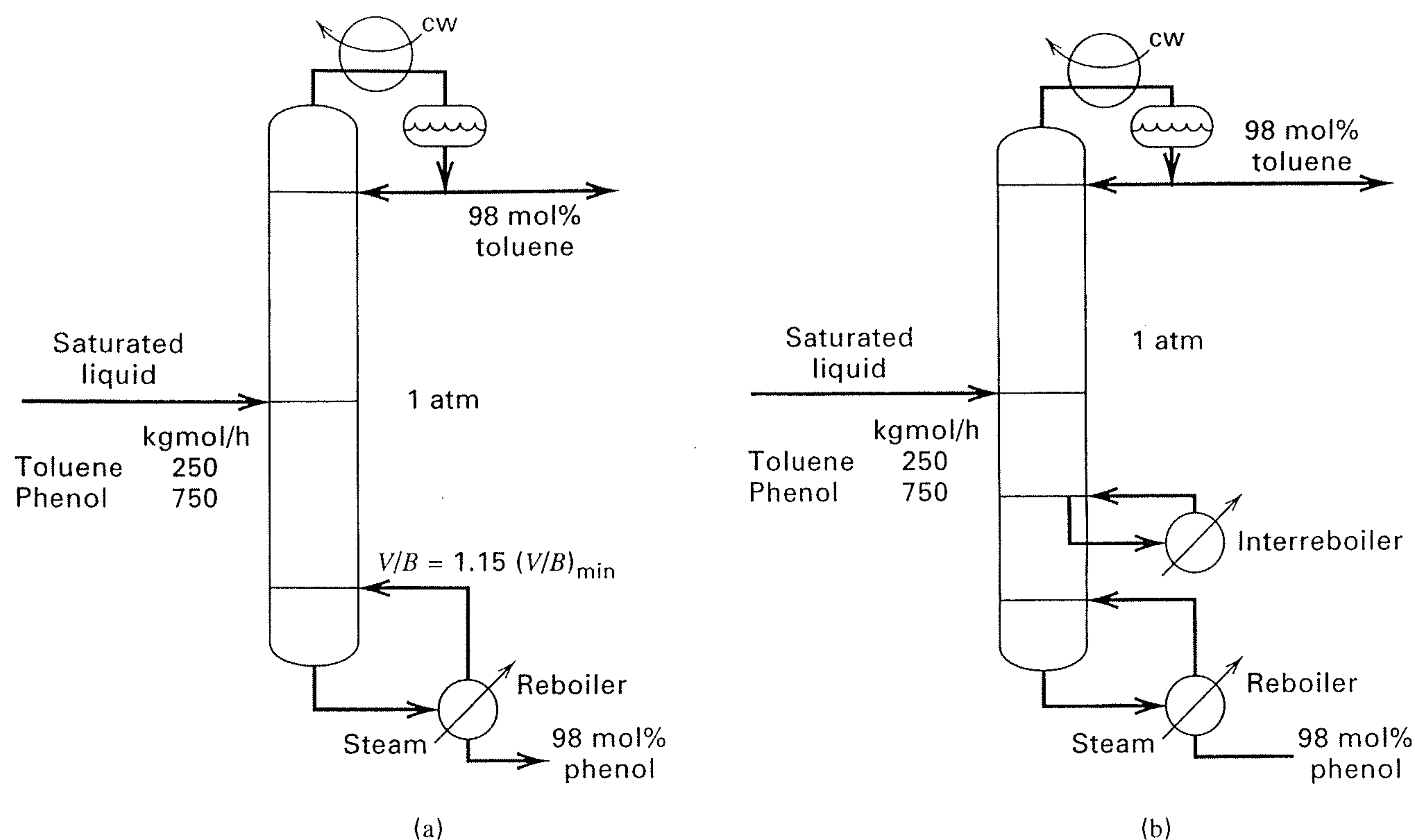


Figure 7.44 Data for Exercise 7.37.

required. Note that heat will have to be supplied to the reboiler at a high temperature because of the high boiling point of phenol.

Therefore, consider the alternative scheme in Figure 7.44b, where an interreboiler, located midway between the bottom plate and the feed stage, is used to provide 50% of the boilup used in Figure 7.44a. The remainder of the boilup is provided by the reboiler. Determine the number of theoretical stages required for the case with the interreboiler and the temperature of the interreboiler stage. Unsmoothed vapor–liquid equilibrium data at 1 atm are [*Trans. AIChE*, **41**, 555 (1945)]:

$x_T$	$y_T$	$T, ^\circ\text{C}$	$x_T$	$y_T$	$T, ^\circ\text{C}$
0.0435	0.3410	172.70	0.6512	0.9260	120.00
0.0872	0.5120	159.40	0.7400	0.9463	119.70
0.1186	0.6210	153.80	0.7730	0.9536	119.40
0.1248	0.6250	149.40	0.8012	0.9545	115.60
0.2190	0.7850	142.20	0.8840	0.9750	112.70
0.2750	0.8070	133.80	0.9108	0.9796	112.20
0.4080	0.8725	128.30	0.9394	0.9861	113.30
0.4800	0.8901	126.70	0.9770	0.9948	111.10
0.5898	0.9159	122.20	0.9910	0.9980	111.10
0.6348	0.9280	120.20	0.9939	0.9986	110.50
			0.9973	0.9993	110.50

**7.38** A distillation column for the separation of *n*-butane from *n*-pentane was recently put into operation in a petroleum refinery. Apparently, an error was made in the design because the column fails to make the desired separation as shown in the following table [*Chem. Eng. Prog.*, **61** (8), 79 (1965)]:

	Design Specification	Actual Operation
Mol% $nC_5$ in distillate	0.26	13.49
Mol% $nC_4$ in bottoms	0.16	4.28

In order to correct the situation, it is proposed to add an intercondenser in the rectifying section to generate more reflux and an interreboiler in the stripping section to produce additional boilup. Show by use of a McCabe–Thiele diagram how such a proposed change can improve the operation.

**7.39** In the production of chlorobenzenes by chlorination of benzene, two close-boiling isomers, *para*-dichlorobenzene (P) and *ortho*-dichlorobenzene (O), are separated by distillation. The feed to the column consists of 62 mol% of the *para* isomer and 38 mol% of the *ortho* isomer. Assume that the pressures at the bottom and top of the column are 20 psia (137.9 kPa) and 15 psia (103.4 kPa), respectively. The distillate is a liquid containing 98 mol% *para* isomer. The bottoms product is to contain 96 mol% *ortho* isomer. At column pressure, the feed is slightly vaporized with  $q = 0.9$ . Calculate the number of theoretical stages required for a reflux ratio equal to 1.15 times the minimum-reflux ratio. Base your calculations on a constant relative volatility obtained as the arithmetic average between the column top and column bottom using appropriate vapor pressure data and the assumption of Raoult's and Dalton's laws. The McCabe–Thiele construction should be supplemented at the two ends by use of the Kremser equations as illustrated in Example 7.4.

**7.40** Relatively pure oxygen and nitrogen can be obtained by the distillation of air using the Linde double column, which, as shown in Figure 7.45, consists of a lower column operating at elevated pressure surmounted by an atmospheric-pressure column. The boiler of the upper column is at the same time the reflux condenser for both columns. Gaseous air plus enough liquid to take care of heat leak into the column (more liquid, of course, if liquid-oxygen product is withdrawn) enters the exchanger at the base of the lower column and condenses, giving up heat to the boiling liquid and thus supplying the vapor flow for this column. The liquid air enters an intermediate point in this column, as shown in Figure 7.45. The vapors rising in this column are partially condensed to form the reflux, and the uncondensed vapor passes to an outer row of tubes and is totally condensed, the liquid nitrogen collecting in an annulus, as shown. By operating this column at 4 to 5 atm, the liquid oxygen boiling at 1 atm is cold enough to condense pure nitrogen. The liquid that collects in the bottom of the lower column contains about 45 mol%  $O_2$  and forms the feed for the upper column. Such a double column can produce very pure oxygen with high oxygen recovery, and relatively pure nitrogen. On a single McCabe–Thiele diagram—using equilibrium lines, operating lines,  $q$ -lines,  $45^\circ$  line, stepped-off stages, and other illustrative aids—show qualitatively how the stage requirements of the double column can be computed.



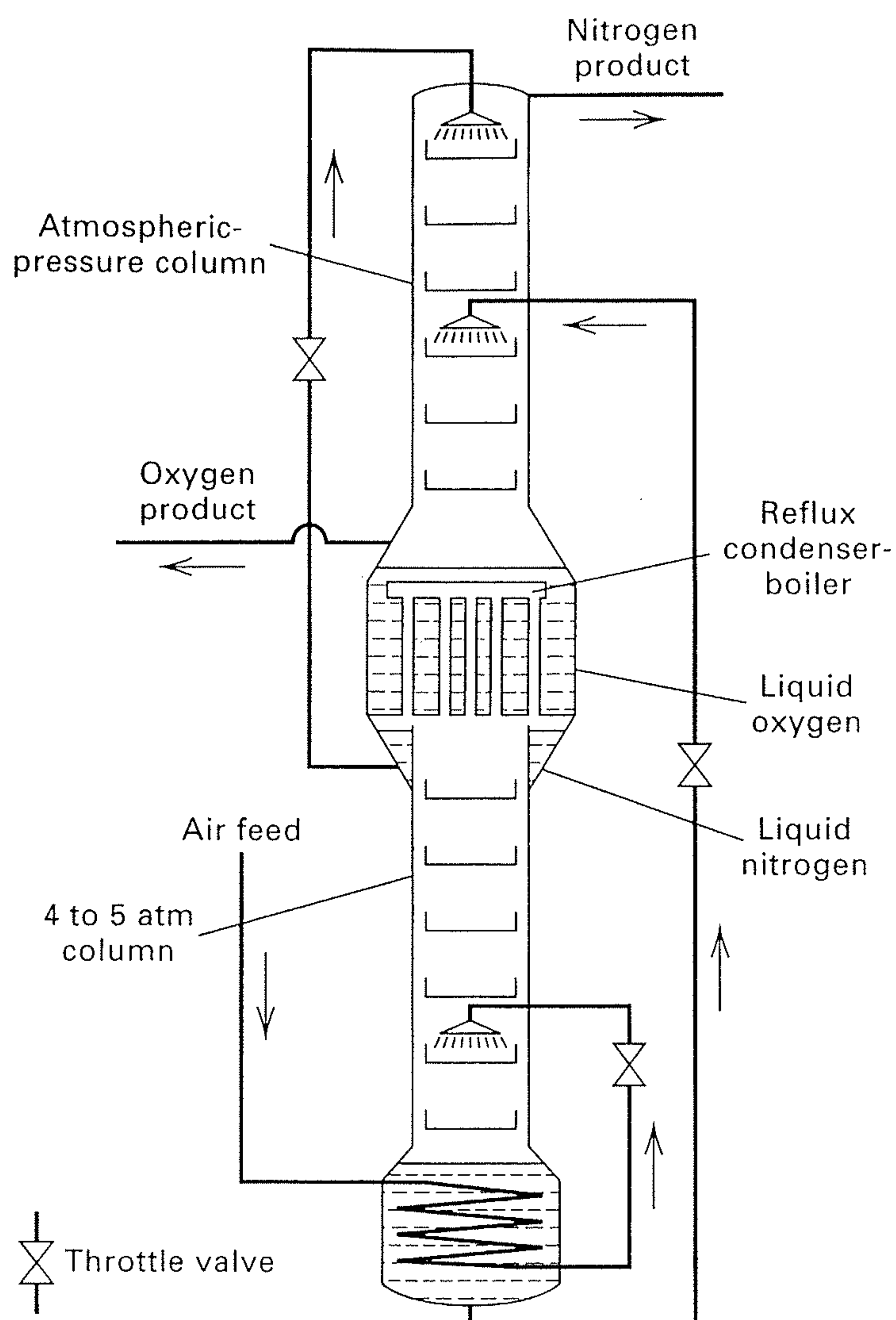


Figure 7.45 Data for Exercise 7.40.

## Section 7.3

7.41 The following performance data have been obtained for a distillation tower separating a 50/50 by weight percent mixture of methanol and water:

Feed rate = 45,438 lb/h, Feed condition = bubble-point liquid at feed-tray pressure, Wt% methanol in distillate = 95.04, and Wt% methanol in bottoms = 1.00

Reflux ratio = 0.947; Reflux condition = saturated liquid

Boilup ratio = 1.138; Pressure in reflux drum = 14.7 psia

Type condenser = total; Type reboiler = partial

Condenser pressure drop = 0.0 psi; Tower pressure drop = 0.8 psi

Trays above feed tray = 5; Trays below feed tray = 6

Total trays = 12; Tray diameter = 6 ft

Type tray = single-pass sieve tray; Flow path length = 50.5 in.

Weir length = 42.5 in.; Hole area = 10%; Hole size = 3/16 in.

Weir height = 2 in.; Tray spacing = 24 in.

Viscosity of feed = 0.34 cP

Surface tension of distillate = 20 dyne/cm; Surface tension of bottoms = 58 dyne/cm

Temperature of top tray = 154°F; Temperature of bottom tray = 207°F

Vapor-liquid equilibrium data at column pressure in mole fraction of methanol are

y	0.0412	0.156	0.379	0.578	0.675	0.729	0.792	0.915
x	0.00565	0.0246	0.0854	0.205	0.315	0.398	0.518	0.793

Based on the above data:

(a) Determine the overall tray efficiency from the data, assuming that the reboiler is the equivalent of a theoretical stage.

(b) Estimate the overall tray efficiency from the Drickamer-Bradford correlation.

(c) Estimate the overall tray efficiency from the O'Connell correlation, accounting for length of flow path.

(d) Estimate the Murphree vapor tray efficiency by the method of Chan and Fair.

7.42 For the conditions of Exercise 7.41, a laboratory Oldershaw column measures an average Murphree vapor-point efficiency of 65%. Estimate  $E_{MV}$  and  $E_o$ .

## Section 7.4

7.43 Conditions for the top tray of a distillation column are as shown in Figure 7.46. Determine the column diameter corresponding to 85% of flooding if a valve tray is used. Make whatever assumptions necessary.

7.44 A separation of propylene from propane is achieved by distillation as shown in Figure 7.47, where two columns in series are used because a single column would be too tall. The tray numbers refer to equilibrium stages. Determine the column diameters, tray efficiency using the O'Connell correlation, number of actual trays, and column heights if perforated trays are used.

7.45 Determine the height and diameter of a vertical flash drum for the conditions shown in Figure 7.48.

7.46 Determine the length and diameter of a horizontal reflux drum for the conditions shown in Figure 7.49.

7.47 Results of design calculations for a methanol-water distillation operation are given in Figure 7.50.

(a) Calculate the column diameter at the top tray and at the bottom tray for sieve trays. Should the column be swaged?

(b) Calculate the length and diameter of the horizontal reflux drum.

7.48 For the conditions given in Exercise 7.41, estimate for the top tray and the bottom tray: (a) Percent of flooding, (b) Tray pressure drop in psi, (c) Whether weeping will occur, (d) Entrainment rate, and (e) Froth height in the downcomer.

7.49 If the feed rate to the tower of Exercise 7.41 is increased by 30% with all other conditions except for tower pressure drop remaining the same, estimate for the top and bottom trays: (a) Per-

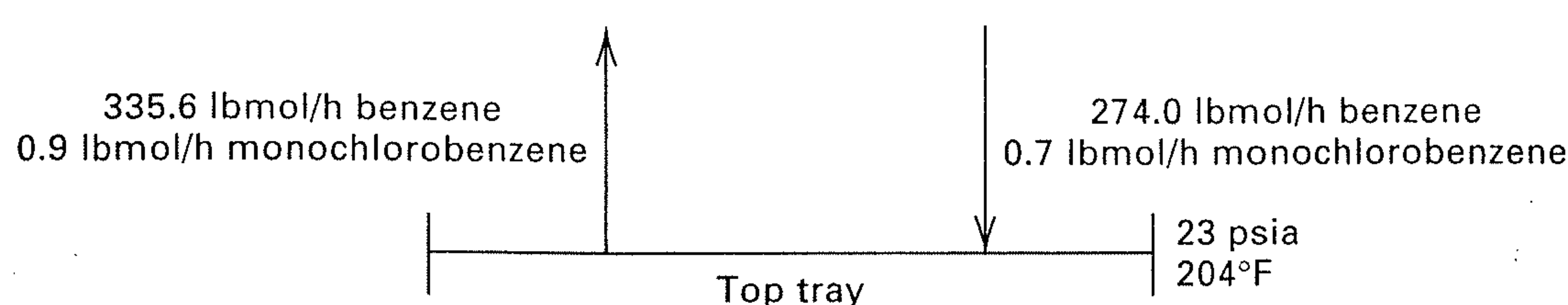


Figure 7.46 Data for Exercise 7.43.

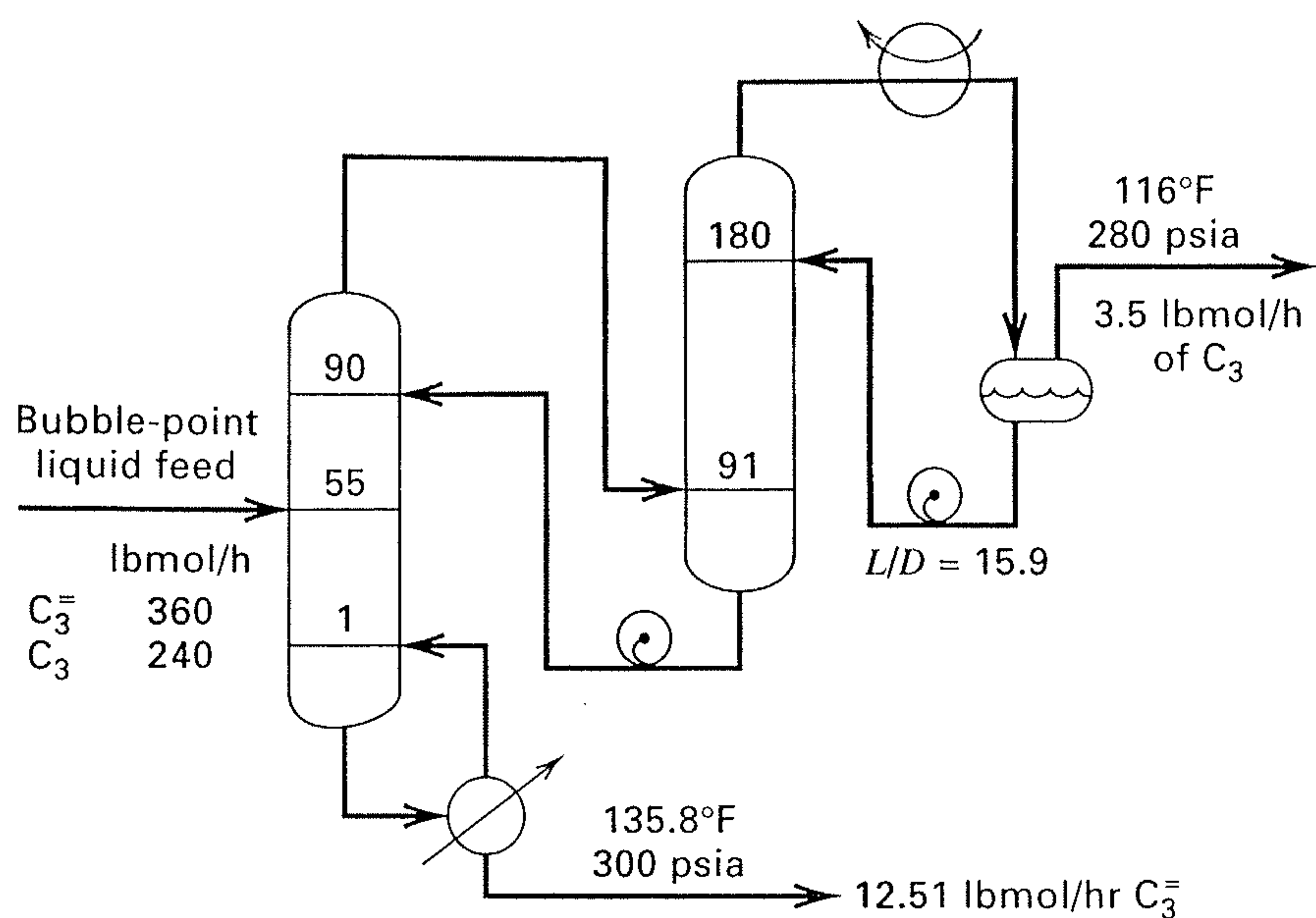


Figure 7.47 Data for Exercise 7.44.

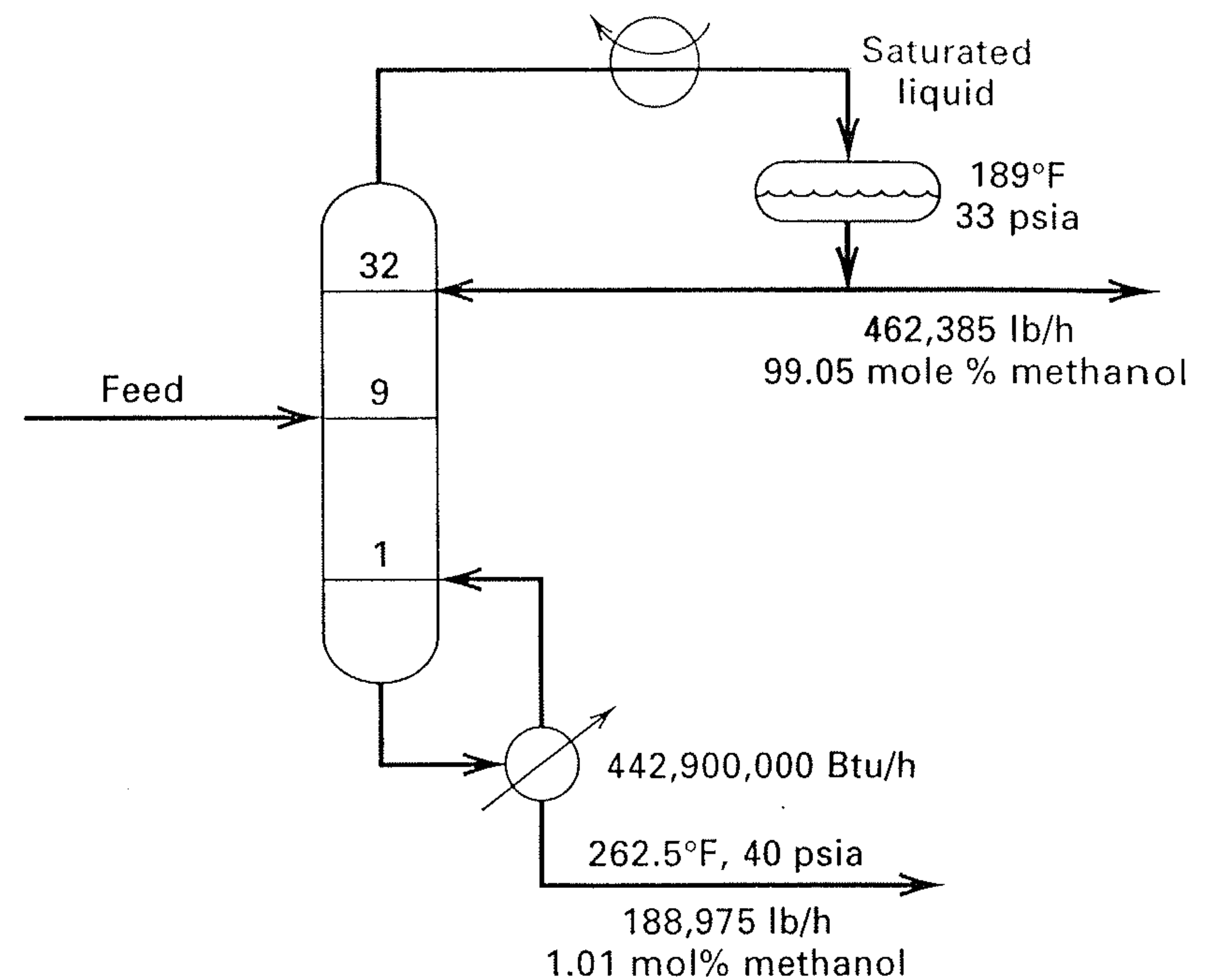


Figure 7.50 Data for Exercise 7.47.

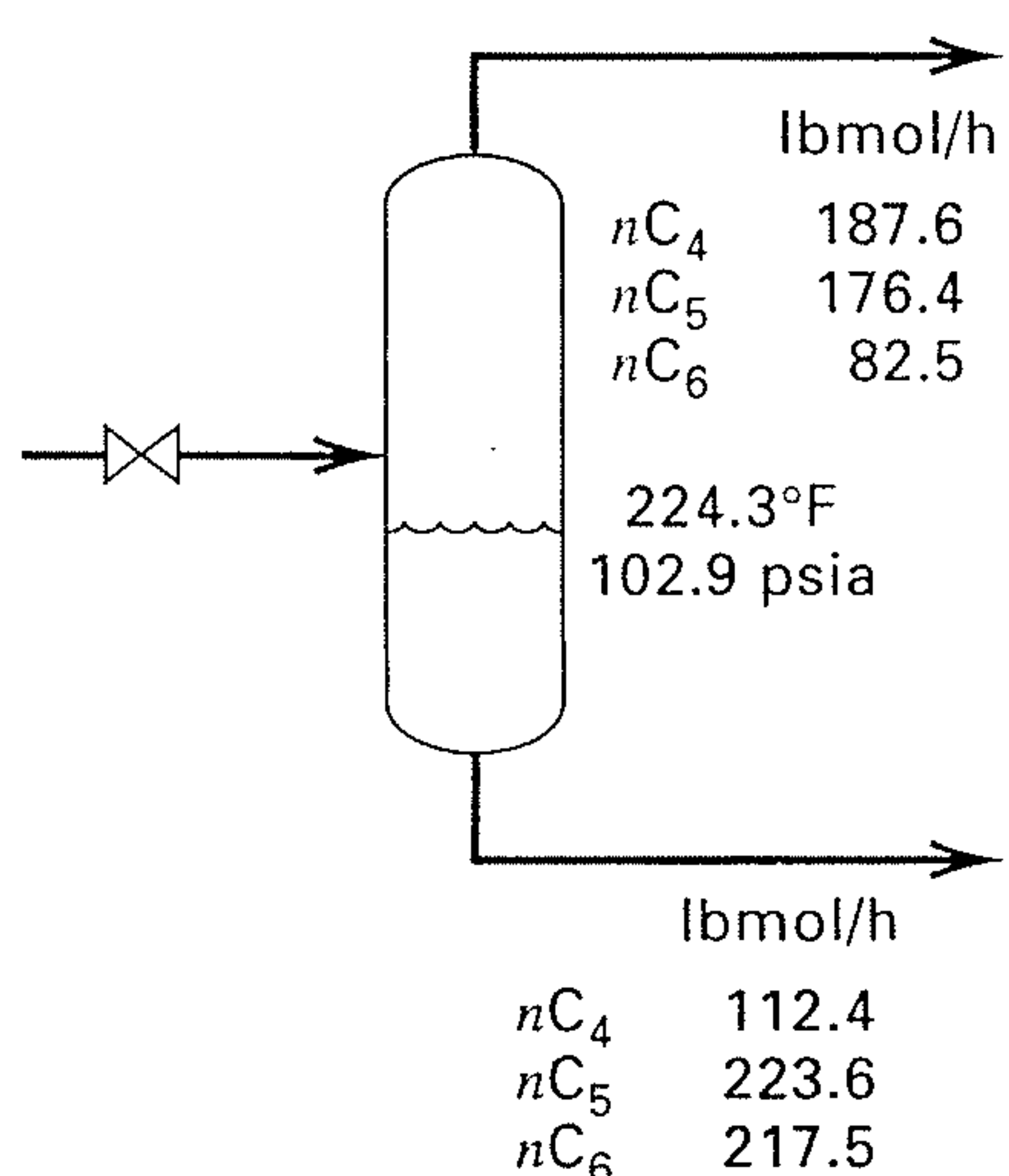


Figure 7.48 Data for Exercise 7.45.

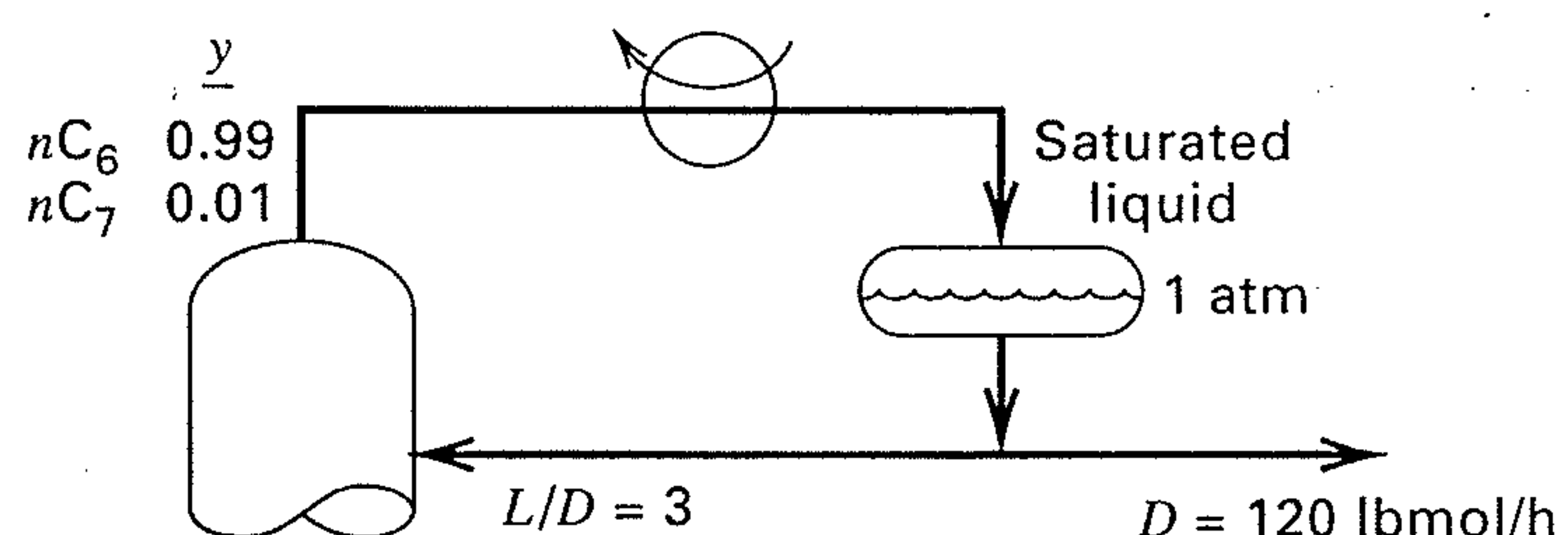


Figure 7.49 Data for Exercise 7.46.

cent of flooding, (b) Tray pressure drop in psi, (c) Entrainment rate, (d) Froth height in the downcomer. Will the new operation be acceptable? If not, should you consider a retrofit with packing? If so, should both sections of the column be packed or could just one section be packed to achieve an acceptable operation?

### Section 7.5

**7.50** A mixture of benzene and dichloroethane is used to test the efficiency of a packed column that contains 10 ft of packing and operates adiabatically at atmospheric pressure. The liquid is charged to the reboiler, and the column is operated at total reflux until equilibrium is established. At equilibrium, liquid samples from the distillate and reboiler, as analyzed by refractive index, give the following compositions for benzene:  $x_D = 0.653$ ,  $x_B = 0.298$ .

Calculate the value of HETP in inches for this packing. What are the limitations on using this calculated value for design?

Data for  $x$ - $y$  at 1 atm (in benzene mole fractions) are

$x$	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
$y$	0.11	0.22	0.325	0.426	0.526	0.625	0.720	0.815	0.91

**7.51** Consider a distillation column for separating ethanol from water at 1 atm. The following specifications are set:

Feed: 10 mol% ethanol (bubble-point liquid)

Bottoms: 1 mol% ethanol

Distillate: 80 mol% ethanol (saturated liquid)

Reflux ratio: 1.5 times the minimum

Constant molar overflow may be assumed and vapor-liquid equilibrium data are given in Exercise 7.29.

- How many theoretical plates are required above and below the feed if a plate column is used?
- How many transfer units are required above and below the feed if a packed column is used?
- Assuming that the plate efficiency is approximately 80% and the plate spacing is 18 in., how high is the plate column?
- Using an  $H_{OG}$  value of 1.2 ft., how high is the packed column?
- Assuming that you had HTU data available only on the benzene-toluene system, how would you go about applying the data to obtain the HTU for the ethanol-water system?

**7.52** Plant capacity for the methanol-water distillation of Exercise 7.41 is to be doubled. Rather than installing a second trayed tower identical to the one in operation, a packed column is to be considered for the new installation. This column will have a feed location identical to the present trayed tower and will be expected to achieve the same product purities with the same top pressure and reflux ratio. Two packings are being considered:

- 50-mm plastic NOR PAC rings (a random packing)
- Montz metal B1-300 (a structured packing)

For each of these two packings, design a packed column to operate at 70% of flooding by calculating for each section: (a) Liquid holdup, (b) Column diameter, (c)  $H_{OG}$ , (d) Packed height, (e) Pressure drop. What are the advantages, if any, of each of the packed-column designs over a second trayed tower? Which packing, if either, is preferable?

**Table 7.8** Methanol–Water Vapor–Liquid Equilibrium and Enthalpy Data for 1 atm (MeOH = Methyl Alcohol)

Mol% MeOH <i>y</i> or <i>x</i>	Enthalpy above 0°C, Btu/lbmol Solution				Vapor Liquid Equilibrium Data		
	Saturated Vapor		Saturated Liquid		Mol% MeOH in		Temperature, °C
	<i>T</i> , °C	<i>h<sub>V</sub></i>	<i>T</i> , °C	<i>h<sub>L</sub></i>	Liquid	Vapor	
0	100	20,720	100	3,240	0	0	100
5	98.9	20,520	92.8	3,070	2.0	13.4	96.4
10	97.7	20,340	87.7	2,950	4.0	23.0	93.5
15	96.2	20,160	84.4	2,850	6.0	30.4	91.2
20	94.8	20,000	81.7	2,760	8.0	36.5	89.3
30	91.6	19,640	78.0	2,620	10.0	41.8	87.7
40	88.2	19,310	75.3	2,540	15.0	51.7	84.4
50	84.9	18,970	73.1	2,470	20.0	57.9	81.7
60	80.9	18,650	71.2	2,410	30.0	66.5	78.0
70	76.6	18,310	69.3	2,370	40.0	72.9	75.3
80	72.2	17,980	67.6	2,330	50.0	77.9	73.1
90	68.1	17,680	66.0	2,290	60.0	82.5	71.2
100	64.5	17,390	64.5	2,250	70.0	87.0	69.3
					80.0	91.5	67.6
					90.0	95.8	66.0
					95.0	97.9	65.0
					100.0	100.0	64.5

Source: J.G. Dunlop, "Vapor–Liquid Equilibrium Data," M.S. thesis, Brooklyn Polytechnic Institute, Brooklyn, NY (1948).

**7.53** For the specifications of Example 7.1, design a packed column using 50-mm metal Hiflow rings and operating at 70% of flooding by calculating for each section: (a) Liquid holdup, (b) Column diameter, (c)  $H_{OG}$ , (d) Packed height, and (e) Pressure drop.

What are the advantages and disadvantages of a packed column as compared to a trayed tower for this service?

### Section 7.6

**7.54** An enthalpy–concentration diagram is given in Figure 7.37 for a mixture of *n*-hexane (H), and *n*-octane (O) at 101 kPa. Using this diagram, determine the following:

- The mole-fraction composition of the vapor when a liquid containing 30 mol% H is heated from point A to the bubble-point temperature at point B.
- The energy required to vaporize 60 mol% of a mixture initially at 100°F and containing 20 mol% H (point G).
- The compositions of the equilibrium vapor and liquid resulting from part (b).

**7.55** Using the enthalpy–concentration diagram of Figure 7.37, determine the following for a mixture of *n*-hexane (H) and *n*-octane (O) at 1 atm:

- The temperature and compositions of equilibrium liquid and vapor resulting from adiabatic mixing of 950 lb/h of a mixture of

30 mol% H in O at 180°F with 1,125 lb/h of a mixture of 80 mol% H in O at 240°F.

(b) The energy required to partially condense, by cooling, a mixture of 60 mol% H in O from an initial temperature of 260°F to 200°F. What are the compositions and amounts of the resulting vapor and liquid phases per pound-mole of original mixture?

(c) If the equilibrium vapor from part (b) is further cooled to 180°F, determine the compositions and relative amounts of the resulting vapor and liquid.

**7.56** One hundred pound-moles per hour of a mixture of 60 mol% methanol in water at 30°C and 1 atm is to be separated by distillation at the same pressure into a liquid distillate containing 98 mol% methanol and a bottoms liquid product containing 96 mol% water. Enthalpy and equilibrium data for the mixture at 1 atm are given in Table 7.8. The enthalpy of the feed mixture is 765 Btu/lbmol.

- Using the given data, plot an enthalpy–concentration diagram.
- Devise a procedure to determine, from the diagram of part (a), the minimum number of equilibrium stages for the condition of total reflux and the required separation.
- From the procedure developed in part (b), determine  $N_{\min}$ . Why is the value independent of the feed condition?
- What are the temperatures of the distillate and the bottoms?