

Separation Process Engineering

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Introduction to Column Distillation

Distillation is by far the most common separation technique in the chemical process industry accounting for 90% to 95% of the separations. The approximately 40,000 distillation columns in use account for approximately 40% of the energy use by the United States' chemical process industry—equivalent to a staggering 1.2 million barrels of crude oil a day (Humphrey and Keller, 1997).

This chapter introduces how continuous distillation columns work and serves as the lead to a series of nine chapters on distillation. The basic calculation procedures for binary distillation are developed in Chapter 4. Multicomponent distillation is introduced in Chapter 5, detailed computer calculation procedures for these systems are developed in Chapter 6, and simplified shortcut methods are covered in Chapter 7. More complex distillation operations such as extractive and azeotropic distillation are the subject of Chapter 8. Chapter 9 switches to batch distillation, which is commonly used for smaller systems. Detailed design procedures for both staged and packed columns are discussed in Chapter 10. Finally, Chapter 11 looks at the economics of distillation and methods to save energy (and money) in distillation systems.

3.1 DEVELOPING A DISTILLATION CASCADE

In Chapter 2, we learned how to do the calculations for flash distillation. Flash distillation is a very simple unit operation, but in most cases it produces a limited amount of separation. In Problems 2.D2, 2.D14, and 2.F3 we saw that more separation could be obtained by adding on (or *cascading*) more flash separators. The cascading procedure can be extended into a process that produces one pure vapor and one pure liquid product. First, we could send the vapor streams to additional flash chambers at increasing pressures and the liquid streams to flash chambers with decreasing pressures, as shown in Figure 3-1. Stream V_1 will have a high concentration of the more volatile component, and stream L_5 will have a low concentration of the more volatile component. Each flash chamber in Figure 3-1 can be analyzed by the methods developed previously.

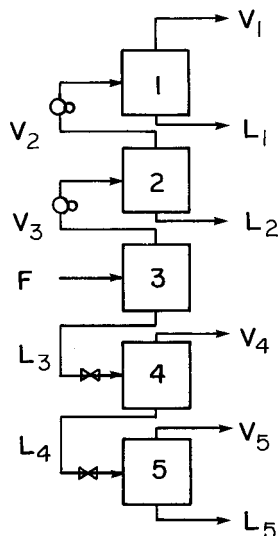


FIGURE 3-1. Cascade of flash chambers, $p_1 > p_2 > p_3 > p_4 > p_5$

One difficulty with the cascade shown in Figure 3-1 is that the intermediate product streams, L_1 , L_2 , V_4 , and V_5 , are of intermediate concentration and need further separation. Of course, each of these streams could be fed to another flash cascade, but then the intermediate products from those cascades would have to be sent to additional cascades, and so forth. A much cleverer solution is to use the intermediate product streams as additional feeds within the same cascade.

Consider stream L_2 , which was generated by flashing part of the feed stream and then condensing part of the resulting vapor. Since the material in L_2 has been vaporized once and condensed once, it probably has a concentration close to that of the original feed stream. (To check this, you can do the appropriate flash calculation on a McCabe-Thiele diagram.) Thus, it is appropriate to use L_2 as an additional feed stream to stage 3. However, since $p_2 > p_3$, its pressure must first be decreased.

Stream L_1 is the liquid obtained by partially condensing V_2 , the vapor obtained from flashing vapor stream V_3 . After one vaporization and then a condensation, stream L_1 will have a concentration close to that of stream V_3 . Thus it is appropriate to use stream L_1 as an additional feed to stage 2 after pressure reduction.

A similar argument can be applied to the intermediate vapor products below the feed, V_4 and V_5 . V_4 was obtained by partially condensing the feed stream and then partially vaporizing the resulting liquid. Since its concentration is approximately the same as the feed, stream V_4 can be used as an additional feed to stage 3 after compression to a higher pressure. By the same reasoning, stream V_5 can be fed to stage 4.

Figure 3-2 shows the resulting *countercurrent cascade*, so called because vapor and liquid streams go in opposite directions. The advantages of this cascade over the one shown in Figure 3-1 are that there are no intermediate products, and the two end products can both be pure *and* obtained in high yield. Thus V_1 can be almost 100% of the more volatile components and contain almost all of the more volatile component of the feed stream.

Although a significant advance, this variable pressure (or isothermal distillation) system is seldom used commercially. Operation at different pressures requires a larger number of

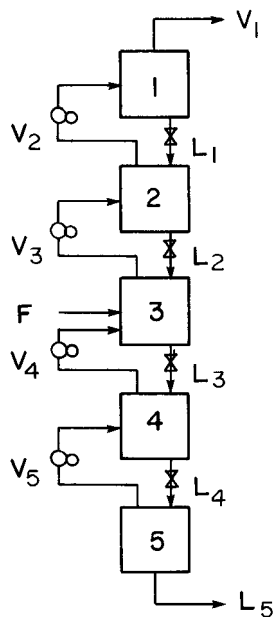


FIGURE 3-2. *Countercurrent cascade of flash chambers,*
 $p_1 > p_2 > p_3 > p_4 > p_5$

compressors, which are expensive. It is much cheaper to operate at constant pressure and force the temperature to vary. Thus, in stage 1 of Figure 3-2 a relatively low temperature would be employed, since the concentration of the more volatile component, which boils at a lower temperature, is high. For stage 5, where the less volatile component is concentrated, the temperature would be high. To achieve this temperature variation, we can use heat exchangers (reboilers) to partially vaporize the liquid streams. This is illustrated in Figure 3-3, where partial condensers and partial reboilers are used.

The cascade shown in Figure 3-3 has a decreasing vapor flow rate as we go from the feed stage to the top stage and a decreasing liquid flow rate as we go from the feed stage to the bottom stage. Operation and design will be easier if part of the top vapor stream V_1 is condensed and returned to stage 1 and if part of the bottom liquid stream L_5 is vaporized and returned to stage 5, as illustrated in Figure 3-4. This allows us to control the internal liquid and vapor flow rates at any desired level. Stream D is the distillate product, while B is the bottom product. Stream L_0 is called the *reflux* while V_6 is the *boilup*.

The use of reflux and boilup allows for a further simplification. We can now apply all of the heat required for the distillation to the bottom reboiler, and we can do all of the required cooling in the top condenser. The required partial condensation of intermediate vapor streams and partial vaporization of liquid streams can be done with the same heat exchangers as shown in Figure 3-5. Here stream V_2 is partially condensed by stream L_1 while L_1 is simultaneously partially vaporized. Since L_1 has a higher concentration of more volatile component, it will boil at a lower temperature and heat transfer is in the appropriate direction. Since the heat of vaporization per mole is usually approximately constant, condensation of 1 mole of vapor will vaporize 1 mole of liquid. Thus liquid and vapor flow rates tend to remain constant. Heat exchangers can be used for all other pairs of *passing streams*: L_2 and V_3 , L_3 and V_4 , and L_4 and V_5 .

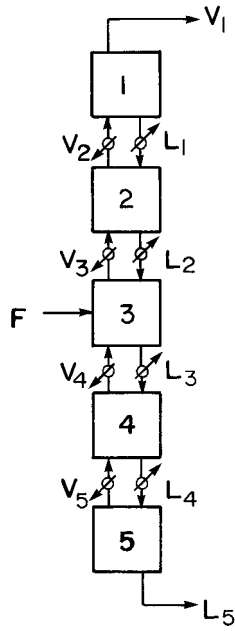


FIGURE 3-3. *Countercurrent cascade of flash chambers with intermediate reboilers and condensers, $p = \text{constant}$; $T_1 < T_2 < T_3 < T_4 < T_5$*

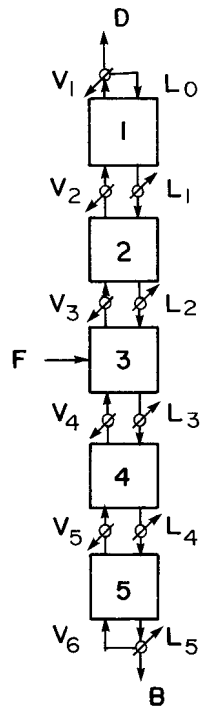


FIGURE 3-4. *Countercurrent cascade of flash chambers with reflux and boilup, $p = \text{constant}$; $T_1 < T_2 < T_3 < T_4 < T_5$*

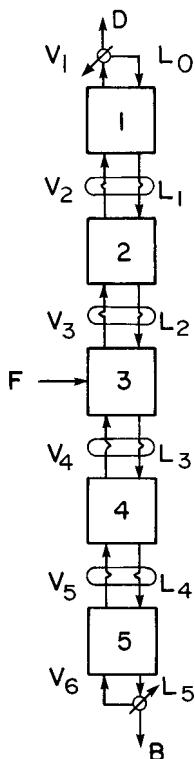


FIGURE 3-5. *Countercurrent cascade with intermediate heat exchangers*

Note that reflux and boilup are *not* the same as recycle. Recycle returns a stream to the feed to the process. Reflux (or boilup) first changes the phase of a stream and then returns the stream to the *same* stage the vapor (or liquid) was withdrawn from. This return at the same location helps increase the concentration of the component that is concentrated at that stage.

The cascade shown in Figure 3-5 can be further simplified by building the entire system in a column instead of as a series of individual stages. The intermediate heat exchange can be done very efficiently with the liquid and vapor in direct contact on each stage. The result is a much simpler and cheaper device. A schematic of such a distillation column is shown in Figure 3-6.

The cascade shown in Figure 3-6 is the usual form in which distillation is done. Because of the repeated vaporizations and condensations as we go up the column, the top product (distillate) can be highly concentrated in the more volatile component. The section of the column above the feed stage is known as the *enriching* or *rectifying* section. The bottom product (bottoms) is highly concentrated in the less volatile component, since the more volatile component has been stripped out by the rising vapors. This section is called the *stripping* section.

The distillation separation works because every time we vaporize material the more volatile component tends to concentrate in the vapor, and the less volatile component in the liquid. As the relative volatility of the system decreases, distillation becomes more difficult. If $\alpha = 1.0$, the liquid and vapor will have the same composition, and no separation will occur. Liquid and vapor also have the same composition when an azeotrope occurs. In this case one

can approach the azeotrope concentration at the top or bottom of the column but cannot get past it except with a heterogeneous azeotrope (see Chapter 8). The third limit to distillation is the presence of either chemical reactions between components or decomposition reactions. This problem can often be controlled by operating at lower temperatures and using vacuum or steam distillation (see Chapter 8).

While we are still thinking of flash distillation chambers, a simple but useful result can be developed. In a flash chamber a component will tend to exit in the vapor if $y_i V > x_i L$. Rearranging this, if $K_i V/L > 1$ a component tends to exit in the vapor. In a distillation column this means that components with $K_i V/L > 1$ tend to exit in the distillate, and components with $K_i V/L < 1$ tend to exit in the bottoms. This is only a qualitative guide, since the separation on each stage is far from perfect, and K_i , V , and L all vary in the column; however, it is useful to remember.

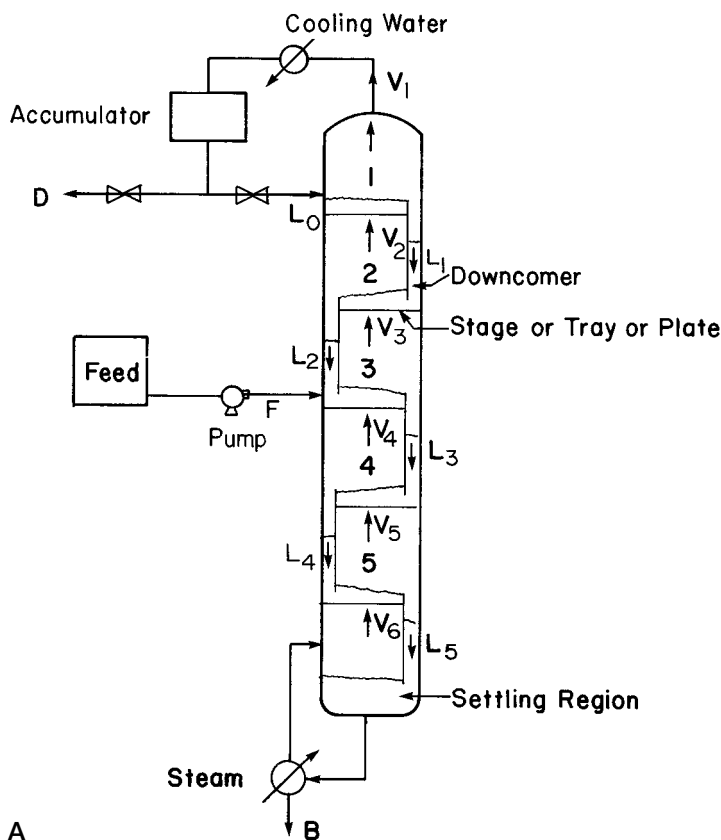
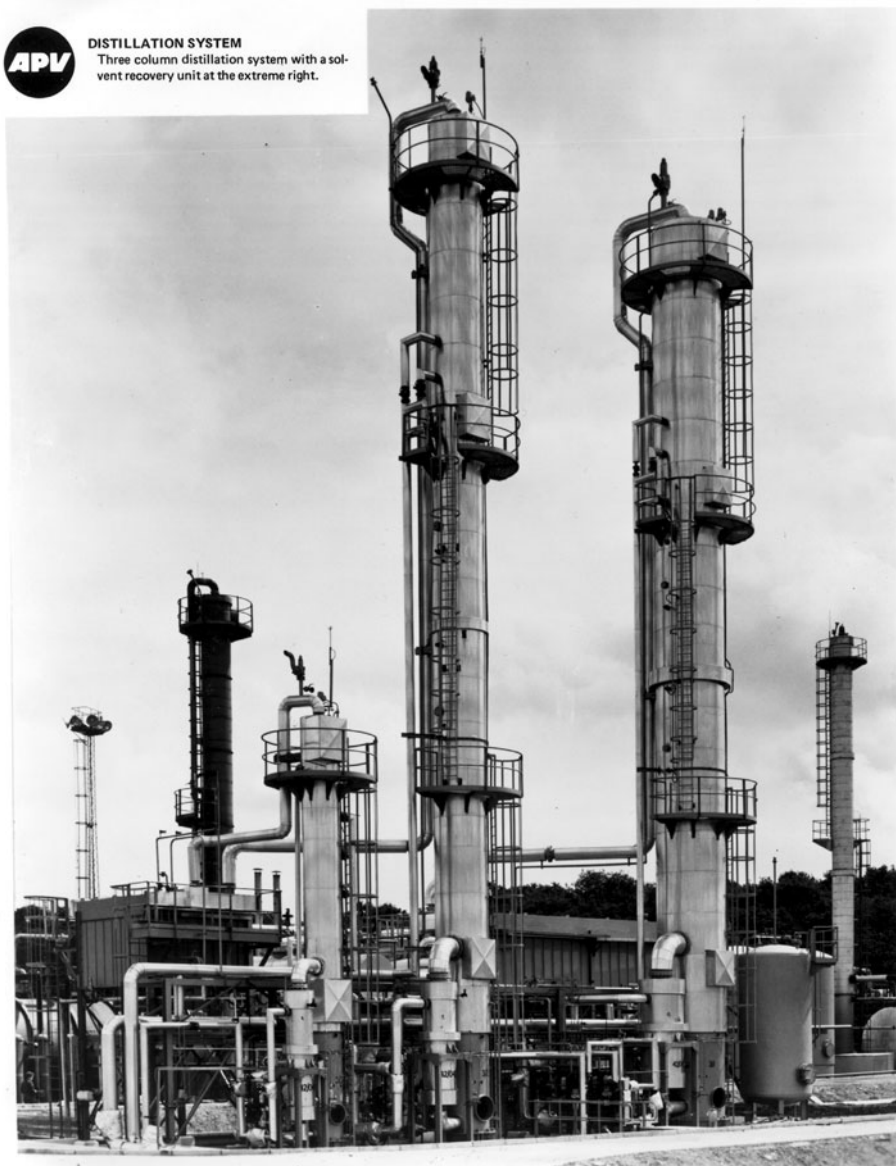


FIGURE 3-6. Distillation column: (A) schematic of five-stage column ($T_1 < T_2 < T_3 < T_4 < T_5 < T_6$), (B) photograph of distillation columns courtesy of APV Equipment, Inc. Tonawanda, NY.



DISTILLATION SYSTEM
Three column distillation system with a solvent recovery unit at the extreme right.



B

FIGURE 3-6. (Continued)

3.2. DISTILLATION EQUIPMENT

It will be helpful for you to have a basic understanding of distillation equipment before studying the design methods. A detailed description of equipment is included in Chapter 10. Figure 3-6A is a schematic of a distillation column, and Figure 3-6B is a photograph of several columns.

The column is usually metal and has a circular cross section. It contains trays (plates or stages) where liquid-vapor contact occurs. The simplest type of tray is a sieve tray, which is a sheet of metal with holes punched into it for vapor to pass through. This is illustrated in Figure 3-7. The liquid flows down from the tray above in a downcomer and then across the sieve tray where it is intimately mixed with the vapor. The vapor flowing up through the holes prevents the liquid from dripping downward, and the metal weir acts as a dam to keep a sufficient level of liquid on the plate. The liquid that flows over the weir is a frothy mixture containing a lot of vapor. This vapor disengages in the downcomer so that clear liquid flows into the stage below. The space above the tray allows for disengagement of liquid from vapor and needs to be high enough to prevent excessive *entrainment* (carryover of liquid from one stage to the next). Distances between trays vary from 2 to 48 inches and tend to be greater the larger the diameter of the column.

To say that there is liquid on the tray is an oversimplification. In practice, any one of four distinct flow regimes can be observed on trays, depending on the gas flow rate. In the *bubble* regime the liquid is close to being a stagnant pool with distinct bubbles rising through it. This regime occurs at low gas flow rates. The poor mixing causes poor liquid and vapor contact, which results in low stage efficiency. Because of the low gas flow rate and low efficiency, the bubble regime is undesirable in commercial applications.

At higher gas flow rates the stage will often be in a *foam* regime. In this regime, the liquid phase is continuous and has fairly distinct bubbles rapidly rising through it. There is a distinct foam similar to the head resting atop the liquid in a mug of beer. Because of the large surface area in a foam, the area for vapor-liquid mass transfer is large, and stage efficiency may be quite high. However, if the foam is too stable it can fill the entire region between stages. When this occurs, entrainment becomes excessive, stage efficiency drops, and the column may *flood* (fill up with liquid and become inoperative). This may require the use of a chemical antifoam agent. The foam regime is usually at vapor flow rates that are too low for most industrial applications.

At even higher vapor flow rates the *froth* regime occurs. In this regime the liquid is continuous and has large, pulsating voids of vapor rapidly passing through it. The surface of the liquid is boiling violently, and there is considerable splashing. The liquid phase is thoroughly mixed, but the vapor phase is not. In most distillation systems where the liquid-phase mass transfer controls, this regime has good efficiency. Because of the good efficiency and reasonable vapor capacity, this is usually the flow regime used in commercial operation.

At even higher gas flow rates the vapor-liquid contact on the stage changes markedly. In the *spray* regime the vapor is continuous and the liquid occurs as a discontinuous spray of droplets. The vapor is very well mixed, but the liquid droplets usually are not. Because of this poor liquid mixing, the mass transfer rate is usually low and stage efficiencies are low. The significance of this is that relatively small increases in vapor velocity can cause the column to go from the froth to the spray regime and cause a significant decrease in stage efficiency (for example, from 65% to 40%).

A variety of other configurations and modifications of the basic design shown in Figures 3-6 and 3-7 are possible. Valve trays (see Chapter 10) are popular. Downcomers can be chords

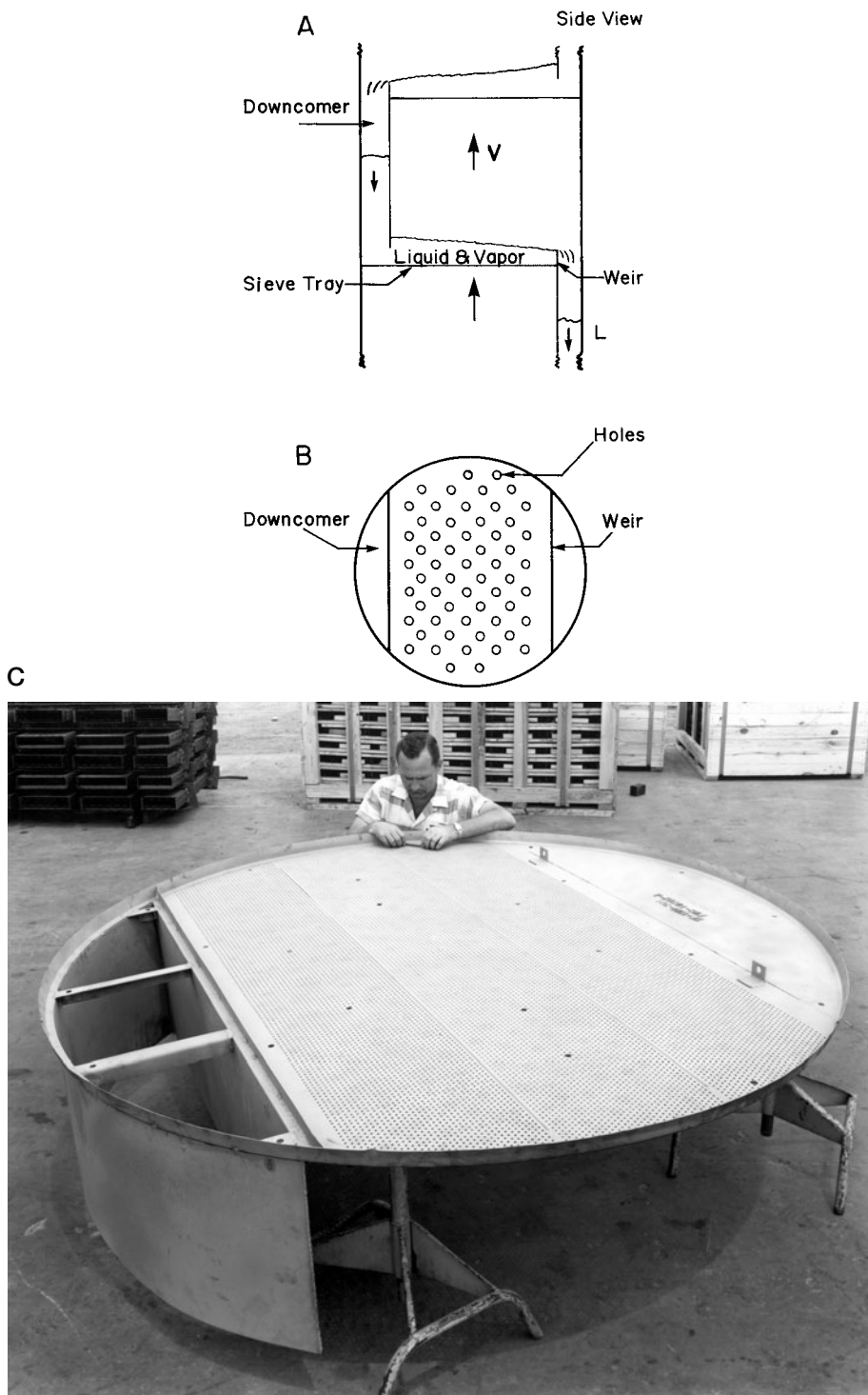


FIGURE 3-7. Sieve trays: A) schematic side view, B) schematic top view, C) photograph courtesy of Glitsch, Inc.

of a circle as shown or circular pipes. Both partial and total condensers and a variety of reboilers are used. The column may have multiple feeds, sidestream withdrawals, intermediate reboilers or condensers, and so forth. The column also usually has a host of temperature, pressure, flow rate, and level measurement and control devices. Despite this variety, the operating principles are the same as for the simple distillation column shown in Figure 3-6.

3.3 SPECIFICATIONS

In the design or operation of a distillation column, a number of variables must be specified. For both design and simulation problems we usually specify column pressure (which sets the equilibrium data); feed composition, flow rate and feed temperature or feed enthalpy or feed quality; and temperature or enthalpy of the reflux liquid. The usual reflux condition set is a saturated liquid reflux. These variables are listed in Table 3-1. The other variables set depend upon the type of problem.

In *design* problems, the desired separation is set, and a column is designed that will achieve this separation. For binary distillation we would usually specify the mole fraction of the more volatile component in the distillate and bottoms products. In addition, the external reflux ratio, L_0/D in Figure 4-6, is usually specified. Finally, we usually specify that the *optimum feed location* be used; that is, the feed location that will result in the fewest total number of stages. The designer's job is to calculate distillate and bottoms flow rates, the heating and cooling requirements in the reboiler and condenser, the number of stages required and the optimum feed stage location, and finally the required column diameter. Alternative specifications such as the splits (fraction of a component recovered in the distillate or bottoms) or distillate or bottoms flow rates are common. Four sets of possibilities are summarized in Table 3-2.

In *simulation* problems, the column has already been built and we wish to predict how much separation can be achieved for a given feed. Since the column has already been built, the number of stages and the feed stage location are already specified. In addition, the column diameter and the reboiler size, which usually control a maximum vapor flow rate, are set. There are a variety of ways to specify the remainder of the problem (see Table 3-3). The desired composition of more volatile component in the distillate and bottoms could be specified, and the engineer would then have to determine the external reflux ratio, L_0/D , that will produce this separation and check that the maximum vapor flow rate will not be exceeded. An alternative is to specify L_0/D and either distillate or bottoms composition, in which case the engineer determines the unknown composition and checks the vapor flow rate. Another alternative is to specify the heat load in the reboiler and the distillate or bottoms composition. The engineer would then determine the reflux ratio and unknown product composition and

TABLE 3-1. *Usual specified variables for binary distillation*

1. Column pressure
2. Feed flow rate
3. Feed composition
4. Feed temperature or enthalpy or quality
5. Reflux temperature or enthalpy (usually saturated liquid)

TABLE 3-2. *Specifications and calculated variables for binary distillation for design problems*

Specified Variables

- A. 1. Mole fraction more volatile component in distillate, x_D
 2. Mole fraction more volatile component in bottoms, x_B
 3. External reflux ratio, L_0/D
 4. Use optimum feed plate
- B. 1,2. Fractional recoveries of components in distillate and bottoms, $(FR_A)_{dist}$, $(FR_B)_{bot}$
 3. External reflux ratio, L_0/D
 4. Use optimum feed plate
- C. 1. D or B
 2. x_D or x_B
 3. External reflux ratio, L_0/D
 4. Use optimum feed plate
- D. 1,2. x_D and x_B
 3. Boilup ratio, V/B
 4. Use optimum feed plate

Designer Calculates

- A. Distillate and bottoms flow rates, D and B
 Heating and cooling loads, Q_R and Q_c
 Number of stages, N
 Optimum feed plate
 Column diameter
- B. x_B , x_D , D, B
 Q_R , Q_c
 N
 N_{feed}
 Column diameter
- C. B or D
 x_B or x_D
 Q_R , Q_c
 N and N_{feed}
 Column diameter
- D. D and B, Q_R and Q_c
 N, N_{feed}
 Column diameter

check the vapor flow rate. The thread that runs through all these alternatives is that since the column has been built, some method of specifying the separation must be used.

The engineer always specifies variables that can be controlled. Several sets of possible specifications and calculated variables are outlined in Tables 3-1 to 3-3. Study these tables to

TABLE 3-3. *Specifications and calculated variables for binary distillation for simulation problems*

<i>Specified Variables</i>	<i>Designer Calculates</i>
A. 1,2. N, N_{feed} 3,4. x_D and x_B Column diameter	A. L_0/D B, D, Q_c, Q_R Check $V < V_{\text{max}}$
B. 1,2. N, N_{feed} 3,4. $L_0/D, x_D$ (or x_B) Column diameter	B. x_B (or x_D) B, D, Q_c, Q_R Check $V < V_{\text{max}}$
C. 1,2. N, N_{feed} 3. x_D (or x_B) 4. Column diameter (set $V = \text{fraction} \times V_{\text{max}}$)	C. $L_0/D, x_B$ (or x_D) B, D, Q_c, Q_R D. B, D, Q_c, x_B (or x_D), L_0/D Check $V < V_{\text{max}}$
D. 1,2. N, N_{feed} 3. Q_R 4. x_D (or x_B) Column diameter	

determine the difference between design-type and simulation-type problems. Note that other combinations of specifications are possible.

In Table 3-1 we find five specified variables common to both types of problems. For design problems (Table 3-2), four additional variables must be set. Note that whereas column diameter is a specified variable in simulation problem C, it serves as a constraint in simulation problems A, B, and D (Table 3-3). Column diameter will allow us to calculate V_{max} and then we can check that $V < V_{\text{max}}$. However, we have not specified a variable for simulation. In problem C, where we specify $V = \text{fraction} \times V_{\text{max}}$, the column diameter serves as a variable for simulation.

Chapter 4 starts with the simple design problem and progresses to simulation and other more complicated problems.

3.4 EXTERNAL COLUMN BALANCES

Once the problem has been specified, the engineer must calculate the unknown variables. Often it is not necessary to solve the entire problem, since only limited answers are required. The first step is to do mass and energy balances around the entire column. For binary design problems, these balances can usually be solved without doing stage-by-stage calculations. Figure 3-8 shows the schematic of a distillation column. The specified variables for a typical design problem are circled. We will assume that the column is well insulated and can be considered adiabatic. All of the heat transfer takes place in the condenser and reboiler. Column pressure is assumed to be constant.

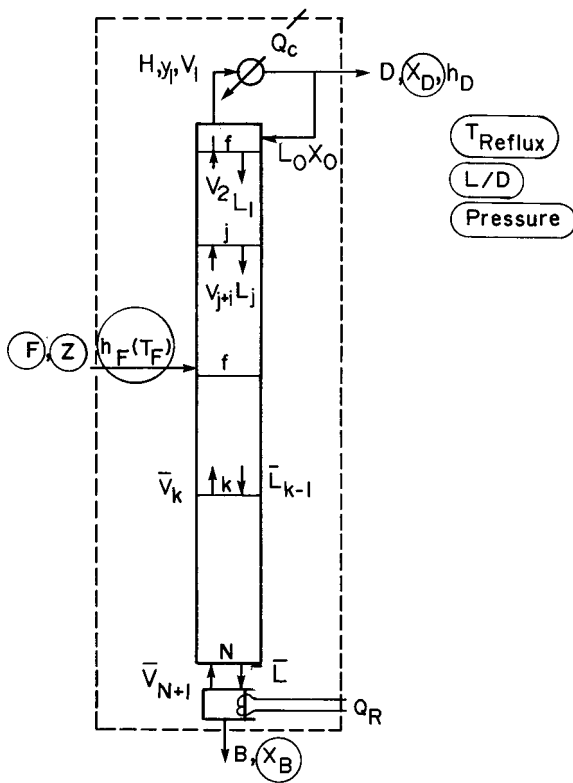


FIGURE 3-8. Binary distillation column: Circled variables are typically specified in design problems

From the balances around the entire column we wish to calculate distillate and bottoms flow rates, D and B , and the heat loads in the condenser and reboiler, Q_c and Q_R . We can start with mass balances around the entire column using the balance envelope shown by the dashed outline in the figure. The overall mass balance is

$$F = B + D \quad (3-1)$$

and the more volatile component mass balance is

$$Fz = Bx_B + Dx_D \quad (3-2)$$

For the design problem shown in Figure 3-8, Eqs. (3-1) and (3-2) can be solved immediately, since the only unknowns are B and D . Solving Eq. (3-1) for B , substituting this into Eq. (3-2), and solving for D , we obtain

$$D = \left(\frac{z - x_B}{x_D - x_B} \right) F \quad (3-3)$$

and

$$B = F - D = \left(\frac{x_D - z}{x_D - x_B} \right) F \quad (3-4)$$

Don't memorize equations like these; they can be derived as needed.

For the energy balance we will use the convention that all heat loads will be treated as inputs. If energy is removed, then the numerical value of the heat load will be negative. The steady-state energy balance around the entire column is

$$Fh_F + Q_c + Q_R = Dh_D + Bh_B \quad (3-5)$$

where we have assumed that kinetic and potential energy and work terms are negligible. The column is assumed to be well insulated and adiabatic. Q_R will be positive and Q_c negative. The enthalpies in Eq. (3-5) can all be determined from an enthalpy-composition diagram (e.g., Figure 2-4) or from the heat capacities and latent heats of vaporization. In general,

$$h_F = h_F(z, T_F, p), h_D = h_D(x_D, T_{\text{reflux}}, p), h_B = h_B(x_B, \text{saturated liquid}, p) \quad (3-6a, b, c)$$

These three enthalpies can all be determined. We would find h_B on Figure 2-4 on the saturated liquid (boiling-line) at $x = x_B$.

Since F was specified and D and B were just calculated, we are left with two unknowns, Q_R and Q_c , in Eq. (3-5). Obviously another equation is required.

For the total condenser shown in Figure 3-8 we can determine Q_c . The total condenser changes the phase of the entering vapor stream but does not affect the composition. The splitter after the condenser changes only flow rates. Thus composition is unchanged and

$$y_1 = x_D = x_0 \quad (3-7)$$

The condenser mass balance is

$$V_1 = L_0 + D \quad (3-8)$$

Since the external reflux ratio, L_0/D , is specified, we can substitute its value into Eq. (3-8).

$$V_1 = \left(\frac{L_0}{D} \right) D + D = \left(1 + \frac{L_0}{D} \right) D \quad (3-9)$$

Then, since the terms on the right-hand side of Eq. (3-9) are known, we can calculate V_1 . The condenser energy balance is

$$V_1 H_1 + Q_c = Dh_D + L_0 h_0 \quad (3-10)$$

Since stream V_1 is a vapor leaving an equilibrium stage in the distillation column, it is a saturated vapor. Thus,

$$H_1 = H_1(y_1, \text{saturated vapor}, p) \quad (3-11)$$

and the enthalpy can be determined (e.g., on the saturated vapor (dew-line) of Figure 2-4 at $y = y_1$). Since the reflux and distillate streams are at the same composition, temperature, and pressure, $h_0 = h_D$. Thus,

$$V_1 H_1 + Q_c = (D + L_0) h_D = V_1 h_D \quad (3-12)$$

Solving for Q_c we have

$$Q_c = V_1 (h_D - H_1) \quad (3-13)$$

or, substituting in Eq. (3-9) and then Eq. (3-3),

$$Q_c = \left(1 + \frac{L_0}{D}\right) D (h_D - H_1) = \left(1 + \frac{L_0}{D}\right) \left(\frac{z - x_B}{x_D - x_B}\right) F (h_D - H_1) \quad (3-14)$$

Note that $Q_c < 0$ because the liquid enthalpy, h_D , is less than the vapor enthalpy, H_1 . This agrees with our convention. If the reflux is a saturated liquid, $H_1 - h_D = \lambda$, the latent heat of vaporization per mole. With Q_c known we can solve the column energy balance, Eq. (3-5), for Q_R .

$$Q_R = D h_D + B h_B - F h_F - Q_c \quad (3-15a)$$

or

$$Q_R = D h_D + B h_B - F h_F + \left(1 + \frac{L_0}{D}\right) D (H_1 - h_D) \quad (3-15b)$$

or

$$Q_R = \left(\frac{z - x_B}{x_D - x_B}\right) F h_D + \left(\frac{x_D - z}{x_D - x_B}\right) F h_B - F h_F + \left(1 + \frac{L_0}{D}\right) \left(\frac{z - x_B}{x_D - x_B}\right) F (H_1 - h_D) \quad (3-16)$$

Q_R will be a positive number. Use of these equations is illustrated in Example 3-1.

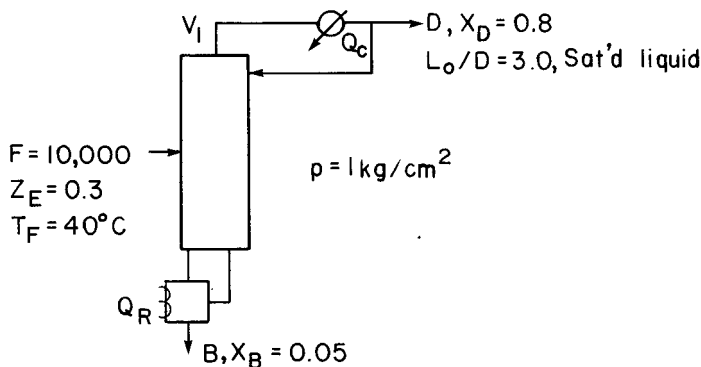
EXAMPLE 3-1. External balances for binary distillation

A steady-state, countercurrent, staged distillation column is to be used to separate ethanol from water. The feed is a 30 wt % ethanol, 70 wt % water mixture at 40 °C. Flow rate of feed is 10,000 kg/hr. The column operates at a pressure of 1 kg/cm². The reflux is returned as a saturated liquid. A reflux ratio of $L/D = 3.0$ is being used. We desire a bottoms composition of $x_B = 0.05$ (weight fraction ethanol) and a distillate composition of $x_D = 0.80$ (weight fraction ethanol). The system has a total condenser and a partial reboiler. Find D , B , Q_c , and Q_R .

Solution

A. Define. The column and known information are sketched in the following figure.

Find D , B , Q_c , Q_R .



- B. Explore. Since there are only two unknowns in the mass balances, B and D, we can solve for these variables immediately. Either solve Eqs. (3-1) and (3-2) simultaneously, or use Eqs. (3-3) and (3-4). For the energy balances, enthalpies must be determined. These can be read from the enthalpy-composition diagram (Figure 2-4). Then Q_c can be determined from the balance around the condenser and Q_R from the overall energy balance.
- C. Plan. Use Eqs. (3-3) and (3-4) to find D and B, Eq. (3-14) to determine Q_c , and Eq. (3-15a) to determine Q_R .
- D. Do It. From Eq. (3-3),

$$D = F \left(\frac{z - x_B}{x_D - x_B} \right) = 10,000 \left[\frac{0.3 - 0.05}{0.8 - 0.05} \right] = 3333 \text{ kg/hr}$$

From Eq. (3-4), $B = F - D = 10,000 - 3333 = 6667 \text{ kg/hr}$

From Figure 2-4 the enthalpies are

$h_D(x_D = 0.8, \text{ saturated liquid}) = 60 \text{ kcal/kg}$

$h_B(x_B = 0.05, \text{ saturated liquid}) = 90 \text{ kcal/kg}$

$h_F(z = 0.3, 40^\circ\text{C}) = 30 \text{ kcal/kg}$

$H_1(y_1 = x_D = 0.8, \text{ saturated vapor}) = 330 \text{ kcal/kg}$

From Eq. (3-14),

$$Q_c = \left(1 + \frac{L_0}{D} \right) D (h_D - H_1) = (1 + 3)(3333)(60 - 330) = -3,559,640 \text{ kcal/hr}$$

From Eq. (3-15a), $Q_R = Dh_D + Bh_B - Fh_F - Q_c$

$$Q_R = (3333)(60) + (6667)(90) - (10,000)(30) - (-3,559,640) = 4,099,650 \text{ kcal/hr}$$

E. Check. The overall balances, Eqs. (3-1) and (3-5), are satisfied. If we set up this problem on a spreadsheet without explicitly solving for D, B, Q_c , and Q_R we obtain identical answers.

F. Generalize. In this case we could solve the mass and energy balances sequentially. This is not always the case. Sometimes the equations must be solved simultaneously (see Problem 3-D3). Also, the mass balances and energy bal-

ances derived in the text were for the specific case shown in Figure 3-8. When the column configuration is changed, the mass and energy balances change (see Problem 3-D2, 3-D3, and 3-D5). For binary distillation we can usually determine the external flows and energy requirements from the external balances. Exceptions will be discussed in Chapter 4.

3.5 SUMMARY—OBJECTIVES

In this chapter we have introduced the idea of distillation columns and have seen how to do external balances. At this point you should be able to satisfy the following objectives:

1. Explain how a countercurrent distillation column physically works
2. Sketch and label the parts of a distillation system; explain the operation of each part and the flow regime on the trays
3. Explain the difference between design and simulation problems; list the specifications for typical problems
4. Write and solve external mass and energy balances for binary distillation systems

REFERENCES

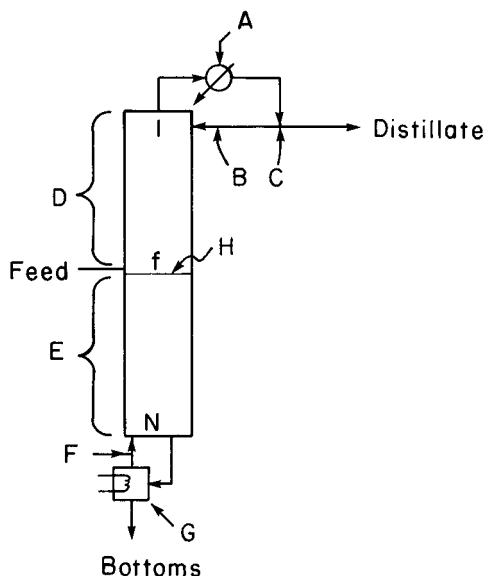
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Humphrey, J. L. and G. E. Keller II, *Separation Process Technology*, McGraw-Hill, New York, 1997.

HOMEWORK

A. Discussion Problems

- A1. Explain how a distillation column works.
- A2. Without looking at the text, define the following:
 - a. Isothermal distillation
 - b. The four flow regimes in a staged distillation column
 - c. Reflux and reflux ratio
 - d. Boilup and boilup ratio
 - e. Rectifying (enriching) and stripping sections
 - f. Simulation and design problemsCheck the text for definitions you did not know
- A3. Explain the reasons a constant pressure distillation column is preferable to:
 - a. An isothermal distillation system.
 - b. A cascade of flash separators at constant temperature.
 - c. A cascade of flash separators at constant pressure.
- A4. In a countercurrent distillation column at constant pressure, where is the temperature highest? Where is it lowest?
- A5. Develop your own key relations chart for this chapter. In one page or less draw sketches, write equations, and include all key words you would want for solving problems.
- A6. Explain the difference between sequential and simultaneous solution of the external mass and energy balances.

- A7.** What type of specifications will lead to simultaneous solution of the mass and energy balances?
- A8.** What are the purposes of reflux? How does it differ from recycle?
- A9.** Without looking at the text, name the streams or column parts labeled A to H in the following figure.



B. Generation of Alternatives

- B1.** There are ways in which columns can be specified other than those listed in Tables 3-1 to 3-3.
- Develop alternative specifications for design problems.
 - Develop alternative specifications for simulation problems.

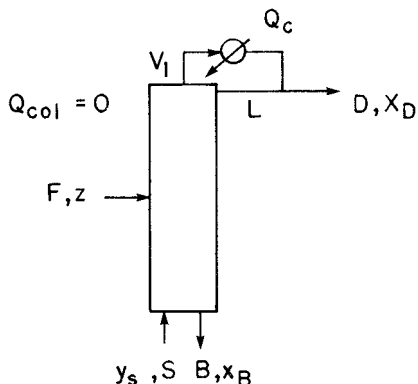
C. Derivations

- C1.** For the column shown in Problem 3-D2, derive equations for D , B , Q_c , and L/D .
- C2.** For the column shown in Problem 3-D3, derive equations for D , B , $\bar{V}$, and Q_R .

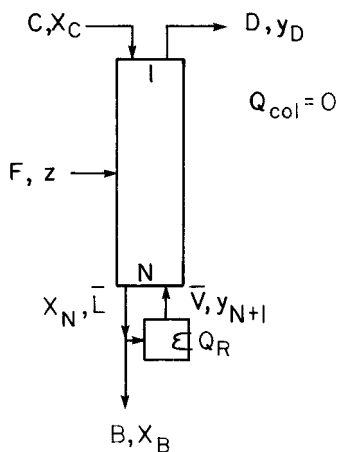
D. Problems

**Answers to problems with an asterisk are at the back of the book.*

- D1.** A very large distillation column is separating p-xylene (more volatile) from o-xylene. The column has two feeds (similar to Figure 4-17, but without the side stream). Both feeds are saturated liquids. Feed 1 is 42 mol % p-xylene, $F_1 = 90$ kmole/hr. Feed 2 is 9 mole % p-xylene, $F_2 = 20$ kmole/hr. The bottoms product should be 97 % o-xylene and the distillate product should be 99% p-xylene. Find the flow rates D and B .
- D2.*** A distillation column separating ethanol from water is shown. Pressure is 1 kg/cm². Instead of having a reboiler, steam (pure water vapor) is injected directly into the bottom of the column to provide heat. The injected steam is a saturated vapor. The feed is 30 wt % ethanol and is at 20 °C. Feed flow rate is 100 kg/min. Reflux is a saturated liquid. We desire a distillate concentration of 60 wt % ethanol and a bottoms product that is 5 wt % ethanol. The steam is input at 100 kg/min. What is the external reflux ratio, L/D ?



- D3.*** A distillation column separating ethanol from water is shown. Pressure is 1 kg/cm^2 . Instead of having a condenser, a stream of pure liquid ethanol is added directly to the column to serve as the reflux. This stream is a saturated liquid. The feed is 40 wt % ethanol and is at -20°C . Feed flow rate is 2000 kg/hr . We desire a distillate concentration of 80 wt % ethanol and a bottoms composition of 5 wt % ethanol. A total reboiler is used, and the boilup is a saturated vapor. The cooling stream is input at $C = 1000 \text{ kg/hr}$. Find the external boilup rate, $\bar{V}$. Note: Set up the equations, solve in equation form for $\bar{V}$ including explicit equations for all required terms, read off all required enthalpies from the enthalpy composition diagram (Figure 2-4), and then calculate a numerical answer.



- D4.** A distillation column with two feeds is separating ethanol from water. The first feed is 60 weight % ethanol, has a total flow rate of 1000 kg/hr and is a mix of liquid and vapor at 81°C . The second feed is 10 weight % ethanol, has a total flow rate of 500 kg/hr and is liquid at 20°C . We desire a bottoms product that is 0.01 weight % ethanol and a distillate product that is 85 weight % ethanol. The column operates at 1 kg/cm^2 and is adiabatic. The column has a partial reboiler, which acts as an equilibrium contact, and

a total condenser. The distillate and reflux are saturated liquids. Find B & D in kg/hr, and find Q_c & Q_R in kcal/hr. Use data in Figure 2-4. Do both parts a & b.

a. External reflux ratio, $L_0/D = 3.0$

b. Boilup ratio, $\bar{V}/B = 2.5$.

D5.* A distillation column is separating ethanol from water at a pressure of 1 kg/cm². A two-phase feed of 20 wt% ethanol at 93 °C is input at 100 kg/min. The column has a total condenser and a partial reboiler. The distillate composition is 90 wt % ethanol. Distillate and reflux are at 20 °C. Bottoms composition is 1 wt % ethanol. Reflux ratio is $L_0/D = 3$. A liquid side stream is withdrawn above the feed stage. Side stream is 70 wt % ethanol, and side stream flow rate is 10 kg/min. Find D, B, Q_c , and Q_R . Data are in Figure 2-4.

D6.* A distillation column receives a feed that is 40 mole % n-pentane and 60 mole % n-hexane. Feed flow rate is 2500 lb moles/hr and feed temperature is 30 °C. The column is at 1 atm. A distillate that is 99.9 mole % n-pentane is desired. A total condenser is used. Reflux is a saturated liquid. The external reflux ratio is $L_0/D = 3$. Bottoms from the partial reboiler is 99.8 mole % n-hexane. Find D, B, Q_R , Q_c . Note: Watch your units on temperature.

Data: λ_{c5} = 11,369 Btu/lb-mole

λ_{c6} = 13,572 Btu/lb-mole, both λ at boiling points.

$C_{PL,C5}$ = 39.7 (assume constant)

$C_{PL,C6}$ = 51.7 (assume constant)

$C_{PV,C5}$ = $27.45 + 0.08148 T - 4.538 \times 10^{-5} T^2 + 10.1 \times 10^{-9} T^3$

$C_{PV,C6}$ = $32.85 + 0.09763 T - 5.716 \times 10^{-5} T^2 + 13.78 \times 10^{-9} T^3$

Where T is in °C and C_{PV} and C_{PL} are

$$\frac{\text{cal}}{\text{g mole } ^\circ\text{C}} \text{ or } \frac{\text{Btu}}{\text{lb mole } ^\circ\text{F}}$$

D7. A continuous, steady-state distillation column is fed a mixture that is 70 mole % n-pentane and 30 mole % n-hexane. Feed rate is 1000 kmol/hr. Feed is at 35 °C. Column is at 101.3 kPa. The vapor distillate product is 99.9 mole % n-pentane and the bottoms product is 99.9 mole % n-hexane. The system has a partial condenser (thus the distillate product is a saturated vapor) and operates at an external reflux ratio of $L/D = 2.8$. The reboiler is a partial reboiler. Find D, B, Q_c & Q_R . Data are given in problem 3.D6. Use DePriester chart to determine boiling points.

D8. (Long problem!) A mixture of methanol and water is being separated in a distillation column with open steam (see Figure in Problem 3.D2). The feed rate is 100 kmol/hr. Feed is 60.0 mole % methanol and is at 40 °C. The column is at 1.0 atm. The steam is pure water vapor ($y_M = 0$) and is a saturated vapor. The distillate product is 99.0 mole % methanol and leaves as a saturated liquid. The bottoms is 2.0 mole % methanol and since it leaves an equilibrium stage must be a saturated liquid. The column is adiabatic. The column has a total condenser. External reflux ratio is $L/D = 2.3$.

Equilibrium data is in Table 2-7 in problem 2.D1. Data for water and methanol is available in Felder and Rousseau (C_p , λ and steam tables) and in Perry's.

Find D, B, Q_c , and S. Be careful with units and in selecting basis for energy balance.

Data:

$\lambda_{\text{methanol}} = \Delta H_{\text{vap}} = 8.43 \text{ kcal / gmole} = 35.27 \text{ kJ / gmole}$ (at boiling point)

$\lambda_{\text{water}} = \Delta H_{\text{vap}} = 9.72 \text{ kcal / gmole} = 40.656 \text{ kJ / gmole}$

$$C_{p,\text{wliquid}} = 1.0 \text{ cal / g } ^\circ\text{C} = 75.4 \text{ J / gmole } ^\circ\text{C}$$

$$C_{PL,\text{Meoh}} = 75.86 + 0.1683T \text{ J/(gmole } ^\circ\text{C)}$$

$$C_{p,\text{wvapor}} = 33.46 + 0.006880T + 0.7604 \times 10^{-5}T^2 - 3.593 \times 10^{-9}T^3$$

$$C_{p,\text{meoh,vapor}} = 42.93 + 0.08301T - 1.87 \times 10^{-5}T^2 - 8.03 \times 10^{-9}T^3$$

For Vapor T is in $^\circ\text{C}$, C_p is in J / gmole $^\circ\text{C}$

VLE data: Table 2-7. Density and MW data Problem 2.D1

Reference for λ and heat capacity data is Felder and Rousseau (2000).

- D9.** (Long problem!) A mixture of methanol and water is being separated in a distillation column with open steam (see Figure in Problem 3.D2). The feed rate is 500 kmol/hr. Feed is 60.0 mole % methanol and is a saturated liquid. The column is at 1.0 atm. The steam is pure water vapor ($y_M = 0$) and is a saturated vapor. The distillate product is 99.8 mole % methanol and leaves as a saturated liquid. The bottoms is 0.13 mole % methanol and since it leaves an equilibrium stage must be a saturated liquid. The column is adiabatic. The column has a total condenser. External reflux ratio is $L/D = 3$. Data for water and methanol is available in problem 3.D8.

Find D, B, Q_c , and S. Be careful with units and in selecting basis for energy balance.

- D10.** A distillation column operating at 2.0 atm. is separating a feed that is 55.0 mole % n-pentane and 45.0 mole % n-hexane. The feed is at 65°C and its flow rate is 1000 kg moles/hr. The distillate is 99.93 mole % n-pentane and we want a 99.50% recovery of n-pentane. The system uses a total condenser and reflux is a saturated liquid with an external reflux ratio of $L/D = 2.8$. There is a partial reboiler. Data is available in problem 3.D6 and in the DePriester charts. Find D, B, x_B , Q_c , Q_R .

F. Problems Requiring Other Resources

- F1.*** A mixture of oxygen and nitrogen is to be distilled at low temperature. The feed rate is 25,000 kg moles/hr and is 21 mole % oxygen and 79 mole % nitrogen. An ordinary column (as shown in Figure 3-8) will be used. Column pressure is 1 atm. The feed is a superheated vapor at 100 K. We desire a bottoms composition of 99.6 mole % oxygen and a distillate that is 99.7 mole % nitrogen. Reflux ratio is $L_0/D = 4$, and reflux is returned as a saturated liquid. Find D, B, Q_R , and Q_c .

- F2.*** A mixture of water and ammonia is to be distilled in an ordinary distillation system (Figure 3-8) at a pressure of 6 kg/cm². The feed is 30 wt % ammonia and is at 20°C . We desire a distillate product that is 98 wt % ammonia and a 95% recovery of the ammonia in the distillate. The external reflux ratio is $L_0/D = 2.0$. Reflux is returned at -20°C . Find D, B, x_B , Q_R , and Q_c per mole of feed.

G. Computer Problems.

- G1.** Solve for Q_c and Q_R in problem 3.D4 with a process simulator.

a. Part a.

b. Part b.

Note: With Aspen Plus, use RADFRAC (see Appendix to Chapter 6, Lab 3) with an arbitrary (but large) number of stages and feed location = $N/2$. Do calculation for D by hand and input correct values for D and L/D (or $\bar{V}/B$)