

## chapter 5

# Countercurrent Multistage Operations

Since the two phases leaving an equilibrium stage are in equilibrium, no separation of valuable constituents greater than that at equilibrium is possible in one stage. It is possible to increase the recovery of a valuable constituent by contacting the phase with fresh solvent in several equilibrium stages, as discussed in Chapter 4. However, the use of a fresh solvent feed to each stage requires large quantities of solvent and produces a very dilute solution of solute in solvent. To conserve solvent and to produce a more highly concentrated extract product, countercurrent multistage contacting of phases is employed. An example of industrial countercurrent multistage contacting is shown in Figure 5.1.

In countercurrent multistage contact the two phases enter at opposite ends of a series, or cascade, of equilibrium stages (Figure 5.2). The phases flow in directions counter to each other. In this way the concentration of solute in the  $V$ -phase product can be increased, and a high recovery of solute is possible with the use of a smaller quantity of solvent. This discussion has used the terms of extraction, but the principles of countercurrent multistage contact apply to all stage operations.

Usually one of two questions is answered by calculations for multistage equipment.

1. How many equilibrium stages are required to obtain the desired product separation or recovery?
2. What product recovery can be obtained with existing equipment of a known number of equilibrium stages?

In answering either question both the equilibrium relationships and the relative quantities of the two phases must be considered.

### THE OVER-ALL MATERIAL BALANCE

In Figure 5.2  $L$  and  $V$  refer to the mass of each phase;  $x$  and  $y$  refer to the composition of phases  $L$  and  $V$

respectively; and  $h$  and  $H$  refer to the concentration of any conserved property in phases  $L$  and  $V$ , respectively. The mass may be expressed in pounds or pound moles, and the composition as mass or mole fraction. The subscripts 0, 1, 2, etc., identify the stage *from* which a stream is flowing.  $N$  refers to the *last* stage in a cascade, whereas  $n$  is used to designate *any* stage in the cascade.

The conserved property most frequently of interest in stage operations is the enthalpy of the phases. Therefore,  $h$  and  $H$  will refer the enthalpy per unit mass of the phase. In the following development the equations involving composition and enthalpy will both be written. It will be seen that the equations are of identical form and that it would be possible to write a single general set of equations which could be used to express the conservation of either mass or enthalpy.

An over-all material and enthalpy balance around the entire cascade gives

Total material balance:

$$L_0 + V_{N+1} = L_N + V_1 = \Sigma \quad (5.1)$$

Component balance:

$$L_0 x_0 + V_{N+1} y_{N+1} = L_N x_N + V_1 y_1 = \Sigma z_\Sigma \quad (5.2)$$

Enthalpy balance:

$$L_0 h_0 + V_{N+1} H_{N+1} = L_N h_N + V_1 H_1 = \Sigma h_\Sigma \quad (5.3)$$

These equations state that the material and enthalpy entering the cascade (in streams  $L_0$  and  $V_{N+1}$ ) must equal the material and enthalpy leaving the cascade (in streams  $L_N$  and  $V_1$ ).

Equations 5.1, 5.2, and 5.3 also define the sum or sigma point, which is useful in calculating balances around the entire cascade. Substitution for  $\Sigma$  in Equation 5.2 from Equation 5.1 gives

$$z_\Sigma = \frac{L_0 x_0 + V_{N+1} y_{N+1}}{L_0 + V_{N+1}} = \frac{L_N x_N + V_1 y_1}{L_N + V_1} \quad (5.4)$$

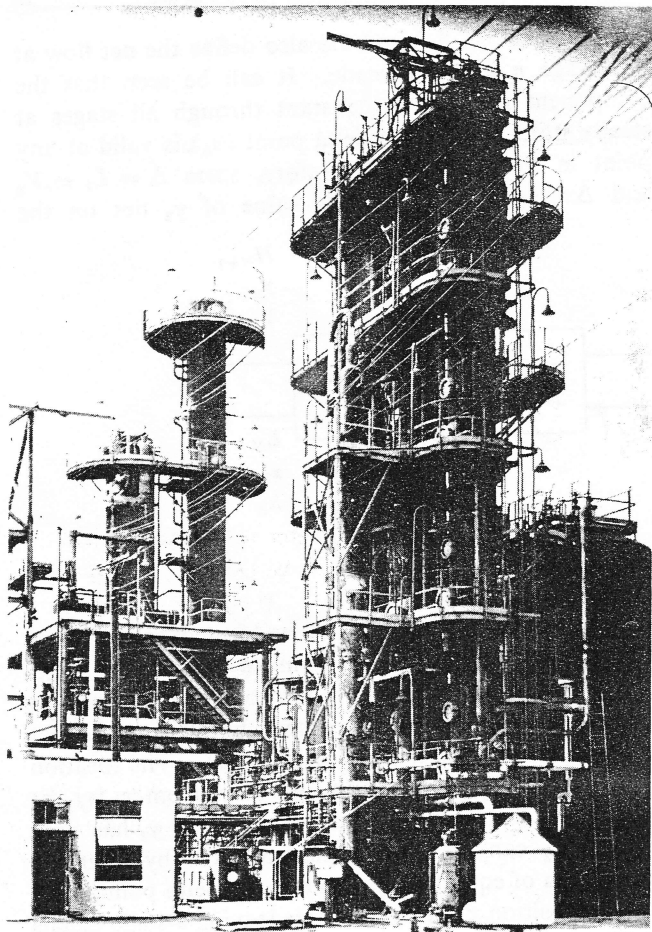


Figure 5.1. Liquid-liquid extraction applied to the purification of gasoline. The three towers pictured in the foreground are used to extract undesirable mercaptans from a gasoline fraction of crude oil to produce a low-sulfur gasoline. The three towers are operated in parallel. Raw gasoline entering at the bottom of each tower is contacted countercurrently with caustic methanol in the lower section to remove the mercaptans. In the upper section of each tower the gasoline is contacted with a caustic solution to recover methanol which has dissolved in the gasoline. The methanol is fed to each tower between the upper and lower sections, and the caustic solution is supplied at the top of the tower. The columns are  $3\frac{1}{2}$  ft, 4 ft, and 5 ft in diameter and 63 ft high. Each column has nine perforated plates. Auxiliary equipment includes a stripping tower where the methanol is removed from the caustic solution by steam stripping and a distillation tower where the methanol is recovered from an aqueous solution. (Atlantic Refining Company.)

For the enthalpy coordinate of the sigma point, combination of Equations 5.1 and 5.3 gives

$$h_{\Sigma} = \frac{L_0 h_0 + V_{N+1} H_{N+1}}{L_0 + V_{N+1}} = \frac{L_N h_N + V_1 H_1}{L_N + V_1} \quad (5.5)$$

The equations are simply analytical expressions of the addition rule developed in Chapter 4. Examination of the group on the right side of Equation 5.4 shows that it is simply the sum of the masses of one component

divided by the sum of the total masses of the two streams  $L_N$  and  $V_1$ . Similarly, Equation 5.5 gives the sum of the enthalpies of the two streams divided by the total masses of the streams. Calculations utilizing Equations 5.4 and 5.5 are often more easily accomplished graphically. The procedure is shown in Figure 5.3. For example, perhaps the mass and composition of  $L_0$  and  $V_{N+1}$  are known. It is then possible to locate  $z_{\Sigma}$ , as shown. Then, if  $x_N$  is known,  $y_1$  can be determined by drawing a straight line between  $x_N$  and  $z_{\Sigma}$  and extending it to intersect the extract curve. The masses of  $L_N$  and  $V_1$  can be determined by applying the inverse lever-arm rule to the line  $\overline{x_N z_{\Sigma} y_1}$ .

### STAGE-TO-STAGE CALCULATIONS

In one type of calculation for countercurrent flow enough information is available to calculate the over-all material balance, as outlined above. After an over-all material balance has been used to determine the end conditions of the cascade, stage-to-stage calculations may be made to determine the number of equilibrium stages required to give the desired end conditions.

Calculation may proceed from either end of the cascade. If, for example,  $y_1$  and  $H_1$  are known, use of the equilibrium relationship will give  $x_1$  and  $h_1$ . It is now necessary to develop an interrelationship between  $x_1$ ,  $h_1$  and  $y_2$ ,  $H_2$ . The two streams  $L_1$  and  $V_2$  are passing each other between stages 1 and 2. In order to calculate the quantity and composition of  $V_2$  from those of  $L_1$  the new concept of *net flow* must be introduced. Since there is no accumulation of a constituent, a property, or a total quantity in the cascade, the net flow is constant throughout the cascade. The net flow is a fictitious stream. It is the difference between two streams which are not in reality subtracted one from the other.

Net flow to the *right* in the cascade shown in Figure 5.2 is defined as the difference between the flow to the right and the flow to the left, or

Total net flow:

$$\Delta = L_0 - V_1 = L_n - V_{n+1} = L_N - V_{N+1} \quad (5.6)$$

Component net flow:

$$\begin{aligned} \Delta x_{\Delta} &= L_0 x_0 - V_1 y_1 = L_n x_n - V_{n+1} y_{n+1} \\ &= L_N x_N - V_{N+1} y_{N+1} \end{aligned} \quad (5.7)$$

Property net flow:

$$\begin{aligned} \Delta h_{\Delta} &= L_0 h_0 - V_1 H_1 = L_n h_n - V_{n+1} H_{n+1} \\ &= L_N h_N - V_{N+1} H_{N+1} \end{aligned} \quad (5.8)$$

The total net flow ( $\Delta$ ) of material at the left end of the cascade is equal to that material flowing to the right ( $L_0$ ) minus that flowing to the left ( $V_1$ ). Similarly, at the right end of the cascade, the total net flow ( $\Delta$ ) is equal to  $L_N - V_{N+1}$ . By Equation 5.1

$$L_0 - V_1 = L_N - V_{N+1}$$

Therefore, the total net flow at any stage in the cascade is constant. If the mass of  $V_{N+1}$  is greater than  $L_N$ ,  $\Delta$  is negative, and the net flow is to the left in Figure 5.2. Since a net flow to the right is defined as a positive quantity, an actual net flow of any component to the left has a negative numerical value.

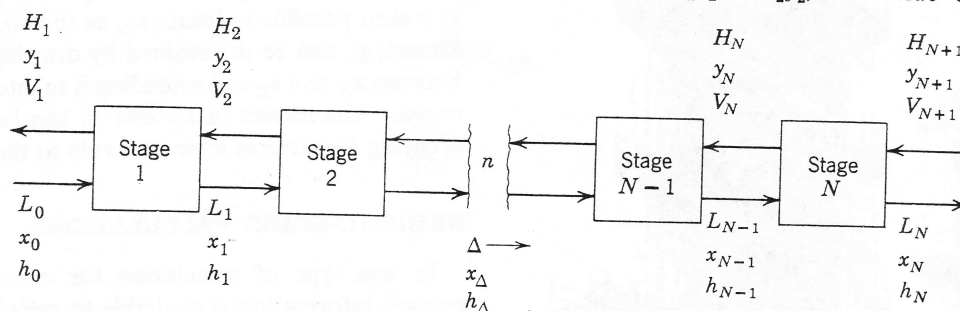


Figure 5.2. Countercurrent multistage contact.

Equation 5.7 defines the net flow of one component. For example, if copper sulfate is to be extracted from a roasted ore ( $L_0$ ) by water ( $V_{N+1}$ ), there will be a net flow of insoluble gangue and copper sulfate to the right and a net flow of water to the left. The term  $x_\Delta$  is a fictitious concentration of  $\Delta$  which would result if  $V$  were actually subtracted from  $L$ . Combining Equations 5.6 and 5.7 gives

$$\begin{aligned} x_\Delta = \frac{\Delta x_\Delta}{\Delta} &= \frac{L_0 x_0 - V_1 y_1}{L_0 - V_1} = \frac{L_n x_n - V_{n+1} y_{n+1}}{L_n - V_{n+1}} \\ &= \frac{L_N x_N - V_{N+1} y_{N+1}}{L_N - V_{N+1}} \\ &= \frac{\text{net flow of component}}{\text{total net flow}} \end{aligned} \quad (5.9)$$

The numerators of Equation 5.9 are the net flow of any component, and the denominators are the total net flow. Therefore, if the net flow of a component is in a direction opposite to the total net flow,  $x_\Delta$  will have a negative value for that component.

Application of the graphical-addition rule to Equations 5.6 and 5.7 for the entire cascade shows that  $x_\Delta$  should lie on the straight line  $\overline{y_1 x_0}$  extended and also on the line  $\overline{y_{N+1} x_N}$  extended. This is shown in Figure 5.4. For the case shown,  $x_{\Delta a}$  and  $x_{\Delta b}$  are negative, and  $x_{\Delta c}$  is positive and greater than 1.0. This indicates that the net flows of the solute and major raffinate component are in a direction opposite to the total net flow, but the net flow of solvent is in the same direction as the total net flow and is larger than the total net flow. Even for the fictitious stream  $\Delta$ ,  $x_a + x_b + x_c$  must equal 1.0. Therefore, if  $x_a$  and  $x_b$  are negative,  $x_c$  must be greater than 1.0. The delta point located by using the concentrations at the ends of the cascade will be used to calculate from stage to stage, as shown in the following discussion.

Equations 5.6, 5.7, and 5.8 also define the net flow at any stage  $n$  in the cascade. It can be seen that the component net flow is constant through all stages at steady state, so that the delta point ( $x_\Delta$ ) is valid at any point in the cascade. Therefore, since  $\Delta = L_1 - V_2$  and  $\Delta x_\Delta = L_1 x_1 - V_2 y_2$ , the value of  $y_2$  lies on the

straight line  $\overline{x_\Delta x_1}$ . It must also be located on the phase envelope since  $V_2$  is flowing from an equilibrium stage. The determination of the compositions of the phases in successive stages may now proceed by alternate use of the equilibrium data and the delta point. The location of  $x_1$  from  $y_1$  by tie-line data and of  $y_2$  from  $x_1$  by the delta point is shown in Figure 5.4. When  $y_2$  has been located,  $x_2$  can be found, and so forth by alternate application of equilibrium data and the delta point, until the composition  $x_n$  of the raffinate leaving a stage equals or surpasses the final desired composition,  $x_N$ . The stage-to-stage calculation can also be made in the opposite direction, from  $x_N$  to  $y_1$ . The choice of direction depends upon the original information given. In some cases the percentage recovery in the extract of the solute originally supplied is specified. It is possible to calculate the exit concentrations from this information.

Combination of Equations 5.6 and 5.8 gives the enthalpy coordinate of the delta point,

$$\begin{aligned} h_\Delta = \frac{L_0 h_0 - V_1 H_1}{L_0 - V_1} &= \frac{L_n h_n - V_{n+1} H_{n+1}}{L_n - V_{n+1}} \\ &= \frac{L_N h_N - V_{N+1} H_{N+1}}{L_N - V_{N+1}} \end{aligned} \quad (5.10)$$

which may be used on enthalpy-composition diagrams together with Equation 5.9 to locate the delta point. Calculation of the over-all material and enthalpy balances and calculation from stage to stage on an enthalpy-composition diagram follows the procedures outlined previously for ternary composition diagrams. The enthalpy may be considered to replace the solvent.

**Illustration 5.1.** Acetic acid is to be extracted from 2000 lb/hr of a 40 per cent aqueous solution by countercurrent extraction with 3000 lb/hr of pure isopropyl ether.

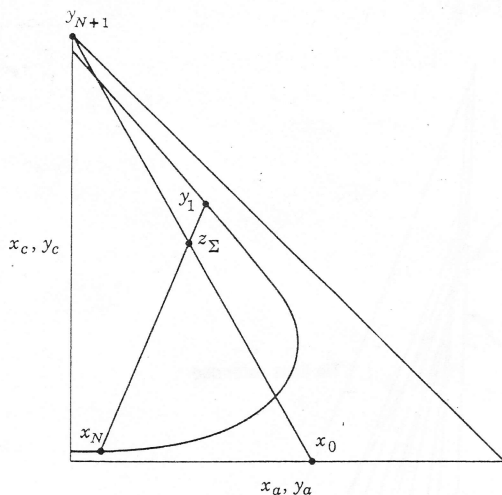


Figure 5.3. Graphical determination of the over-all material balance.

The concentration of acetic acid is to be reduced to 3 per cent in the exit raffinate. How many equilibrium stages are required to reduce the acetic acid concentration to 3 per cent?

**SOLUTION.** The complete solution is given on Figure 5.5. In this case there are three components and no conserved property to consider. Therefore, a three-component (ternary) diagram will be used.  $y_1$  is located by an over-all material balance: First  $z_{\Sigma}$  is located on line  $x_0y_{N+1}$  by the inverse lever-arm rule.

$$\frac{L_0}{V_{N+1}} = \frac{2000}{3000} = \frac{y_{N+1} - z_{\Sigma}}{z_{\Sigma} - x_0} = \frac{2}{3}.$$

Then  $y_1$  must lie on the phase envelope on the extension of line  $x_Nz_{\Sigma}$ , since  $L_N + V_1 = \Sigma$ .

The delta point is now located on the extensions of the straight lines  $x_0y_1$  and  $x_Ny_{N+1}$ . The calculation of stages may start at either  $y_1$  or  $x_N$ . Starting with the final extract ( $y_1$ ),  $x_1$  must be in equilibrium with  $y_1$ , and the value of  $x_1$  is determined from the equilibrium curve as shown by the construction. Since  $L_1 - V_2 = \Delta$ ,  $y_2$  lies at the intersection of the straight line  $x_{\Delta}x_1$  and the phase envelope.  $x_2$  is now determined as before from equilibrium data, and the construction proceeds until  $x_n$  equals or surpasses  $x_N$ . This occurs between  $x_{14}$  and  $x_{15}$ . Therefore, approximately 14.5 equilibrium stages are required. The dashed lines of Figure 5.4 are equilibrium tie lines for the stages of this illustration. The construction lines to  $x_{\Delta}$  have been omitted after the first two to avoid obscuring the stage tie lines.

The coordinates of the delta point can be read from Figure 5.5 or calculated from Equation 5.9 as  $x_a = -0.012$ ,  $x_b = -0.45$ ,  $x_c = 1.46$ . The negative values for  $x_a$  and  $x_b$  indicate that the net flows of acetic acid and water are in a direction opposite to the total net flow. In this case the total net flow is to the left. Therefore, the net flow of acetic acid and water is to the right. Even for this fictitious composition,  $x_a + x_b + x_c = -0.012 + (-0.45) + 1.46 \doteq 1$ .

## OPERATING VARIABLES

If the flow rates of the  $L$  and  $V$  phases have been specified, the number of equilibrium stages required for a specified separation may be determined. On the other hand it may be necessary to evaluate the operation of existing equipment of a known number of equilibrium stages for a new separation. In this case usually either the  $L$ -phase or the  $V$ -phase may be specified, and the other phase may be adjusted to give the desired separation. If both  $L$  and  $V$  and the number of stages are specified, the separation is determined.

In a typical extraction problem, as the solvent flow ( $V$ ) is decreased, more equilibrium stages are required to give the specified recovery of solute. Finally a value of solvent flow is reached where an infinite number of stages is required to give the recovery. This is referred to as the minimum  $V$ -phase flow or as the minimum  $V/L$ . Although the minimum  $V/L$  has no direct practical use, it is useful as a limiting value for the actual flow. Often the actual  $V/L$  will be taken as some arbitrary factor times the minimum.

The minimum  $V/L$  occurs at the highest value of  $V$  which gives a line through the delta point that coincides with any extended tie line. Such a coincidence results in no change of composition from stage to stage and requires an infinite number of stages to give a finite change in composition. Because the lines representing the stages are crowded together, the point at which the infinite number of stages occurs is referred to as the "pinch." At values of  $V/L$  lower than the minimum, the desired separation cannot be made, even with an

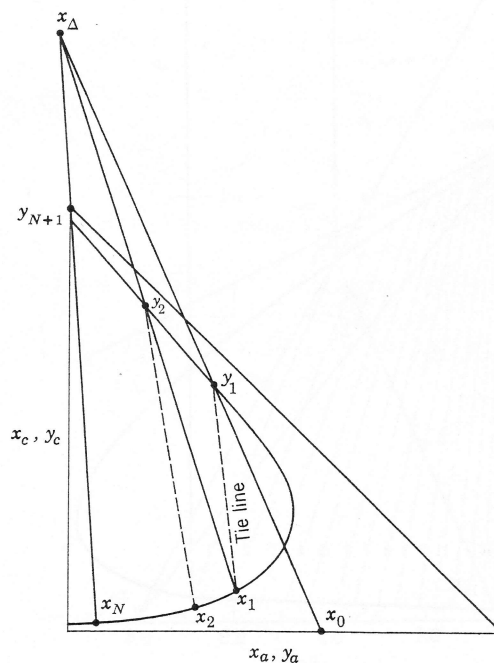


Figure 5.4. Graphical determination of delta point.





A minimum  $V/L$  can be defined in a separation process where the component of interest is to be transferred from the  $L$ -phase to the  $V$ -phase and the recovery of the component is specified, such as in the usual case of liquid extraction. On the other hand, in many operations, such as gas absorption, the component of interest is transferred in the opposite direction, from the  $V$ -phase to the  $L$ -phase. In this case it is possible to define a minimum  $L$ -phase flow or a minimum  $L/V$  ratio which requires an infinite number of stages to give the desired recovery. The methods of determining the minimum  $L/V$  are similar to those presented for minimum  $V/L$ . However, some of the procedures must be modified when  $L$  equals  $V$  and beyond, since in this case  $\Delta$  equals zero and the delta point goes to infinity.

The maximum separation which is theoretically possible in simple countercurrent flow is obtained when an infinite number of stages is used. As the number of stages is increased for given feed rates, the degree of separation of components will increase until a pinch occurs in the cascade. The pinch will result in an infinite number of stages which gives a maximum separation of components. If, for example, the pinch occurs at the last stage at the left end of the cascade (Figure 5.2) the  $V$ -phase product leaving will be in equilibrium with the  $L$ -phase feed. Similarly, if the pinch occurs at the right end, the  $L$ -phase leaving will be in equilibrium with the  $V$ -phase entering. A pinch may also occur at an intermediate point.

## THE SOLVENT-FREE BASIS AND ENTHALPY-CONCENTRATION DIAGRAM

The equations derived in this chapter apply also to calculations on a solvent-free basis and on enthalpy-concentration diagrams. New symbols for extraction are defined and used in the derived equations.

$L'$  = mass of two components ( $a$  and  $b$ ) disregarding the solvent ( $c$ ), in phase  $L$

$V'$  = mass of two components ( $a$  and  $b$ ) disregarding the solvent ( $c$ ), in phase  $V$

$$X = \frac{\text{mass of any component (a, b, or c) per unit}}{\text{mass of } L'}$$

$Y$  = mass of any component ( $a$ ,  $b$ , or  $c$ ) per unit mass of  $V'$

With this notation:

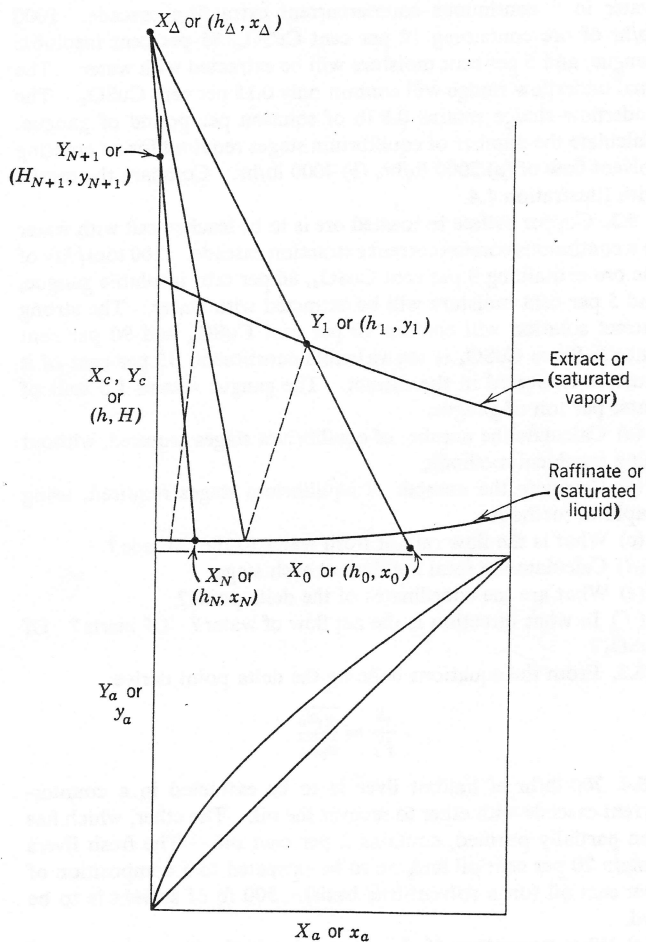
$X_a + X_b = 1$ ,  $X_c$  can have any value.

$Y_a + Y_b = 1$ ,  $Y_c$  can have any value.

Since  $Y_c$  is now the mass of solvent per unit mass of solvent-free material ( $a$  plus  $b$ ), it may have any value between 0 and  $\infty$ . When pure solvent is used,  $Y_{c(N+1)} = \infty$ , and the inverse lever-arm expressions involving

$Y_{e(N+1)}$  are indeterminate. Material balances involving the solvent stream are, for this reason, more easily calculated analytically than graphically. For example, Equations 5.1 and 5.2 may be used to calculate the overall material balance.

Calculation of two-component distillation on an enthalpy-concentration diagram closely resembles calculations on the solvent-free basis. Since only two material components are present in distillation, the material balances of this chapter hold without redefining the symbols. The enthalpy coordinate of the delta point is defined by Equation 5.10. The solvent-free coordinates emphasize the analogy between enthalpy in distillation and solvent in extraction. Enthalpy  $H$  is analogous to the solvent concentration in the extract ( $Y_c$ ) since they are both expressions of a property of the system per unit mass of two components of the system. A typical problem is shown on Figure 5.7. The diagram could apply to a hypothetical case in either extraction or distillation. Therefore, the diagram is labeled both for extraction and for distillation (in parentheses) to show the analogy. The solvent feed is shown to contain a small quantity of the major raffinate component, so



**Figure 5.7.** Analogy between extraction and distillation.

that  $Y_{N+1}$  does not lie at infinity. The vapor feed in distillation which corresponds to this point would be a superheated vapor composed of pure less-volatile component. The raffinate feed is shown to contain a small quantity of solvent ( $X_0$ ). Analogously, the liquid feed in distillation contains a small quantity of enthalpy ( $h_0$ ). Distillation calculations utilizing the enthalpy-composition diagram are usually referred to as the Ponchon-Savarit method. (1, 2).

The "adsorbent-free" diagram given in Figure 3.12 may also be used in calculation in the manner outlined above.

## REFERENCES

1. Ponchon, M., *Tech. moderne* **13**, 20 (1921).
2. Savarit, R., *Arts et métiers*, 65ff. (1922).

## PROBLEMS

5.1. Copper sulfate in a roasted ore is to be leached out with water in a continuous countercurrent extraction cascade. 1000 lb/hr of ore containing 10 per cent  $\text{CuSO}_4$ , 85 per cent insoluble gangue, and 5 per cent moisture will be extracted with water. The final underflow sludge will contain only 0.15 per cent  $\text{CuSO}_4$ . The underflow sludge retains 0.8 lb of solution per pound of gangue. Calculate the number of equilibrium stages required for an entering solvent flow of (a) 2000 lb/hr, (b) 4000 lb/hr. Compare the results with Illustration 4.4.

5.2. Copper sulfate in roasted ore is to be leached out with water in a continuous countercurrent extraction cascade. 100 tons/day of the ore containing 9 per cent  $\text{CuSO}_4$ , 86 per cent insoluble gangue, and 5 per cent moisture will be extracted with water. The strong extract solution will contain 10 per cent  $\text{CuSO}_4$  and 90 per cent water. Since  $\text{CuSO}_4$  is the valuable constituent, 95 per cent of it must be recovered in the extract. The gangue retains 1.5 tons of water per ton of gangue.

(a) Calculate the number of equilibrium stages required, without using graphical methods.

(b) Calculate the number of equilibrium stages required, using graphical methods.

(c) What is the flow rate of fresh water to the cascade?

(d) Calculate the total net flow at each stage.

(e) What are the coordinates of the delta point?

(f) In what direction is the net flow of water? Of inerts? Of  $\text{CuSO}_4$ ?

5.3. From the equations defining the delta point derive

$$\frac{\Delta}{V_1} = \frac{y_1 x_0}{x_0 x_\Delta}$$

5.4. 700 lb/hr of halibut liver is to be extracted in a countercurrent cascade with ether to recover the oil. The ether, which has been partially purified, contains 2 per cent oil. The fresh livers contain 20 per cent oil and are to be extracted to a composition of 1 per cent oil (on a solvent-free basis). 500 lb of solvent is to be used.

(a) What percentage of the oil entering in the livers is recovered in the extract?

(b) How many equilibrium stages are required?

(c) Calculate the mass and direction of the total and component net flows.

(d) Calculate the coordinates of the delta point. Underflow data are given in Figure 3.14.

5.5. It is proposed to separate liquid *a* from its solution with liquid *b* by contacting the solution with solvent *c*. Liquids *b* and *c* are completely immiscible with each other at all concentrations of *a*. Liquid *a* is soluble in *b* and *c* at equilibrium such that the concentration of *a* in liquid *c* is always equal to that of *a* in *b*.

An existing mixer-settler unit is available for the separation. The unit is equivalent to five equilibrium stages. The feed solution is 20 per cent *a* and 80 per cent *b* and must be processed at the rate of 1000 lb/hr. The fresh solvent rate is limited to 1500 lb/hr by the design of the equipment.

(a) Plot the ternary diagram for the system *a-b-c*. Label the extract and raffinate loci and show typical tie lines.

(b) What percentage of the component *a* in the feed can be recovered in the leaving extract, using the maximum permitted solvent flow rate?

(c) What solvent flow rate would give 80 per cent recovery of *a*?

(d) Determine the coordinates of the delta point in parts (b) and

(c). In which direction are the total and component net flows for each case?

5.6. A gaseous mixture of 50 mole percent propane, 50 mole percent propylene is to be separated by passing it countercurrent to silica gel in a rotating-plate column. The gas leaving the top of the unit is to be 95 mole percent propane.

(a) How many equilibrium stages would be required if 350 lb of silica gel are supplied for each pound mole of gas?

(b) What is the minimum mass of silica gel per pound mole of gas which will give a product with 95 mole percent propane?

5.7. A countercurrent extraction cascade is processing an aqueous solution of acetic acid with pure isopropyl ether. The following data are available:

Entering raffinate: 1000 lb/hr  
35 per cent acetic acid, 65 per cent water  
Final Extract: 2500 lb/hr  
12 per cent acetic acid

How many equilibrium stages is the cascade equivalent to?

5.8. The following information is available on a multistage cod-liver oil extraction unit:

Entering Livers: 1000 lb/hr  
32.6 mass percent oil  
67.4 mass percent inerts  
Entering Solvent: 2000 lb/hr  
1 mass percent oil  
99 mass percent ether  
Exit Underflow: 1180 lb/hr  
1.14 mass percent oil  
41.0 mass percent ether  
57.9 mass percent inerts  
Exit Extract: 1820 lb/hr  
18.3 mass percent oil  
81.7 mass percent ether

Calculate the three coordinates of the delta point.

5.9. Isopropyl ether is to be used to extract acetic acid from an aqueous solution. An existing countercurrent extraction column which is equivalent to four theoretical stages is to be used.

(a) Determine the flow rate of pure isopropyl ether solvent required for the following conditions:

Feed flow rate: 1000 lb/hr  
Feed composition: 35 per cent acetic acid,  
65 per cent  $\text{H}_2\text{O}$

Final extract composition: 10 per cent acetic acid

(b) What percentage recovery of acetic acid is achieved?

5.10. Tung meal containing 55 mass percent oil is to be extracted at a rate of 3000 lb/hr, using 12,000 lb/hr of solvent. The solvent contains 98 per cent *n*-hexane and 2 per cent tung oil. The solution adhering to the insoluble meal in the underflow was determined experimentally, as tabulated below:

Composition of solution adhering, Mass Fraction Oil	lb Solution/lb Inerts
0.0	2.0
0.2	2.5
0.4	3.0
0.6	3.5

The tung meal was so finely divided that some of it goes out suspended in the overflow solution. This amounts to 0.05 lb of solids per pound of solution. The underflow must have a concentration no more than 2 mass percent oil.

(a) How many equilibrium stages are required?

(b) What is the percentage recovery of tung oil extracted from the meal?

5.11. Acetic acid is to be extracted from aqueous solution by countercurrent extraction with isopropyl ether. The feed is 10,000 lb/hr of 28 per cent acetic acid.

(a) Determine the number of equilibrium stages required for a solvent feed rate of 30,000 lb/hr and an extract composition of 8 per cent acetic acid.

(b) Determine the minimum solvent feed ratio which will give the same raffinate composition as in part (a).

5.12. A liquid containing 40 per cent styrene and 60 per cent ethylbenzene is to be extracted in a countercurrent cascade by diethylene glycol solvent. The raffinate is to contain 10 per cent styrene on a solvent-free basis.

(a) Determine the minimum solvent to feed ratio.

(b) Using 1.3 times the minimum solvent to feed ratio, calculate the number of equilibrium stages required on ternary coordinates.

(c) Repeat parts (a) and (b) using coordinates plotted on a solvent-free basis.

5.13. A mixture of 50 mole percent ethanol, 50 mole percent water contains a very small quantity of nonvolatile impurity which discolors the solution. It has been proposed to concentrate and purify the ethanol by stripping the ethanol from solution with an available supply of superheated steam. The solution will flow downward in a bubble-cap column countercurrent to the upward-flowing steam. The solution is at 70°F and the steam at 350°F and 1 atm.

(a) What is the maximum ethanol vapor concentration that can be obtained with an infinite number of equilibrium stages?

(b) What ethanol concentration and recovery in the vapor is obtained with a column equivalent to five equilibrium stages with a steam to ethanol feed ratio of 1:2 mole/mole?

5.14. A new solvent extraction unit has just been installed, and it is not giving the desired separation. The unit was designed to extract 10,000 lb/hr of a 30 per cent diphenylhexane–70 per cent docosane mixture with 20,000 lb/hr of solvent. It was desired to

produce an extracted docosane phase with only a 1 per cent concentration of diphenylhexane, but the actual concentration during the test run was much higher. Since a diphenylhexane concentration in the extracted docosane above 1 per cent is unacceptable, you must make a recommendation to the operating engineer to enable him to make the desired separation. Of course, it is necessary to maintain the feed rate of 10,000 lb/hr, and no more than 20,000 lb/hr of solvent can be used, since the unit can handle no more. The solvent used in the test was 98 per cent furfural and 2 per cent diphenylhexane, recycled from the solvent recovery system. The unit has fifteen actual stages and the over-all efficiency is about 30 per cent.

(a) Utilizing the principles and calculations of liquid-liquid extraction, determine why the desired separation was not accomplished in the test run.

(b) Suggest a change in operating conditions which will give the desired separation with no modification in the extraction equipment. Tell how you would calculate to determine that your suggestion is valid before it is tried.

Your answer should include any required calculations and a careful account of the reasoning used in arriving at your conclusions.

SOLUBILITY DATA FOR THE SYSTEM DOCOSANE–DIPHENYLHEXANE–FURFURAL AT 45°C.

Briggs, S. W., and E. W. Comings, *Ind. Eng. Chem.* Vol. 35, No. 4, pp. 411–417 (1943).

Mass Fraction Furfural	Phase Envelope Mass Fraction Diphenylhexane	Mass Fraction Docosane	
0.040	0.0	0.960	
0.050	0.110	0.840	
0.070	0.260	0.670	
0.100	0.375	0.525	
0.200	0.474	0.326	
0.300	0.487	0.213	
0.400	0.468	0.132	
0.500	0.423	0.077	
0.600	0.356	0.044	
0.700	0.274	0.026	
0.800	0.185	0.015	
0.900	0.090	0.010	
0.993	0.0	0.007	
Tie Lines			
{ Furfural phase 0.891	0.098	0.011	} in equilibrium
{ Docosane phase 0.048	0.100	0.852	
{ Furfural phase 0.736	0.242	0.022	} in equilibrium
{ Docosane phase 0.065	0.245	0.690	
{ Furfural phase 0.523	0.409	0.068	} in equilibrium
{ Docosane phase 0.133	0.426	0.439	