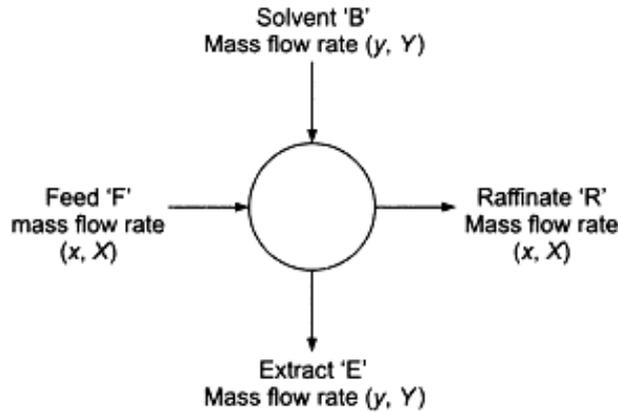# EXTRACTION

10.1 INTRODUCTION

Liquid extraction is the separation of the constituents of a liquid by contact with another insoluble liquid called *solvent*. The constituents get distributed between the two phases. The solvent rich phase is called *extract* and the residual liquid from which the solute has been removed is called *raffinate*. Some of the complicated systems may use two solvents to separate the components of a feed. A mixture of para or ortho–nitro benzoic acids can be separated by distributing them between the insoluble liquids chloroform and water. The chloroform dissolves the para isomer and water the ortho isomer. This is called *dual solvent* or *double solvent* or *fractional extraction*. Some of the components which are difficult to separate by other separation processes like distillation can effectively be separated by extraction or extraction followed by distillation, (e.g.) acetic acid – water separation. Similarly long chain fatty acids can be separated from vegetable oils economically by extraction rather than high vacuum distillation. The separation of fission products from nuclear energy process and separation of low cost metals can be effectively carried out by liquid extraction. Pharmaceutical products like penicillin are also separated by this technique. Mercaptans can be removed by using hydrocarbon oil as solvent. Phenol is extracted from coal tar using alkaline solution as solvent. Caprolactum is extracted with benzene as solvent.

10.2 EQUILIBRIA

In extraction operation generally ternary systems are involved. The solute distributes between solvent rich phase called *extract* and solvent lean phase called *raffinate*. The schematic diagram shown in Fig. 10.1 indicates the various streams involved in a typical liquid–liquid extraction operation. The equilibrium concentration of such systems can be represented in a triangular coordinate system.

x = mass fraction of solute in Feed and Raffinate stream
y = mass fraction of solute in Solvent and Extract stream
X = Mass of solute/mass of solute free components in Feed or Raffinate phase
Y = Mass of solute/mass of solute free components in Extract or Solvent phase

Fig. 10.1 Streams in extraction.

10.2.1 Equilateral–Triangular Coordinates

A mixture having a typical composition of 50% A, 30% B and 20% C is represented by point M as shown in Fig. 10.2. Now let us consider that P kg of a mixture at a point P is added to Q kg of mixture at Q, the resulting mixture is shown by point R on line PQ such that

$$\frac{P}{Q} = \frac{\text{Length of QR}}{\text{Length of PR}} = \frac{x_Q - x_R}{x_R - x_P} \qquad (10.1)$$

The ternary systems usually follow any one of the two categories given below:

(i) one pair partially soluble and two pairs partially soluble
(ii) Insoluble systems.
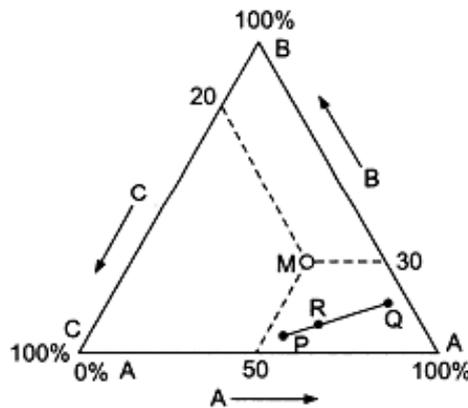
Fig. 10.2 Representation of ternary data in a triangular chart.

In all our subsequent discussions 'C' indicates the distributing solute, 'B' the solvent and 'A' the solute-free component in feed. Some of the common combinations of A, B and C are as follows:

A	B	C
Water	Chloroform	Acetone
Benzene	Water	Acetic acid

The equilibrium composition of mixtures can be represented in a triangular coordinate system. These diagrams drawn at constant temperatures are also called *isotherms*. A typical isotherm is shown in Fig. 10.3 in which 'C' is the solute which dissolves in A and B completely. A and B mutually dissolve to a limited extent. If the solubility of 'A' and 'B' is very minimal, then the points S and T will be very close to apexes A and B respectively. The curve SPQT is the *binodal solubility curve*. Any mixture outside the curve SPQT will be a homogeneous solution of the one liquid phase. Any point within the area bounded by the curve and the axis AB will form two insoluble saturated liquid phases, one rich in A phase and the other rich in B phase.
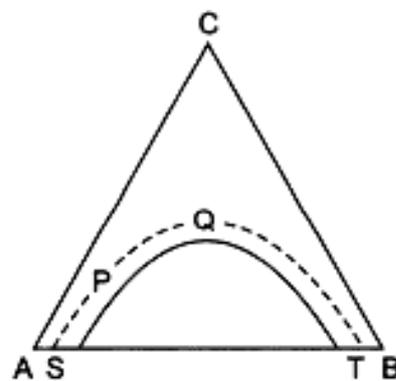

Fig. 10.3 Extraction isotherm.

10.7 FACTORS INFLUENCING CHOICE OF SOLVENT

1. *Selectivity, β:* The effectiveness of solvent B for separating a solution of A and C into its components is measured by comparing the ratio of C to A in the B–rich phase to that in the A–rich phase at equilibrium and is called *selectivity* or *separation factor.* This is also analogous to relative volatility in distillation and it is defined as

$$\beta = \frac{[(\text{weight fraction of C})/(\text{weight fraction of A})]_{\text{Extract}}}{[(\text{weight fraction of C})/(\text{weight fraction of A})]_{\text{Raffinate}}}$$

$$= \left(\frac{y_E^*}{x_R}\right)\left(\frac{\text{weight fraction of A in raffinate}}{\text{weight fraction of A in extract}}\right) \tag{10.2}$$

It is preferable to choose a solvent with selectivity higher than unity. Selectivity also varies with concentration and in some systems it will vary from high values through unity to fractional values. Such systems are analogous to azeotropes.

2. *Distribution coefficient:* It is defined as the concentration of solute in extract(y) to that in raffinate(x). It is preferable to have a higher ratio of y/x as it results in the use of lesser quantity of solvent.

3. *Recoverability of solvent:* The solvent has to be recovered from extract phase for reuse. This is normally done by distillation. Hence, one should ensure that the mixture does not form an azeotrope which has a higher relative volatility and its latent heat of vaporization shall be low so that lesser energy is spent during vaporization.

4. *Density:* A larger difference in densities is necessary both for stagewise and continuous contact operations as it will help in easier separation of phases. However, at plait point the density difference is zero.

5. *Interfacial tension:* If the interfacial tension of solvent is large, more readily the coalescence of droplets or emulsions will occur but the dispersion of one liquid in the other will be difficult. Since coalescence is usually of greater importance in extraction operation, the interfacial tension should therefore be high. It is zero at plait point.

6. *Chemical reactivity:* Solvent should be thermally stable and chemically inert towards the other components of the system and also towards the material of construction.

7. *Other properties:* Viscosity, vapour pressure and freezing point should be low for ease in handling and storage. They should also be non-toxic, non-flammable and of low cost.

10.8 OPERATIONS

Extraction operations can be carried out either as a single stage or as a multistage operation. Again the multistage operation could be either a cross-current or a counter-current operation. The leaving streams, viz. the extract and raffinate from each stage is always in equilibrium. A combination of mixer-settler is said to constitute a stage and in a multistage operation they are arranged in cascades.

10.8.1 Single Stage Operation

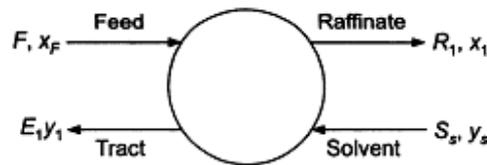A typical flow diagram of a single stage extraction operation is shown in Fig. 10.8.

Fig. 10.8 Streams in a single stage operation.

F, R_1, E_1, and S are either the flow rates or quantities of different streams such as feed, raffinate, extract and solvent respectively and x_F, x_1, y_1, y_S are all weight fractions of solute in their respective streams.

The material balance gives

$$F + S = M_1 = E_1 + R_1 \qquad (10.3)$$

where M_1 is the total weight of mixture (Feed + solvent or extract + raffinate)

A solute balance yields

$$Fx_F + Sy_S = M_1x_{M_1} = E_1y_1 + R_1x_1 \qquad (10.4)$$

where x_{M_1} is the effective solute concentration in the extractor.

Eliminating M_1 from Eqs. (10.3) and (10.4), we get

$$\frac{S}{F} = \frac{x_F - x_{M_1}}{x_{M_1} - y_S} \qquad (10.5)$$

The quantities of extract and raffinate can be computed from mixture rule given by Eq. (10.1) or by material balance given in Eq. (10.4)

$$E_1y_1 + R_1x_1 = M_1x_{M_1} \qquad (10.6)$$

$$E_1y_1 + (M_1 - E_1)x_1 = M_1x_{M_1} \qquad (10.7)$$

$$E_1 = M_1 \left(\frac{x_{M_1} - x_1}{y_1 - x_1} \right) \qquad (10.8)$$

10.8.2 Multistage Cross-current Operation

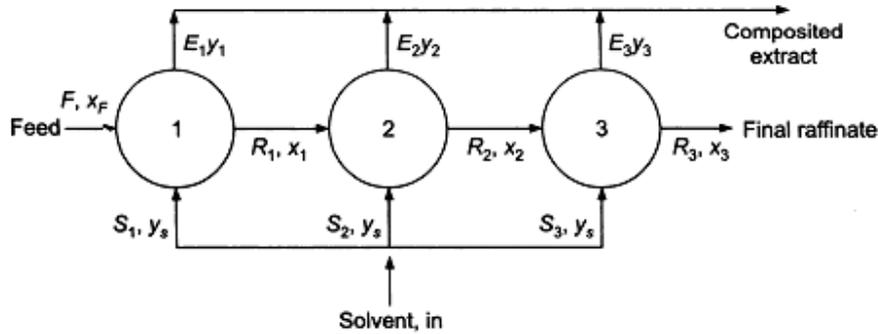A typical flow diagram of a multistage cross-current operation is shown in Fig. 10.10

Fig. 10.10 A three stage cross-current extraction operation.

Consider a three-stage cross-current extraction process as shown in Fig. 10.10. The feed enters the first stage and the raffinate successively passes from stage (1) to (2) and (2) to (3) and finally leaves the system. Fresh or recovered solvent enters each stage. The solvent used could be of different concentrations but generally it will have the same value as it enters either fresh or after recovery from extract. The values of M_i, x_{Mi}, x_i and y_i, where i stands for the ith stage, can be computed as indicated in the single stage operation using material balances and tie lines. From these values the quantities of extract and raffinate from each stage can be computed.

Material balance across stage (1) gives

$$F + S_1 = R_1 + E_1 = M_1 \quad \text{(say)} \tag{10.9}$$

Component balance gives

$$Fx_F + S_1y_s = R_1x_1 + E_1y_1 = M_1x_{M1} \tag{10.10}$$

$$\therefore \qquad x_{M_1} = \frac{(Fx_F + S_1y_s)}{(F + S_1)} \tag{10.11}$$

Similarly for any stage i

$$x_{M_i} = \frac{(R_{i-1}x_{i-1} + S_iy_s)}{(R_{i-1} + S_i)} \tag{10.12}$$

10.8.3 Multistage Countercurrent Extraction

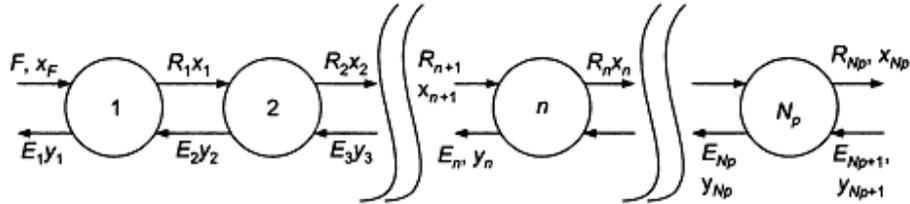A typical flow diagram of a multistage countercurrent operation is shown in Fig. 10.12.

Fig. 10.12 Multistage countercurrent extraction operation.

Material balance for the system gives

$$F + E_{Np+1} = E_1 + R_{Np} \tag{10.13}$$

i.e.

$$F - E_1 = R_{Np} - E_{Np+1} \tag{10.14}$$

A component balance gives,

$$Fx_F + E_{Np+1}y_{Np+1} = E_1y_1 + R_{Np} \cdot x_{Np} \tag{10.15}$$

i.e.

$$Fx_F - E_1y_1 = R_{Np} \cdot x_{Np} - E_{Np+1} \cdot y_{Np+1} \tag{10.16}$$

A material balance from 1 to n stages gives

$$F + E_{n+1} = E_1 + R_n \tag{10.17}$$

$$F - E_1 = R_n - E_{n+1} \tag{10.18}$$

Hence, from Eqs. (10.14) and (10.18) , we get

$$F - E_1 = R_{Np} - E_{Np+1} = R_n - E_{n+1} \tag{10.19}$$

By substituting for n as 1, 2, 3, ... we can show that

$$F - E_1 = R_1 - E_2 = R_2 - E_3 = \Delta_R$$

Here Δ_R, which is defined as a difference point, is the net flow outward not only at the last stage but also between any two adjacent stages and it remains constant. In other words, any line joining FE_1, R_1E_2, R_2E_3, ... and extended must pass through the point Δ_R as shown in Fig. 10.13.

10.9 INSOLUBLE SYSTEMS (IMMISCIBLE SYSTEMS)

10.9.1 Cross-current Operation

In insoluble systems, the solvent (B) and the non-solute component in feed solution (A) are insoluble and remain so at all solute concentrations. Since A and B are insoluble, the amount of A and B both in their feed streams and the leaving streams remain constant. If X is the solute concentration in feed stream or raffinate stream expressed in mass ratio (kg of C/kg of A) and Y is the solute concentration in solvent or extract stream expressed in mass ratio (kg of C / kg of B), then a mass balance around stage n with reference to Fig. 10.15 yields
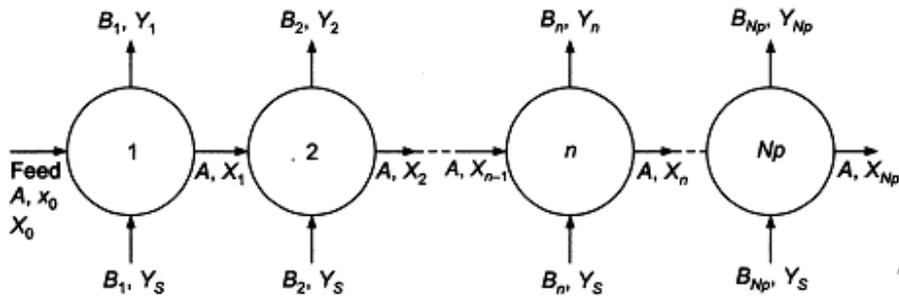

Fig. 10.15 Multistage cross-current operation for an insoluble system.

$$A \cdot X_{n-1} + B_n \cdot Y_S = B_n \cdot Y_n + A \cdot X_n \qquad (10.20)$$

$$A [X_{n-1} - X_n] = B_n [Y_n - Y_S] \qquad (10.21)$$

$$-\frac{A}{B_n} = \frac{(Y_n - Y_s)}{(X_n - X_{n-1})} \qquad (10.22)$$

where A is the non-solute component in feed and B_n is the quantity of pure solvent used in nth stage, $-A/B_n$ is the slope of the operating line for stage n. For a typical three stage cross-current operation the construction of operating lines and the determination of final concentration of raffinate is shown in Fig. 10.16.
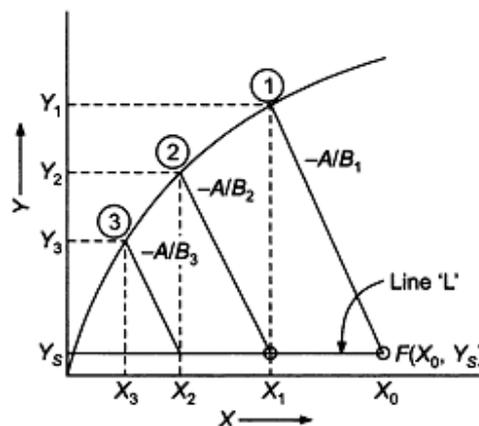

Fig. 10.16 Determination of number of stages in a cross-current operation.

10.9.1.1 Steps involved (Fig. 10.16)

1. Draw the equilibrium curve (X vs Y) on mass ratio basis.
2. Locate F (X_0, Y_s) and also draw a horizontal line L at $Y = Y_S$.
3. Draw a line with the slope ($-A/B_1$) and allow it to intersect the curve at ①.
4. Draw a vertical line from ① to the horizontal line L and the point of intersection corresponds to (X_1, Y_S).
5. From (X_1, Y_S) draw a line with a slope of ($-A/B_2$) to intersect curve at ②.
6. The vertical line drawn from ② to the horizontal line L gives the coordinates (X_2, Y_S).
7. Similarly proceed till X_{Np} is crossed and determine the number of stages needed or for the given number of stages, determine the X_{Np} value and hence the percentage extraction.

10.9.2 Countercurrent Operation

The flow of various streams in a countercurrent immiscible system with their compositions in a multistage operation is shown in Fig. 10.17.
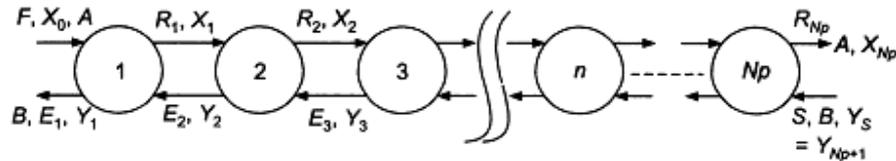

Fig. 10.17 Multistage countercurrent extraction operation for an insoluble system.

The material balance based on solute is given below:

$$A \cdot X_0 + B \cdot Y_{Np+1} = BY_1 + A \cdot X_{Np} \tag{10.23}$$

$$A [X_0 - X_{Np}] = B [Y_1 - Y_{Np+1}] \tag{10.24}$$

i.e.

$$\frac{A}{B} = \frac{(Y_1 - Y_{Np+1})}{(X_0 - X_{Np})} \tag{10.25}$$

i.e.

$$\frac{A}{B} = \frac{(Y_{out} - Y_{in})}{(X_{in} - X_{out})} \tag{10.26}$$

The operating line will have a slope of A/B and also pass through the points (X_0, Y_1) and (X_{Np}, Y_{Np+1}).

Once the operating line is constructed, the number of stages needed either for a specified percentage recovery or the exit concentration of raffinate stream can be found.

Sometimes the percentage recovery and the number of stages will be specified. The objective will be to fix the amount of solvent needed for the operation. This can be done by fixing the operating line by trial and error,

which will exactly yield both the exit concentration of raffinate and the specified number of stages.

Minimum solvent requirement is estimated by drawing either a tangent to the equilibrium curve or based on the equilibrium solute concentration in the solvent rich layer for the exit concentration of raffinate. The slope of the tangent gives the slope of operating line under minimum solvent conditions. In the later case, it is estimated by the slope of the line joining the terminal conditions.

When the equilibrium curve is of constant slope, say m', then $m' = (Y^*/X)$. The number of stages N_p can be estimated by

$$\frac{(X_F - X_{Np})}{X_F - \dfrac{(Y_{Np+1})}{m'}} = \frac{\left(\dfrac{m'B}{A}\right)^{Np+1} - \left(\dfrac{m'B}{A}\right)}{\left(\dfrac{m'B}{A}\right)^{Np+1} - 1} \qquad (10.27)$$

where, $\dfrac{m'B}{A}$ is called the *extraction factor*.

10.9.2.1 Steps involved (Fig. 10.18)

1. Draw the equilibrium curve.
2. Locate X_0, X_{Np} and Y_{Np+1}.
3. From the point (X_{Np}, Y_{Np+1}) draw a tangent to the equilibrium curve which will give slope of the operating line at minimum solvent condition, $(A/B)_{min}$.
4. If B_{actual} in terms of B_{min} is known, then we can determine $(A/B)_{actual}$ and draw the actual operating line. Otherwise, if the quantity of B is given, draw the operating line directly.
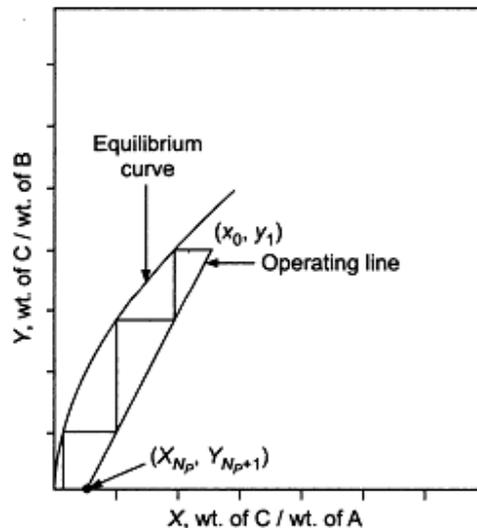

Fig. 10.18 Stages for countercurrent extraction.

5. At X_0 from the operating line draw a horizontal line to equilibrium curve which will give Y_1, the concentration of solute in final extract.

6. By stepwise construction from (X_0, Y_1), determine the number of stages needed to cross X_{Np}.

7. However, if the number of stages are prescribed, X_{Np} will have to be fixed by trial and error and checked for the prescribed number of stages.

8. In case the amount of solvent used is not given and X_{Np} along with the stages are known then the operating line has to be fixed by trial and error to ensure that both the prescribed X_{Np} and the number of stages are reached. From the slope of the operating line so fixed, we can estimate the solvent needed for the operation.

WORKED EXAMPLES

1. A 5% (by weight) solution of acetaldehyde in toluene is extracted with water in a three stage cross-current unit. If 100 kg of water is used per stage for 500 kg of feed, calculate (using graphical method) the percentage extraction of acetaldehyde and the weights of final raffinate and mixed extract. The equilibrium relationship is given by the equation, $Y = 2.3 \ X$ where Y = kg acetaldehyde/kg water and X = kg acetaldehyde/kg toluene. Assume that toluene and water are immiscible with each other.

Solution.

A : toluene, B : water, C : acetaldehyde,

$F = 500$ kg, $x_F = 0.05$, $Y = 2.3X$

$B = 100$ kg water/stage

Three stage cross-current operation

Assume solvent to be pure, i.e. $y_s = 0$

$$F = 500 \text{ kg}, \ A = 475 \text{ kg, and } C = 25 \text{ kg}$$

Slope = $(- A/B)$

So $(- A/B)$ for each stage = $(- 475/100) = (- 4.75)$

Draw the operating line with a slope of $- 4.75$ for each stage

$$X_F = \frac{x_F}{(1 - x_F)} = \frac{0.05}{1 - 0.95} = 0.0526$$

X (kg acetaldehyde/ kg toluene)	0	0.01	0.02	0.03	0.04	0.05	0.06
Y (kg acetaldehyde/ kg water)	0	0.023	0.046	0.069	0.092	0.115	0.138

Since system is immiscible, the whole of solvent goes in extract. The feed introduced in 1st stage just passes through all stages and comes out as final raffinate:

A plot between X and Y is drawn. The operating line is drawn with a slope of $- 4.75$ for each of the three stages.

Weight of A in final raffinate $= A = 475$ kg

Final raffinate contains $X_3 = 0.0161$ kg C/kg A (from graph)

Amount of C in raffinate $= 475 \times 0.016 = 7.6$ kg

Total weight of raffinate $= 475 + 7.6 = 482.6$ kg

Total C extracted $= (Y_1 + Y_2 + Y_3) \times 100$

$= 100 \times (0.082 + 0.055 + 0.037) = 17.4$ kg

In extract, the amount of B $= 100$ kg (in each stage)

$Y_3 = 0.037$ kg C/kg B (from graph)

Amount of C in final stage extract $= 0.037 \times 100 = 3.7$ kg

Total weight of extract $= 300 + 17.4 = 317.4$ kg

% Extraction $= (17.4/25) \times 100 = 69.6\%$

10.15.7 Other Extractors

Apart from these we have conventional packed towers, spray towers and for lesser density difference systems the centrifugal extractors.

WORKED EXAMPLES

1. A 5% (by weight) solution of acetaldehyde in toluene is extracted with water in a three stage cross-current unit. If 100 kg of water is used per stage for 500 kg of feed, calculate (using graphical method) the percentage extraction of acetaldehyde and the weights of final raffinate and mixed extract. The equilibrium relationship is given by the equation, $Y = 2.3\ X$ where $Y =$ kg acetaldehyde/kg water and $X =$ kg acetaldehyde/kg toluene. Assume that toluene and water are immiscible with each other.

Solution.

A : toluene, $\qquad$ B : water, $\qquad$ C : acetaldehyde,

$F = 500$ kg, $\qquad$ $x_F = 0.05$, $\qquad$ $Y = 2.3X$

$B = 100$ kg water/stage

Three stage cross-current operation

Assume solvent to be pure, i.e. $y_s = 0$

$$F = 500 \text{ kg}, \quad A = 475 \text{ kg, and } C = 25 \text{ kg}$$

$$\text{Slope} = (-A/B)$$

So $(-A/B)$ for each stage $= (-475/100) = (-4.75)$

Draw the operating line with a slope of -4.75 for each stage

$$X_F = \frac{x_F}{(1 - x_F)} = \frac{0.05}{1 - 0.95} = 0.0526$$

X (kg acetaldehyde/ kg toluene)	0	0.01	0.02	0.03	0.04	0.05	0.06
Y (kg acetaldehyde/ kg water)	0	0.023	0.046	0.069	0.092	0.115	0.138

Since system is immiscible, the whole of solvent goes in extract. The feed introduced in 1st stage just passes through all stages and comes out as final raffinate:

A plot between X and Y is drawn. The operating line is drawn with a slope of -4.75 for each of the three stages.

Weight of A in final raffinate $= A = 475$ kg

Final raffinate contains $\quad X_3 = 0.0161$ kg C/kg A (from graph)

Amount of C in raffinate $\quad = 475 \times 0.016 = 7.6$ kg

Total weight of raffinate $\quad = 475 + 7.6 = 482.6$ kg

Total C extracted $\quad = (Y_1 + Y_2 + Y_3) \times 100$

$\quad = 100 \times (0.082 + 0.055 + 0.037) = 17.4$ kg

In extract, the amount of B $\quad = 100$ kg (in each stage)

$$Y_3 = 0.037 \text{ kg } C/\text{kg } B \text{ (from graph)}$$

Amount of C in final stage extract $= 0.037 \times 100 = 3.7$ kg

Total weight of extract $\quad = 300 + 17.4 = 317.4$ kg

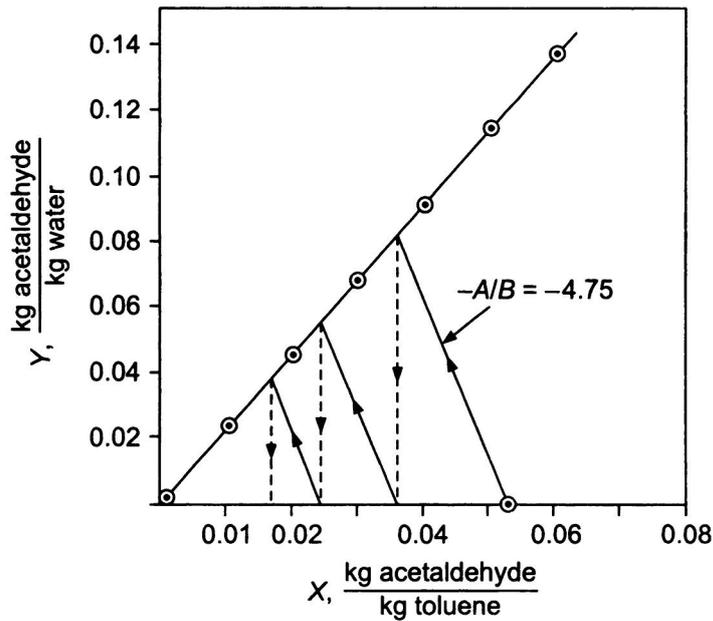% Extraction $\quad = (17.4/25) \times 100 = 69.6\%$

Fig. 10.25 Example 1.

2. 100 kg of a solution containing acetic acid and water containing 25% acid by weight is to be extracted with isopropyl ether at 20°C. The total solvent used for extraction is 100 kg. Determine the compositions and quantities of various streams if,

   (i) The extraction is carried out in single stage

  (ii) The extraction is carried out in two stages with 50 kg of solvent in each stage.

Equilibrium data:

Water layer (wt. %)		Ether layer (wt. %)	
Acid (x)	Water (A)	Acid (y)	Water (A)
0.69	98.1	0.18	0.5
1.41	97.1	0.37	0.7
2.9	95.5	0.79	0.8
6.42	91.7	1.93	1.0
13.3	84.4	4.82	1.9
25.5	71.1	11.4	3.9
36.7	58.9	21.6	6.9
44.3	45.1	31.1	10.8
46.4	37.1	36.2	15.1

Solution.

$A \rightarrow$ water, $B \rightarrow$ isopropyl ether, $C \rightarrow$ Acetic acid,

$F = 100$ kg, $A = 75$ kg,   and $C = 25$ kg, $x_F = 0.25$

Total solvent used $= 100$ kg $= B$

B	0.0121	0.0149	0.016	0.0188	0.023	0.034	0.044	0.106	0.165
x	0.0069	0.0141	0.029	0.0642	0.133	0.255	0.367	0.443	0.464
B	0.9932	0.9893	0.9841	0.9707	0.9328	0.847	0.715	0.581	0.487
y	0.0018	0.0037	0.0079	0.0193	0.0482	0.114	0.216	0.311	0.362

(i) Single stage operation:

By total and component material balances,

$$F + S = M_1$$

$$100 + 100 = M_1 = 200 \text{ kg}$$

$$x_{M_1} = \frac{Fx_y + Sy_s}{F + S} = \frac{100 \times 0.25 + 100 \times 0}{100 + 100} = 0.125$$

Locate M_1 on the FS line corresponding to x_{M_1}. By trial and error, a tie line is drawn which passes through M_1.

The co-ordinates (x_1, y_1) obtained are (0.18, 0.075).

By material balance,

$$R_1 x_1 + y_1 E_1 = M_1 x_{M_1}$$

$$R_1 + E_1 = M_1$$

$$R_1 \times 0.18 + 0.075 E_1 = 200 \times 0.125$$
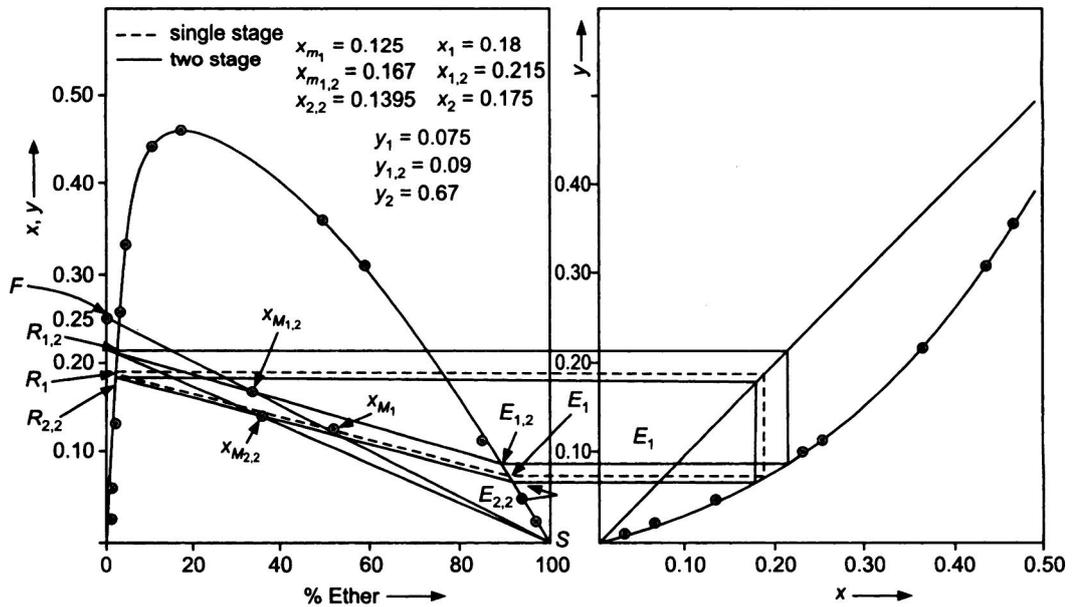
$$R_1 + E_1 = 200$$

Fig. 10.26   Example 2.

Solving we get, $E_1 = M_1 \left(\dfrac{x_{M_1} - x_1}{y_1 - x_1} \right)$

Quantities of product streams are

$$E_1 = 104.76 \text{ kg}$$

$$R_1 = 95.24 \text{ kg}$$

(ii) Two-stage operation:

$$F = 100 \text{ kg}, \quad S = 50 \text{ kg}$$

$$S + F = M_1$$

$$x_{M_{1,2}} = \frac{Fx_F + Sy_s}{F + S} = \frac{100 \times 0.25 + 50 \times 0}{100 + 50} = 0.167$$

$$M_1 = 50 + 100 = 150 \text{ kg}$$

Locate $M_{1,2}$ on the Fs line corresponding to $x_{M_{1,2}}$. By trial and error, a tie line is drawn which passes through M_{12}.

The co-ordinates (x_{12}, y_{22}) obtained are (0.215, 0.09)

By following the same procedure mentioned above and solving, we get

$$E_{12} = M_{12}\left(\frac{x_{M_{12}} - x_{12}}{y_{12} - x_{12}}\right) = 150\left(\frac{0.167 - 0.215}{0.09 - 0.215}\right) = 57.6 \text{ kg}$$

$$R_{12} = 150 - 57.6 = 92.4 \text{ kg}$$

Similarly for II stage, $x_{M22} = 0.1395$, $M_2 = 92.4 + 50 = 142.4$ kg

$$x_2 = 0.175 \text{ and } y_2 = 0.07 \text{ (from tie line)}$$

$$E_2 = 48.14 \text{ kg}$$

$$R_2 = 94.26 \text{ kg}$$

Percentage recovery $= \dfrac{(25 - 94.26 \times 0.175)}{25} \times 100 = 34.02\%$

3. 1000 kg/hr of an acetone-water mixture containing 20% by weight of acetone is to be counter-currently extracted with trichloroethane. The recovered solvent to be used is free from acetone. The water and trichloroethane are insoluble. If 90% recovery of acetone is desired estimate the number of stages required if 1.5 times the minimum solvent is used. The equilibrium relationship is given by $y = 1.65x$, where x and y are weight fractions of acetone in water and trichloroethane respectively.

Solution.

$$X_F = 0.2/(1 - 0.2) = 0.25$$

$$X_{NP} = 0.25 \times 0.1 = 0.025$$

$$y_1 = 1.65 \times 0.2 = 0.33$$

$$Y_1 = 0.33/0.67 = 0.49$$

$$Y_s = 0 \text{ (Pure solvent)}$$

(the same value is got from plotting of the graph also)

X	0.05	0.1	0.175	0.25	0.325
$x = \dfrac{x}{1+x}$	0.0476	0.0909	0.149	0.20	0.245
$y = 1.65x$	0.0785	0.15	0.246	0.33	0.404
$Y = \dfrac{y}{1-y}$	0.085	0.176	0.326	0.493	0.678

$$\frac{A}{B_{min}} = \frac{Y_1 - Y_s}{X_F - X_{Np}}$$

$$\frac{800}{B_{min}} = \frac{0.49 - 0}{0.25 - 0.025}$$

$$B_{min} = 367.35 \ kg$$

$$B_{act} = 1.5 \times B_{min} = 1.5 \times 367.35 = 551.025 \ kg$$

$$\frac{A}{B_{act}} = \frac{Y_{1,act} - Y_s}{X_F - X_{Np}}$$

$$\frac{800}{551.025} = \frac{Y_{1,act} - 0}{0.25 - 0.025} = 1.452$$

$$Y_{1,act} = 0.327$$

An operating line with a slope of 1.452 is drawn and by stepwise construction the number of stages is determined as 5.
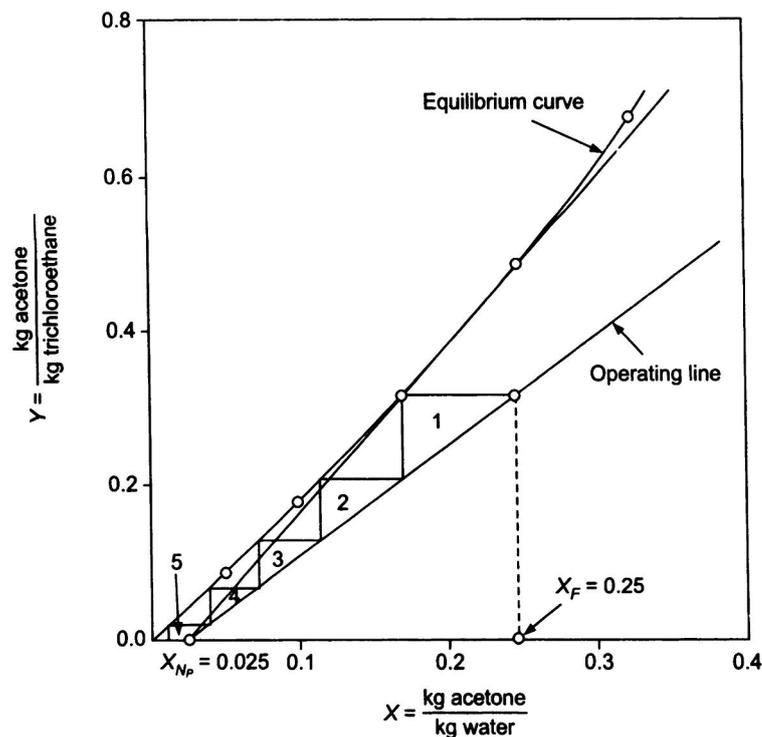

Fig. 10.27   Example 3.

4. Water–dioxane solution is to be separated by extraction process using benzene as solvent. At 25°C the equilibrium distribution of dioxane between water and benzene is as follows:

Weight % of dioxane in water	5.1	18.9	25.2
Weight % of dioxane in benzene	5.2	22.5	32.0

At these concentrations water and benzene are substantially insoluble. 1000 kg of a 25% dioxane water solution is to be extracted to remove 95% of dioxane. The benzene is dioxane free.

(i) Calculate the benzene requirement for a single batch operation.

(ii) Calculate the benzene requirement for a five-stage cross-current operation with 600 kg of solvent used in each stage.

Solution.

Solvent = amount of feed or raffinate in each stage

(B)   =   (F)   or   (R)

x	0.051	0.189	0.252
y	0.052	0.225	0.32
$X = x/(1 - x)$	0.054	0.233	0.337
$Y = y/(1 - y)$	0.05485	0.29	0.471

(i) $F = 1000$ kg ($A = 750$ kg, $C = 250$ kg)

$$x_F = 0.25, \quad X_F = 0.25/0.75 = 0.333$$
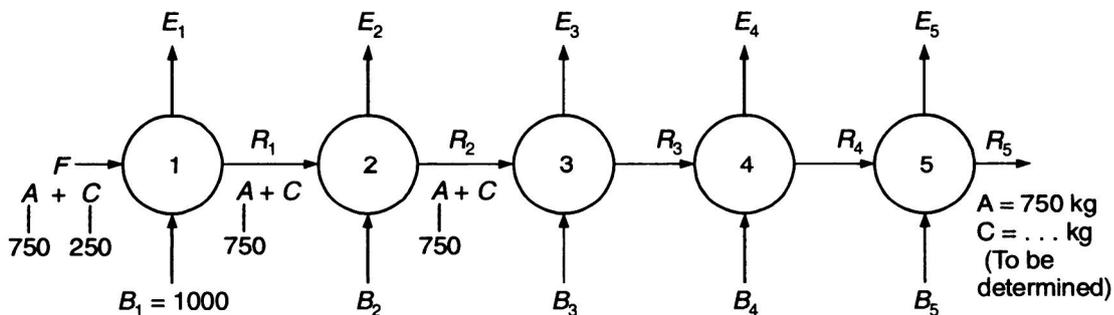
$$X_{R_{Np}} = 0.05 \times 0.333 = 0.01665$$

$$Y_{in} = 0$$

$$Y_1 = 0.0175 \text{ (From graph)}$$

$$\frac{A}{B} = \frac{Y_1 - Y_s}{X_F - X_{Np}}$$

$$\frac{750}{B} = \frac{0.0175 - 0}{0.333 - 0.01665}$$

$$B = 13557.86 \text{ kg}$$

(ii) Five-stage cross-current operation

Amount of solvent used is 600 kg

$$\frac{A}{B} = \frac{750}{600} = 1.25$$

Draw operating lines with a slope of -1.25 and determine the raffinate concentration.

$$X_{final} = 0.0175$$

$$\% \text{ recovery} = \frac{(0.333 - 0.0175) \times 100}{0.333} = 94.75\%$$
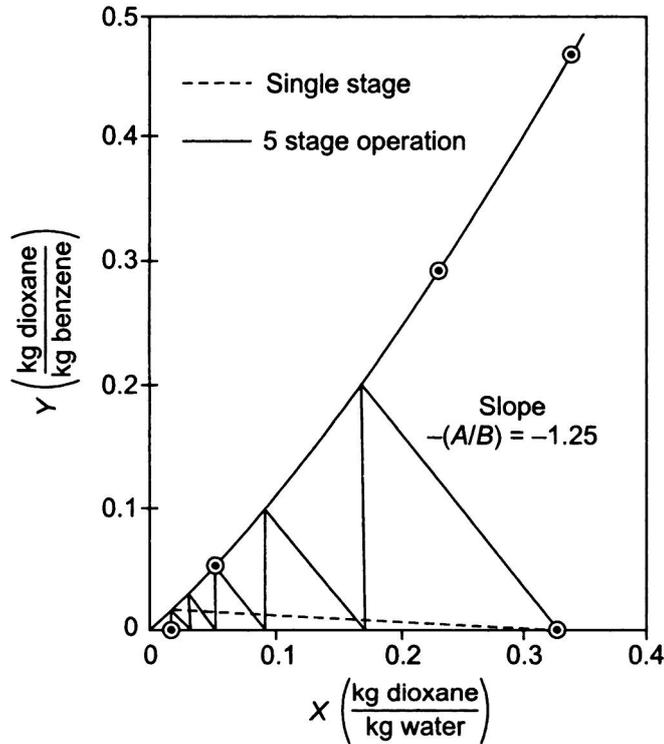

Fig. 10.28   Example 4.

5. 1000 kg per hour of a solution of C in A containing 20% C by weight is to be countercurrently extracted with 400 kg per hour of solvent B. The components A and B are insoluble. The equilibrium distribution of component C between A and B are as follows;

Wt. of C/Wt. of A	0.05	0.20	0.30	0.45	0.50	0.54
Wt. of C/Wt. of B	0.25	0.40	0.50	0.65	0.70	0.74

How many theoretical stages will be required to reduce the concentration of C to 5% in effluent?

Solution.

$F = 1000$ kg/h, $(A = 800$ kg/h, $C = 200$ kg/h)

$x_F = 0.2$, $x_{R_{Np}} = 0.05$

Assume solvent to be pure, then

countercurrent extraction $y_s = Y_s = 0$

solvent $= B = 400$ kg/h

A and *B* are insoluble

$$X_F = 0.2/(1 - 0.2) = 0.25, \qquad X_{R_{Np}} = 0.05/(1 - 0.05) = 0.0526$$

$$\frac{A}{B} = \frac{Y_1 - Y_s}{X_F - X_{Np}}$$

$$\text{Slope} = \frac{A}{B} = \frac{800}{400} = 2$$

$$\frac{A}{B} = \frac{Y_1 - 0}{0.25 - 0.0526}$$

$$Y_1 = 0.395$$

Plot *X* vs *Y* to obtain the equilibrium curve.

Draw an operating line between $(X_{R_{Np}}, Y_s)$ and (X_F, Y_1) and determine the number of stages by stepwise construction.
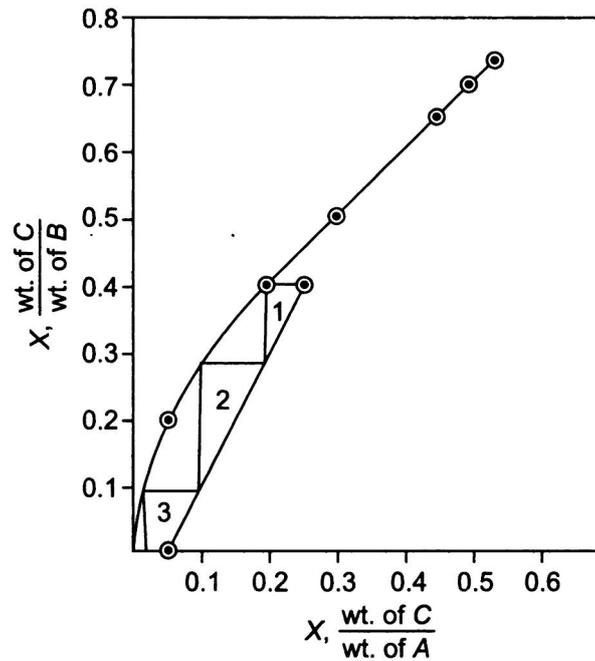
Number of stages obtained = 3.

Fig. 10.29 Example 5.

6. Water–dioxane solution is to be separated by extraction process using benzene as solvent. At 25°C the equilibrium distribution of dioxane between water and benzene is as follows:

wt. % of dioxane in water	5.1	18.9	25.2
wt. % of dioxane in benzene	5.2	22.5	32.0

At these concentrations water and benzene are substantially insoluble. 1000 kg of a 25% dioxane water solution is to be extracted to remove 95% of dioxane. The benzene is dioxane free. Calculate minimum solvent

required in kg/h if the extraction is done in countercurrent fashion. Estimate the number of stages needed if 1.5 times the minimum amount of solvent is used.

Solution.

Benzene: B Water: A Dioxane: C

$$F = 1000 \text{ kg } (A = 750 \text{ kg}, \ C = 250 \text{ kg})$$

x	0.051	0.189	0.252
y	0.052	0.225	0.32
$X = x/(1 - x)$	0.054	0.233	0.337
$Y = y/(1 - y)$	0.05485	0.29	0.471

$$x_F = 0.25,$$

$$X_F = 0.25/0.75 = 0.333$$

$$X_{R_{Np}} = 0.05 \times 0.333 = 0.01665$$

$$\frac{A}{B_{\min}} = \frac{Y_{Np+1} - Y_1}{X_{Np} - X_F}$$

$$\frac{A}{B_{\min}} = \frac{Y_{Np+1} - Y_1}{X_{Np} - X_F} = \frac{0 - 0.365}{0.01665 - 0.333} = 1.1154$$

$$B_{\min} = 650 \text{ kg}$$

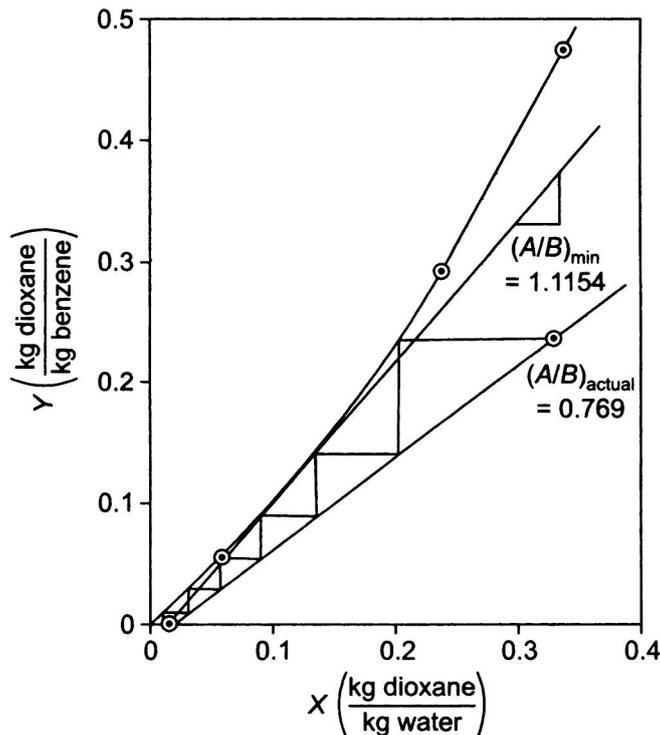$$B_{\text{act}} = 1.5 \times 650 = 975 \text{ kg}$$

Fig. 10.30 Example 6.

$$\frac{A}{B_{act}} = \frac{Y_{Np+1} - Y_{1,act}}{X_{Np} - X_F}$$

$$\frac{750}{975} = \frac{0 - Y_{1,act}}{0.01665 - 0.333}$$

$$Y_{1,act} = 0.243$$

By stepwise construction, the number of stages can be determined as 6.

7. Nicotine in a water solution containing 1% nicotine is to be extracted once with kerosene at 20°C. Kerosene and water are insoluble.

Determine the percentage extraction if 1000 kg of feed solution is extracted once with 1500 kg solvent. What will be the extraction if three ideal stages are used with 500 kg solvent in each stage?

Equilibrium data:

X	0	0.00101	0.00246	0.00502	0.00751	0.00998	0.0204
Y	0	0.00081	0.001962	0.00456	0.00686	0.00913	0.0187

where X is kg Nicotine/kg water and Y is kg Nicotine/kg kerosene.

Solution.

Water: A,   Kerosene: B,   Nicotine: C

$$x_F = 0.01 \qquad X_F = X_0 = \frac{0.01}{(1 - 0.01)} = 0.0101$$

$F = 1000$ kg,   ($C = 10$ kg, $A = 990$ kg), $B = 1500$ kg

$$-\frac{A}{B_n} = \frac{(Y_n - Y_s)}{(X_n - X_{n-1})} \quad \text{when } n = 1, \quad -\frac{A}{B} = \frac{Y_1 - Y_s}{X_1 - X_0}$$

$$\frac{990}{1500} = \frac{Y_1 - 0}{0.0101 - X_1}$$

A line with a slope of -0.66 is drawn from $(0.0101, 0)$ to obtain X_1 and Y_1.

$$Y_1 = 0.66 \, [(0.0101) - X_1]$$

$$Y_1 = 0.0037 \text{ (From graph)}$$

$$X_1 = 0.0045$$

Amount of nicotine in extract $= 0.0037 \times 1500 = 5.55$ kg

$$\% \text{ extraction} = (5.55/10) \times 100 = 55.5\%$$

For three stages

$$(-A/B) = A \, 990/500 = -1.98.$$

3 lines with a slope of -1.98 each are drawn starting from $(0.0101,0)$

$$X_3 = 0.0035, \qquad Y_3 = 0.003$$

Amount of nicotine in final extract $= 0.003 \times 500 = 1.5$ kg

Total C extracted $= (Y_1 + Y_2 + Y_3) \times 500$

$$= (0.0061 + 0.0037 + 0.003) \times 500 = 6.4 \text{ kg}$$
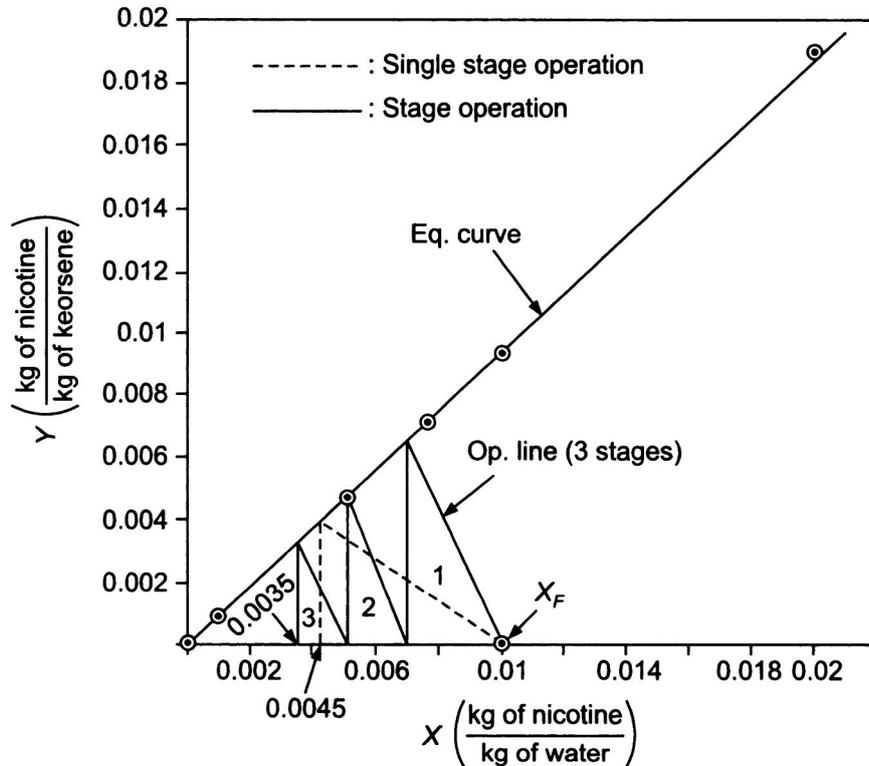
% extraction $= (6.4/10) \times 100 = 64\%$

Fig. 10.31   Example 7.

8. 1000 kg/h of a water–dioxane solution containing 20% dioxane is to be continuously and counter-currently extracted with benzene at 25°C to recover 80% dioxane. Water and benzene are essentially insoluble and the equilibrium distribution of dioxane between them are as follows:

Dioxane in water wt. %	5.1	18.9	25.2
Dioxane in benzene wt. %	5.2	22.5	32.0

Determine the number of theoretical stages if the solvent rate is 1.5 times the minimum.

Solution.

Water: A Dioxane: C Benzene: B

x	0.051	0.189	0.252
y	0.052	0.225	0.32
$X = x/(1 - x)$	0.054	0.233	0.337
$Y = y/(1 - y)$	0.05485	0.29	0.471

$$F = 1000 \text{ kg/h}$$

$$x_F = 0.2, \qquad X_F = X_0 = 0.2/0.8 = 0.25$$

Countercurrent extraction

$$X_{Np} = 0.2 \times 0.25 = 0.05$$

$$\frac{A}{B_{min}} = \frac{Y_{Np+1} - Y_1}{X_{Np} - X_F}$$

$$\frac{800}{B_{min}} = \frac{0 - 0.3075}{0.05 - 0.25} \quad \text{(From Graph)}$$

$$B_{min} = 520.33 \text{ kg}$$

$$B_{act} = 1.5 B_{min} = 1.5 \times 520.33 = 780.5 \text{ kg}$$

$$\frac{A}{B_{act}} = \frac{800}{780.5} = 1.025$$

Draw the operating line with a slope of 1.025 from (0.05,0) and by stepwise construction determine the number of stages.
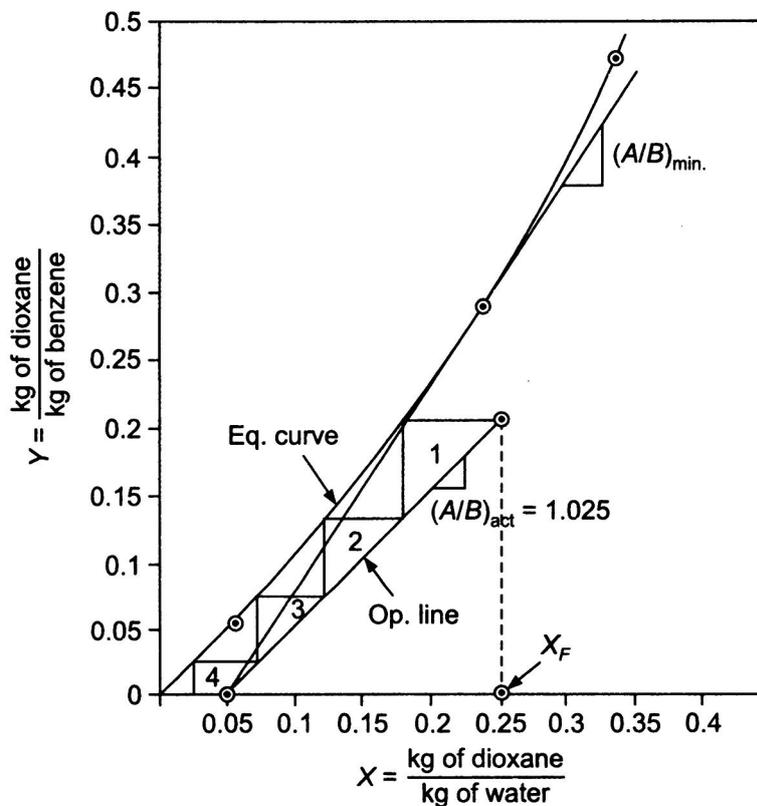
No. of stages = 4

Fig. 10.32 Example 8.