Multicomponent distillation

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Introduction

The problem of determining the stage and reflux requirements for multicomponent distillations is much more complex than for binary mixtures.

With a multicomponent mixture, fixing one component composition does not uniquely determine the other component compositions and the stage temperature.

Also when the feed contains more than two components it is not possible to specify the complete composition of the top and bottom products independently.

The separation between the top and bottom products is usually specified by setting limits on two "**key components**", between which it is desired to make the separation.

Calculation procedure

The normal procedure for a typical problem is to solve the **MESH** (Material balance, Equilibrium, Summation and Heat) balance equations stage-by-stage, from the top and bottom of the column toward the feed point.

For such a calculation to be exact, the compositions obtained from both the bottom-up and top-down calculations must mesh at the feed point and mesh the feed composition.

The calculated compositions will depend on the compositions assumed for the top and bottom products at the commencement of the calculations.

Though it is possible to match the key components, the other components will not match unless the designer was particularly fortunate in choosing the trial top and bottom compositions.

Calculation procedure

For a completely rigorous solution the compositions must be adjusted and the calculations repeated until a satisfactory match at the feed point is obtained by iterative **trial-and-error calculations**.

Clearly, the greater the number of components, the more difficult the problem.

For other than ideal mixtures, the calculations will be further complicated by the fact that the component volatilities will be functions of the unknown stage compositions.

If more than a few stages are required, stage-by-stage calculations are complex and tedious.

“Short-cut" methods

Before the advent of the modern digital computer, various **"short-cut" methods** were developed to simplify the task of designing multicomponent columns.

Though computer programs will normally be available for the rigorous solution of the MESH equations, short-cut methods are still useful in the preliminary design work, and as an aid in defining problems for computer solution.

Intelligent use of the short-cut methods can reduce the computer time and costs.

“Short-cut" methods

The short-cut methods available can be divided into two classes:

1. **Simplifications of the rigorous stage-by-stage procedures** to enable the calculations to be done using hand calculators, or graphically.

Typical examples of this approach are the methods given by Hengstebeck (1961), and the Smith-Brinkley method (1960); which are described in Section 11.7 (C&R Vol. VI).

2. **Empirical methods**, which are based on the performance of operating columns, or the results of rigorous designs.

Typical examples of these methods are Gilliland's correlation, which is given in (C&R Vol. II, Chapter 11) and the Erbar-Maddox correlation given in Section 11.7.3 (C&R Vol. VI).

"*Key*" components

The designer must select the two **"key" components** between which it is desired to make the separation.

The **light key** will be the component that it is desired to keep out of the bottom product, and the heavy key the component to be kept out of the top product.

Specifications will be set on the maximum concentrations of the keys in the top and bottom products.

The keys are known as "adjacent keys" if they are "adjacent" in a listing of the components in order of volatility, and "split keys" if some other component lies between them in the order; they will usually be adjacent.

If any uncertainty exists in identifying keys components (*e.g.* isomers), trial calculations should be made using different components as the keys to determine the pair that requires the largest number of stages for separation (the worst case).

The "non-key" components that appear in both top and bottom products are known as "distributed" components; and those that are not present, to any significant extent, in one or other product, are known as "non-distributed" components.

Number and sequencing of columns

In multicomponent distillations it is not possible to obtain more than one pure component, one sharp separation, in a single column.

If a multicomponent feed is to be split into two or more virtually pure products, several columns will be needed.

Impure products can be taken off as side streams; and the removal of a side stream from a stage where a minor component is concentrated will reduce the concentration of that component in the main product.

For separation of N components, with one essentially pure component taken overhead, or from the bottom of each column, (N — 1) columns will be needed to obtain complete separation of all components.

For example, to separate a mixture of benzene, toluene and xylene two columns are needed (3-1), Benzene is taken overhead from the first column

and the bottom product, essentially free of benzene, is fed to the second column.

This column separates the toluene and xylene.

Costs considerations

The order in which the components are separated will determine the capital and operating costs.

Where there are several components the number of possible sequences can be very large; for example, with five components the number is 14, whereas with ten components it is near 5000.

When designing systems that require the separation of several components, efficient procedures are needed to determine the optimum sequence of separation.

Separation schemes for a 4 components mixture

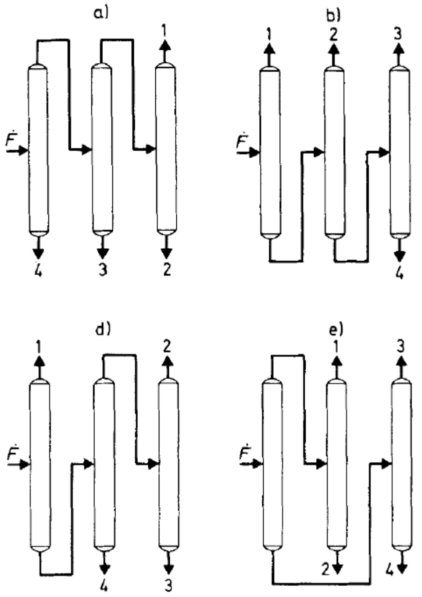


Fig. 2-20. Different methods of connecting three columns in a quaternary mixture distillation unit.
a)–d) Serial connections of columns
e) Parallel connection
F Feed
1, 2, 3, 4 Components in the order of increasing boiling points

Heuristic rules for optimum sequencing

1. Remove the components one at a time.

2. Remove any components that are present in large excess early in the sequence.

3. With difficult separations, involving close boiling components, postpone the most difficult separation to late in the sequence.

Difficult separations will require many stages, so to reduce cost, the column diameter should be made a small as possible. As the column diameter is dependent on flow-rate, the further down the sequence the smaller will be the amount of material that the column has to handle.

Tall and vacuum columns

Where a large number of stages is required, it may be necessary to split a column into two or more separate columns to reduce the height of the column, even though the required separation could, theoretically, have been obtained in a single column.

This may also be done in vacuum distillations, to reduce the column pressure drop and limit the bottom temperatures.

Short-cuts methods

Short-cut methods for stage and reflux requirements

Some of the more useful short-cut procedures which can be used to estimate stage and reflux requirements without the aid of computers are given in this section.

Most of the short-cut methods were developed for the design of separation columns for hydrocarbon systems in the petroleum and petrochemical systems industries, and caution must be exercised when applying them to other systems (as it is assumed almost ideal behavior of mixtures).

They usually depend on the **assumption of constant relative volatility**, *and should not be used for severely non-ideal systems.*

Pseudo-binary systems

If the presence of the other components does not significantly affect the volatility of the key components, the keys can be treated as a pseudo-binary pair.

The number of stages can then be calculated using a McCabe-Thiele diagram, or the other methods developed for binary systems.

This simplification can often be made when the amount of the non-key components is small, or where the components form near-ideal mixtures.

Where the concentration of the non-keys is small, say less than 10%, they can be lumped in with the key components.

For higher concentrations the method proposed by Hengstebeck (1946) can be used to reduce the system to an equivalent binary system.

Hengstebeck's method

For any component *i* the Lewis-Sorel material balance equations and equilibrium relationship can be written in terms of the individual component molar flow rates; in place of the component composition:

Rectifying section

$$v_{n+1,i} = l_{n,i} + d_i$$

$$v_{n,i} = K_{n,i} \frac{V}{L} l_{n,i}$$

Stripping section

$$l'_{n+1,i} = v'_{n,i} + b_i$$

$$v'_{n,i} = K_{n,i} \frac{V'}{L'} l'_{n,i}$$

where $l_{n,i}$ = the liquid flow rate of any component i from stage n,

$v_{n,i}$ = the vapour flow rate of any component i from stage n,

d_i = the flow rate of component i in the tops,

b_i = the flow rate of component i in the bottoms,

$K_{n,i}$ = the equilibrium constant for component i at stage n.

The superscript $'$ denotes the stripping section.

V and L are the total flow-rates, assumed constant.

Hengstebeck's method

To reduce a multicomponent system to an equivalent binary it is necessary to estimate the flow-rate of the key components throughout the column.

Hengstebeck considers that in a typical distillation the flow-rates of each of the light non-key components approaches a constant, limiting, rate in the rectifying section; and the flows of each of the heavy non-key components approach limiting flow-rates in the stripping section.

Hengstebeck's method

Putting the flow-rates of the non-keys equal to these limiting rates in each section enables the combined flows of the key components to be estimated.

lighter species

Rectifying section

$$L_e = L - \Sigma \underline{l}_i \quad (11.46)$$

$$V_e = V - \Sigma \underline{v}_i \quad (11.47)$$

Stripping section

heavier species

$$L'_e = L' - \Sigma \underline{l}'_i \quad (11.48)$$

$$V'_e = V' - \Sigma \underline{v}'_i \quad (11.49)$$

where V_e and L_e are the estimated flow rates of the combined keys,

$\underline{l}_i$ and $\underline{v}_i$ are the limiting liquid and vapour rates of components *lighter* than the keys in the rectifying section,

$\underline{l}'_i$ and $\underline{v}'_i$ are the limiting liquid and vapour rates of components *heavier* than the keys in the stripping section.

Hengstebeck's method

The method used to estimate the limiting flow-rates is that proposed by Jenny (1939). The equations are:

Rectifying section

$$\underline{l_i} = \frac{d_i}{\alpha_i - 1} \quad (11.50)$$

$$\underline{v_i} = l_i + d_i \quad (11.51)$$

Stripping section

$$\underline{v_i'} = \frac{\alpha_i b_i}{\alpha_{LK} - \alpha_i} \quad (11.52)$$

$$\underline{l_i'} = v_i' + b_i \quad (11.53)$$

where α_i = relative volatility of component i, relative to the heavy key (HK),

α_{LK} = relative volatility of the light key (LK), relative to the heavy key.

d_i and b_i = corresponding top and bottom flow rate of component *i*.

Hengstebeck's method

Estimates of the flows of the combined keys enable operating lines to be drawn for the equivalent binary system.

The equilibrium line is drawn by assuming a constant relative volatility for the light key:

$$y = \frac{\alpha_{LK} x}{1 + (\alpha_{LK} - 1)x}$$

where **y** and ***x*** *refer to the vapor and liquid concentrations of the light key.* Hengstebeck shows how the method can be extended to deal with situations where the relative volatility cannot be taken as constant, and how to allow for variations in the liquid and vapor molar flow rates.

He also gives a more rigorous graphical procedure based on the Lewis-Matheson method (see Section 11.8).

Bubble and Dew Point calculations

Equilibrium relationships

$$\begin{cases} y_i = K_i x_i & K_i=F(T) \quad \text{[De Priester charts]} \\ \sum_{i=1}^{c} y_i = 1 & (i = 1, 2, \ldots, c) \\ \sum_{i=1}^{c} x_i = 1 & \end{cases}$$

Bubble point temperature (x_i known):

$$\sum_{i=1}^{c} K_i(T) x_i = 1 \quad \Rightarrow \quad f(T) = \sum_{i=1}^{c} K_i(T) x_i - 1$$

Dew point temperature (y_i known):

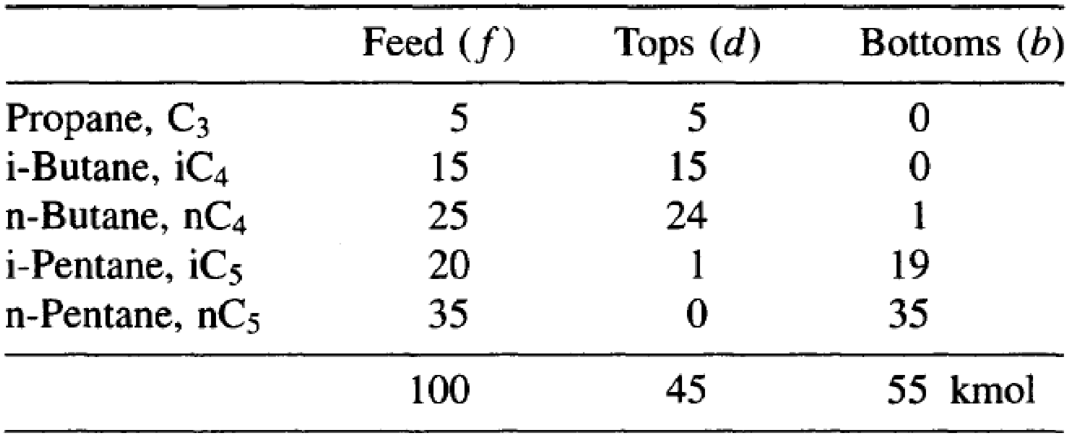
$$\sum_{i=1}^{c} y_i \big/ K_i(T) = 1 \quad \Rightarrow \quad f(T) = \sum_{i=1}^{c} y_i \big/ K_i(T) - 1$$

Solution: find the temperature *T* by trial-and-error procedure as f(T)=0

Example 11.5

Estimate the number of ideal stages needed in the butane-pentane splitter defined by the compositions given in the table below.

The column will operate at a pressure of 8.3 bar, with a reflux ratio R=2.5. The feed is at its boiling point (q=1).



	Feed (f)	Tops (d)	Bottoms (b)
Propane, C_3	5	5	0
i-Butane, iC_4	15	15	0
n-Butane, nC_4	25	24	1
i-Pentane, iC_5	20	1	19
n-Pentane, nC_5	35	0	35
	100	45	55 kmol

Assumed composition
(for DP & BP calculations)

	x_d	x_b
C_3	0.111	0.000
iC_4	0.333	0.000
nC_4	0.533	0.018
iC_5	0.022	0.345
nC_5	0.000	0.636

Note: a similar problem has been solved by Lyster et al. (1959) using a rigorous computer method and it was found that **10 stages were needed**.

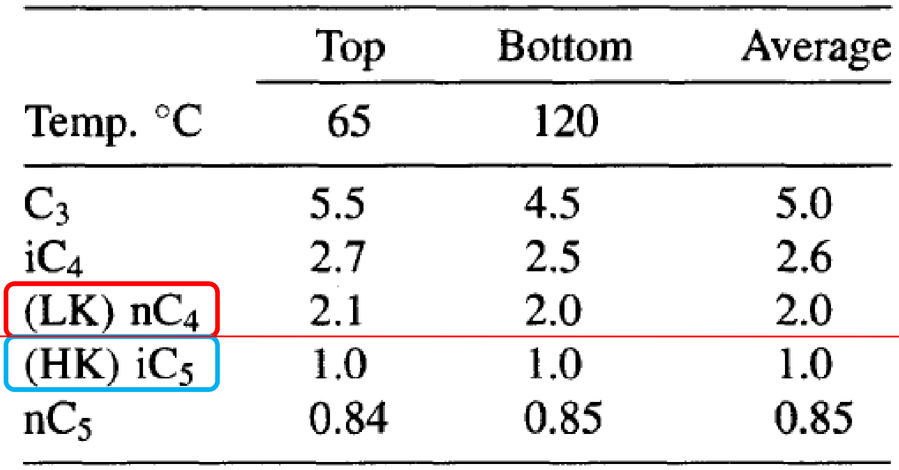
Example 11.5 - solution

The top and bottom temperatures (dew points and bubble points) were calculated by the methods illustrated in Example 11.9.

Relative volatilities are given by equation 8.30:

$$\alpha_i = \frac{K_i}{K_{HK}}$$

Equilibrium constants were taken from the De Priester charts. Relative volatilities estimated:



	Top	Bottom	Average	
Temp. °C	65	120		
C_3	5.5	4.5	5.0	Light non-key comp.
iC_4	2.7	2.5	2.6	Light non-key comp.
(LK) nC_4	2.1	2.0	2.0	
(HK) iC_5	1.0	1.0	1.0	
nC_5	0.84	0.85	0.85	Heavy non-key comp.

Example 11.5 - solution

Calculations of non-key flows:

Rectifying section

$$\underline{l}_i = \frac{d_i}{\alpha_i - 1} \quad (11.50)$$

$$\underline{v}_i = l_i + d_i \quad (11.51)$$

Stripping section

$$\underline{v}'_i = \frac{\alpha_i b_i}{\alpha_{\mathrm{LK}} - \alpha_i} \quad (11.52)$$

$$\underline{l}'_i = v'_i + b_i \quad (11.53)$$

		α_i	d_i	$\underline{l}_i = d_i/(\alpha_i - 1)$	$\underline{v}_i = \underline{l}_i + d_i$
Stripping section	C_3	5	5	1.3	6.3
	iC_4	2.6	15	9.4	24.4
			$\Sigma \underline{l}_i = 10.7$	$\Sigma \underline{v}_i = 30.7$	
		α_i	b_i	$\underline{v}'_i = \alpha_i b_i/(\alpha_{\mathrm{LK}} - \alpha_i)$	$\underline{l}'_i = \underline{v}'_i + b_i$
Rectifying section	nC_5	0.85	35	25.9	60.9
				$\Sigma \underline{v}'_i = 25.9$	$\Sigma \underline{l}'_i = 60.9$

Example 11.5 - solution

Flow of combined keys:

Rectifying section

$$L_e = L - \Sigma \underline{l_i} \quad (11.46)$$

$$V_e = V - \Sigma \underline{v_i} \quad (11.47)$$

Stripping section

$$L'_e = L' - \Sigma \underline{l'_i} \quad (11.48)$$

$$V'_e = V' - \Sigma \underline{v'_i} \quad (11.49)$$

L=RD
V=(R+1)D
V'=V-(1-q)F
L'=L+qF

$$L_e = 2.5 \times 45 - 10.7 = 101.8 \qquad (11.46)$$

$$V_e = (2.5 + 1)45 - 30.7 = 126.8 \qquad (11.47)$$

$$V'_e = (2.5 + 1)45 - 25.9 = 131.6 \qquad (11.49)$$

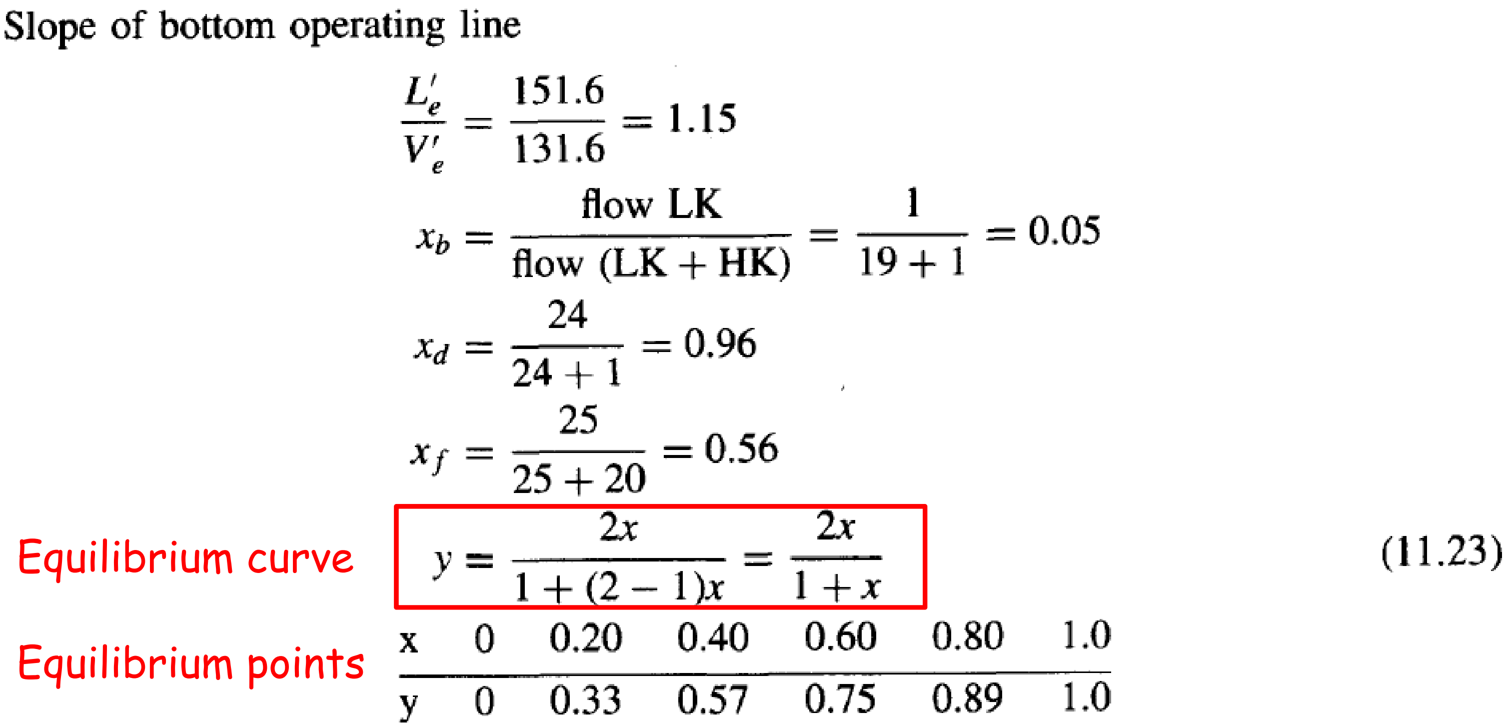
$$L'_e = (2.5 + 1)45 + 55 - 60.9 = 151.6 \qquad (11.48)$$

Slope of top operating line

$$\frac{L_e}{V_e} = \frac{101.8}{126.8} = 0.8$$

R= reflux ratio; q= feed thermal index (1 for boiling liquid; 0 for saturated vapour)

Example 11.5 - solution



Slope of bottom operating line

$$\frac{L'_e}{V'_e} = \frac{151.6}{131.6} = 1.15$$

$$x_b = \frac{\text{flow LK}}{\text{flow (LK + HK)}} = \frac{1}{19+1} = 0.05$$

$$x_d = \frac{24}{24+1} = 0.96$$

$$x_f = \frac{25}{25+20} = 0.56$$

Equilibrium curve

$$\boxed{y = \frac{2x}{1+(2-1)x} = \frac{2x}{1+x}} \qquad (11.23)$$

Equilibrium points

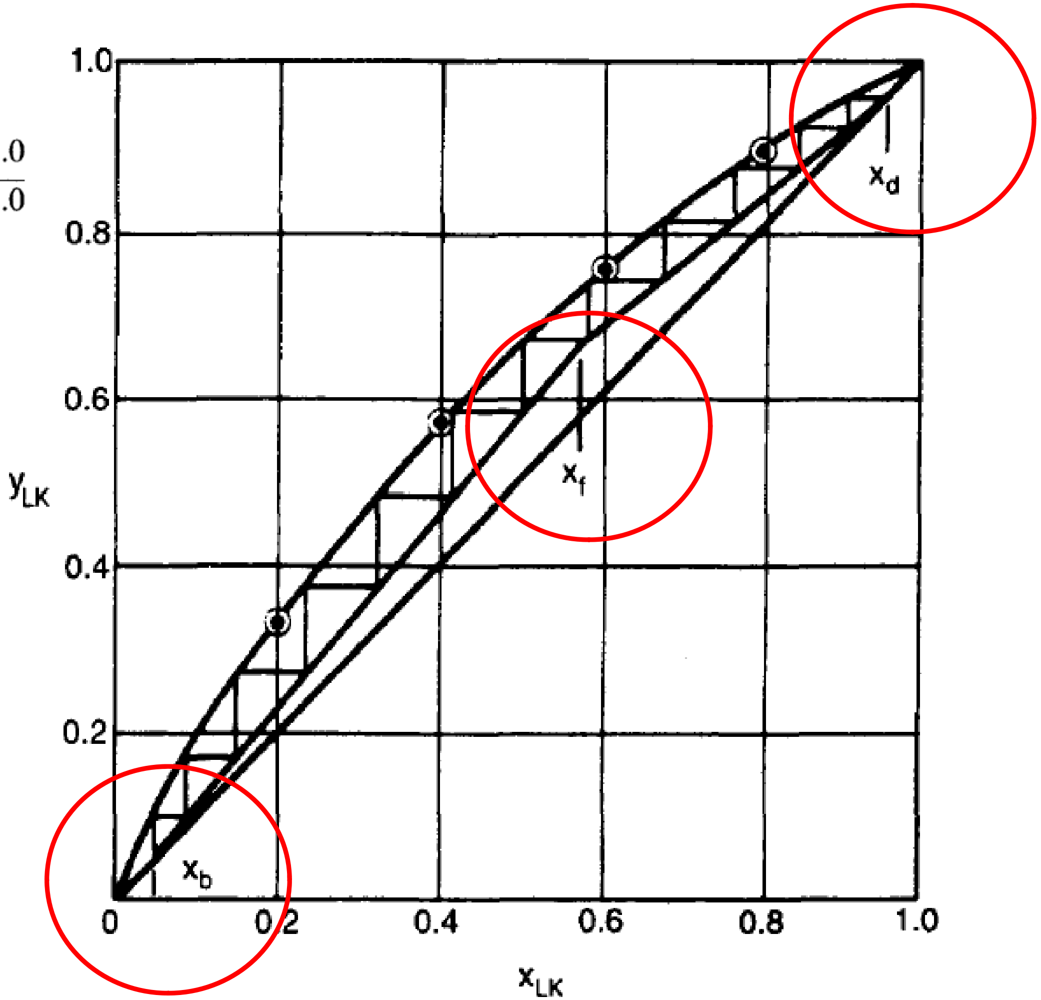
x	0	0.20	0.40	0.60	0.80	1.0
y	0	0.33	0.57	0.75	0.89	1.0

Example 11.5 - solution

The McCabe-Thiele diagram is shown in Figure: 12 stages required; feed on seventh from base.

$$y = \frac{2x}{1 + (2-1)x} = \frac{2x}{1+x}$$

x	0	0.20	0.40	0.60	0.80	1.0
y	0	0.33	0.57	0.75	0.89	1.0



Empirical correlations methods

The two most frequently used empirical methods for estimating the stage requirements for multicomponent distillations are the correlations published by Gilliland (1940) and by Erbar and Maddox (1961).

These relate the number of ideal stages required for a given separation, at a given reflux ratio, to the number at total reflux (minimum possible) and the minimum reflux ratio (infinite number of stages).

Gilliland's correlation is given in C-R Vol. 2, Chapter 11.

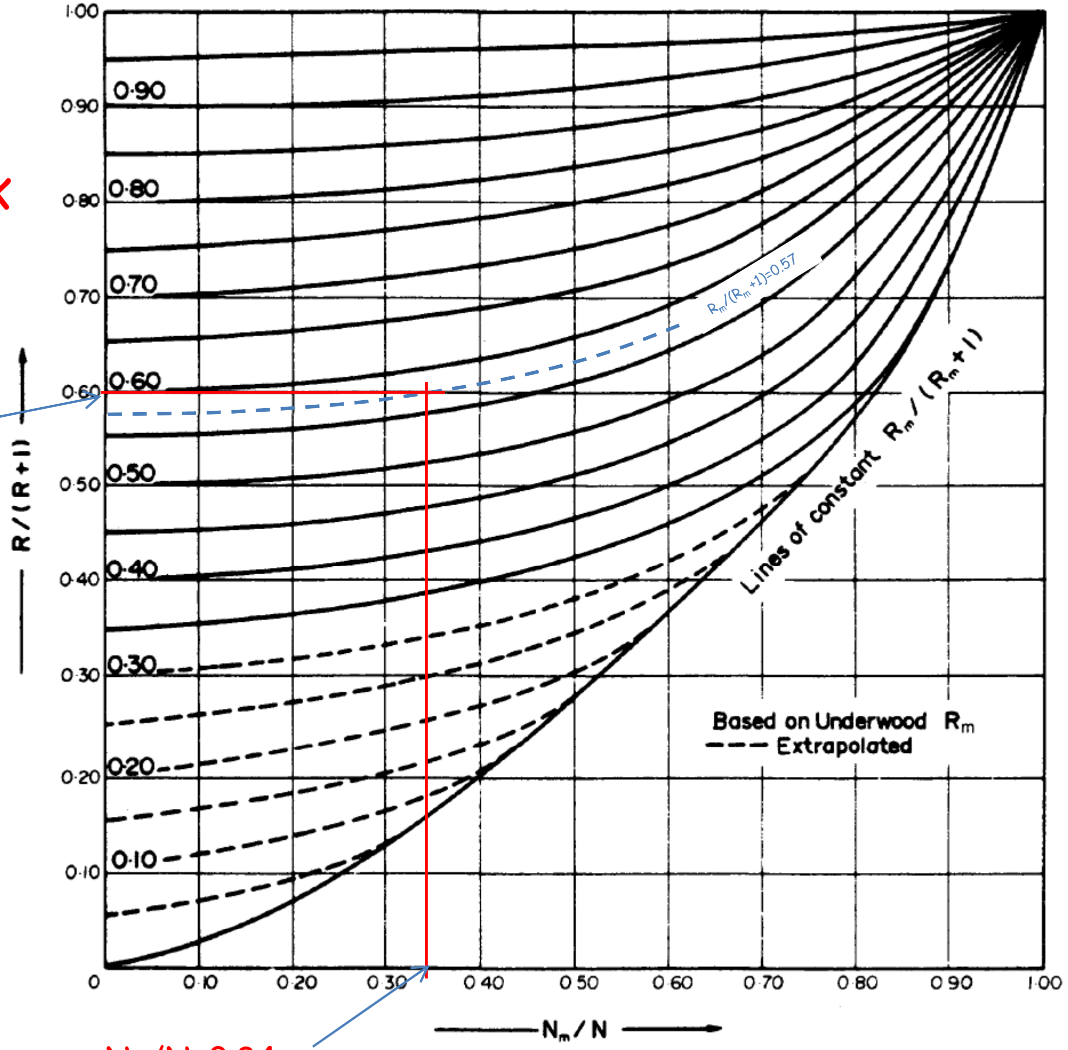
The Erbar-Maddox correlation is given in this section, as it is now generally considered to give more reliable predictions.

Their correlation is shown in Figure 11.11; which gives the ratio of number of stages required to the number at total reflux, as a function of the reflux ratio, with the minimum reflux ratio as a parameter.

To use Figure 11.11, estimates of the number of stages at total reflux and the minimum reflux ratio are needed.

Number of stages calculation

Fig.11.11
Erbar-Maddox
correlation
(1961)



R/(R+1)=0.6

$N_m/N=0.34$

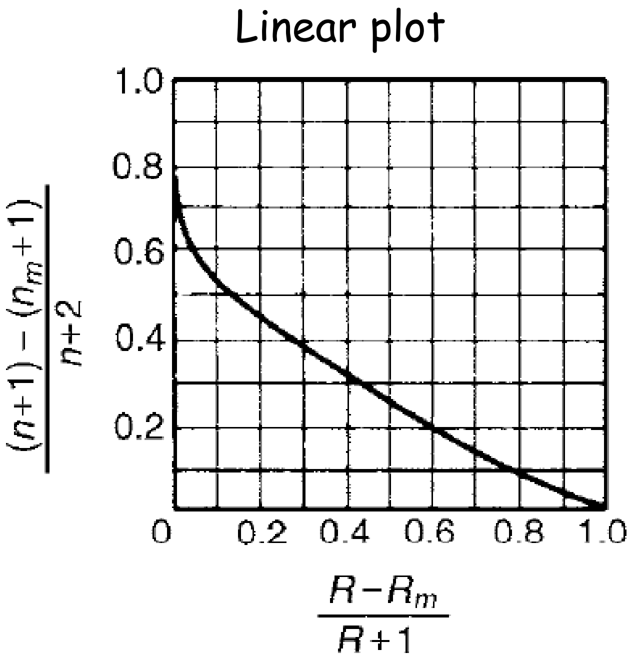
Example:
$R_m=1.33$
R=1.5

$R_m/(R_m+1)=0.57$
R/(R+1)=0.6

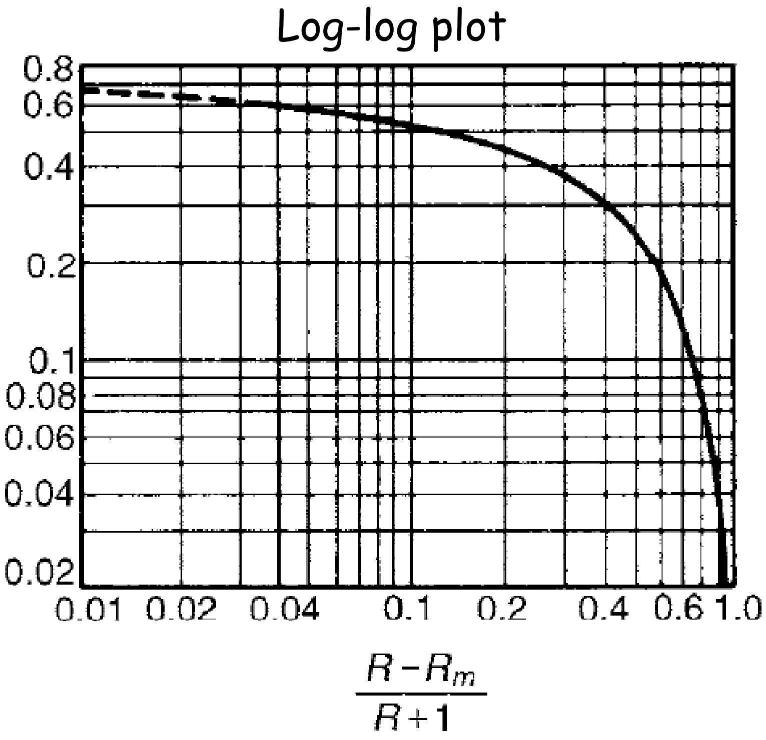
$N_m/N=0.34$

Number of stages calculation

Gilliland correlation (1940), C&R II Vol.



Linear plot



Log-log plot

$$\frac{n-n_m}{n+1}=1-\exp\left[\left(\frac{1+54.4\Psi}{11+117.2\Psi}\right)\left(\frac{\Psi-1}{\Psi^{0.5}}\right)\right]$$

where:

$$\Psi=\frac{R-R_m}{R+1}$$

Minimum number of stages (Fenske Equation)

The Fenske equation (Fenske, 1932) can be used to estimate the minimum stages required at total reflux.

The derivation of this equation for a binary system is given in C-R Vol. 2, Chapter 11. The equation applies equally to multicomponent systems and can be written as:

$$\left[\frac{x_i}{x_r}\right]_d = \alpha_i^{N_m}\left[\frac{x_i}{x_r}\right]_b \qquad (11.57)$$

where $[x_i/x_r]$ = the ratio of the concentration of any component i to the concentration of a reference component r, and the suffixes d and b denote the distillate (tops) (d) and the bottoms (b),

N_m = minimum number of stages at total reflux, including the reboiler,

α_i = average relative volatility of the component i with respect to the reference component.

Minimum number of stages (Fenske Equation)

Normally the separation required will be specified in terms of the key components, and equation 11.57 can be rearranged to give an estimate of the number of stages.

$$N_m = \frac{\log \left[\frac{x_{LK}}{x_{HK}}\right]_d \left[\frac{x_{HK}}{x_{LK}}\right]_b}{\log \alpha_{LK}} \qquad (11.58)$$

where α_{LK} is the average relative volatility of the light key with respect to the heavy key, and x_{LK} and x_{HK} *are the light and heavy key concentrations.*

The relative volatility is taken as the geometric mean of the values at the column top and bottom temperatures.

To calculate these temperatures initial estimates of the compositions must be made, so the calculation of the minimum number of stages by the Fenske equation is a trial-and- error procedure.

Large relative volatilities and feed location

If there is a wide difference between the relative volatilities at the top and bottom of the column the use of the average value in the Fenske equation will underestimate the number of stages.

In these circumstances, a better estimate can be made by calculating the number of stages in the rectifying and stripping sections separately.

The feed concentration is taken as the base concentration for the rectifying section and as the top concentration for the stripping section, and estimating the average relative volatilities separately for each section.

This procedure will also give an estimate of the feed point location

cannot be taken as constant.

Components split estimate

If the number of stages is known, equation 11.57 can be used to estimate the split of components between the top and bottom of the column at total reflux.

It can be written in a more convenient form for calculating the split of components:

Fenske Equation

$$\frac{d_i}{b_i} = \alpha_i^{N_m}\left[\frac{d_r}{b_r}\right] \quad (11.59)$$

where d_i and b_i are the flow-rates of the component *i* in the tops and bottoms, d_r and b_r are the flow-rates of the reference (light key) component in the tops and bottoms.

Note: from the column material balance:

$$d_i + b_i = f_i$$

where f_i is the flow rate of component *i* in the feed.

Minimum reflux ratio – Underwood equation

Colburn (1941) and Underwood (1948) have derived equations for estimating the minimum reflux ratio for multicomponent distillations. These equations are discussed in C-R Vol. 2, Chapt. 11.

As the Underwood equation is more widely used it is presented in this section. The equation can be stated in the form:

$$\sum \frac{\alpha_i x_{i,d}}{\alpha_i - \theta} = R_m + 1 \qquad (11.60)$$

α_i = the relative volatility of component i with respect to some reference component, usually the heavy key;

R_m = the minimum reflux ratio;

$x_{i,d}$ = concentration of component i in the tops at minimum reflux;

and θ is the root of the equation: $\sum \frac{\alpha_i x_{i,f}}{\alpha_i - \theta} = 1 - q \qquad 1 < \theta < \alpha_{LK}$

$x_{i,f}$ = the concentration of component i in the feed, and q depends on the condition of the feed and was defined in Section 11.5.2.

The value of θ must lie between the values of the relative volatility of the light and heavy keys, and is found by trial and error.

Minimum reflux ratio

In the derivation of equations 11.60 and 11.61 the relative volatilities are taken as constant.

The geometric average of values estimated at the top and bottom temperatures should be used. This requires an estimate of the top and bottom compositions.

Though the compositions should strictly be those at minimum reflux, the values determined at total reflux, from the Fenske equation, can be used.

A better estimate can be obtained by replacing the number of stages at total reflux in equation 11.59 by an estimate of the actual number; a value equal to $N_m/0.6$ *is often used.*

Feed-point location

A limitation of the Erbar-Maddox, and similar empirical methods, is that they do not give the feed-point location.

An estimate can be made by using the Fenske equation to calculate the number of stages in the rectifying and stripping sections separately, but this requires an estimate of the feed-point temperature.

An alternative approach is to use the empirical equation given by **Kirkbride** (1944):

$$\log\left[\frac{N_r}{N_s}\right] = 0.206 \log\left[\left(\frac{B}{D}\right)\left(\frac{x_{f,\mathrm{HK}}}{x_{f,\mathrm{LK}}}\right)\left(\frac{x_{b,\mathrm{LK}}}{x_{d,\mathrm{HK}}}\right)^2\right] \tag{11.62}$$

where N_r = number of stages above the feed, including any partial condenser,

N_s = number of stages below the feed, including the reboiler,

B = molar flow bottom product,

D = molar flow top product,

$x_{f,\mathrm{HK}}$ = concentration of the heavy key in the feed,

$x_{f,\mathrm{LK}}$ = concentration of the light key in the feed,

$x_{d,\mathrm{HK}}$ = concentration of the heavy key in the top product,

$x_{b,\mathrm{LK}}$ = concentration of the light key if in the bottom product.

Distribution of non-key components (graphical method)

The graphical procedure proposed by Hengstebeck (1946), which is based on the Fenske equation, is a convenient method for estimating the distribution of components between the top and bottom products.

Hengstebeck and Geddes (1958) have shown that the Fenske equation can be written in the form:

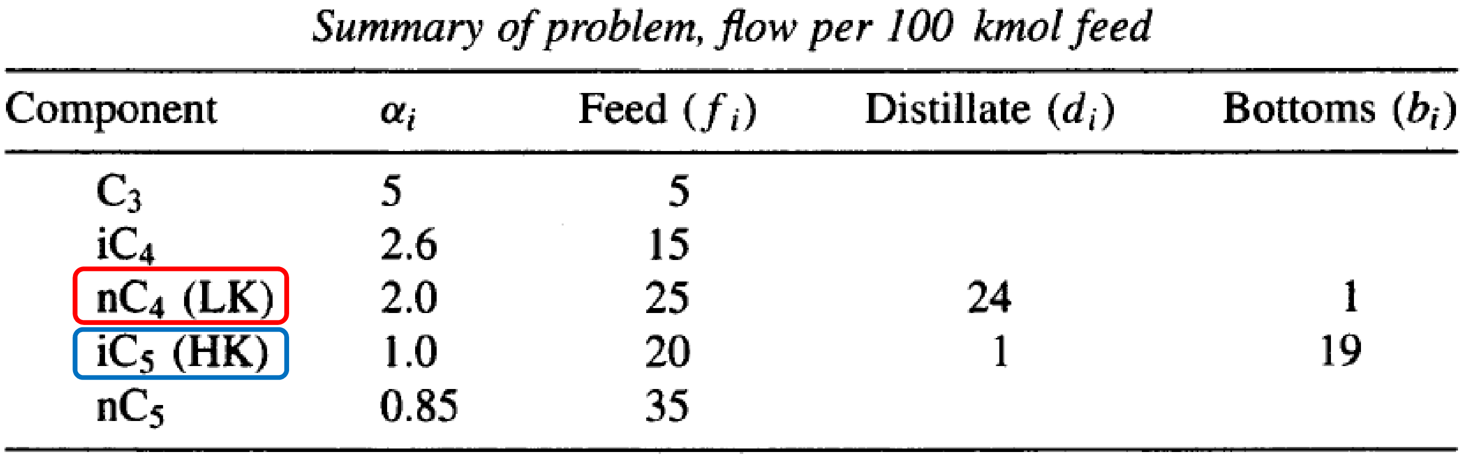
$$\log\left(\frac{d_i}{b_i}\right) = A + C\log\alpha_i \qquad (11.63)$$

Specifying the split of the key components determines the constants *A and C in the* equation.

The distribution of the other components can be readily determined by plotting the distribution of the keys against their relative volatility on log-log paper, and drawing a straight line through these two points.

Example 11.6: Geddes-Hengstebeck method

Use the Geddes-Hengstebeck method to check the component distributions for the separation specified in Example 11.5



Summary of problem, flow per 100 kmol feed

Component	α_i	Feed (f_i)	Distillate (d_i)	Bottoms (b_i)
C_3	5	5		
iC_4	2.6	15		
nC_4 (LK)	2.0	25	24	1
iC_5 (HK)	1.0	20	1	19
nC_5	0.85	35		

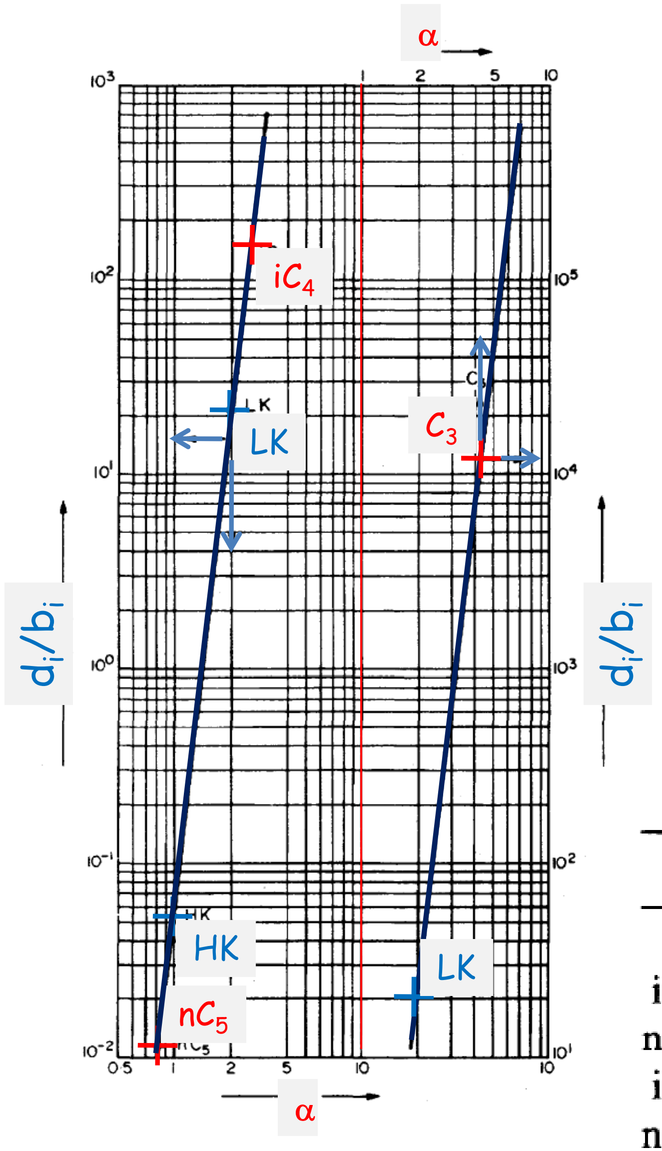
Example 11.6: solution

The average volatilities will be taken as those estimated in Example 11.5. Normally, the volatilities are estimated at the feed bubble point, which gives a rough indication of the average column temperatures. The dew point of the tops and bubble point of the bottoms can be calculated once the component distributions have been estimated, and the calculations repeated with a new estimate of the average relative volatilities, as necessary.

$$\text{For the light key, } \frac{d_i}{b_i} = \frac{24}{1} = 24$$

$$\text{For the heavy key, } \frac{d_i}{b_i} = \frac{1}{19} = 0.053$$

The distribution of the non-keys are read from Figure.
As these values are close to those assumed for the calculation of the dew points and bubble points, there is no need to repeat with new estimates of the relative volatilities.



Component distribution

Light key ➔ $d_i/b_i=24$, $\alpha_{LK}= 2$

Heavy key ➔ $d_i/b_i=0.053$, $\alpha_{HK}= 1$

The distribution of the non-keys are read from Figure at the appropriate relative volatility and the component flows are calculated from the following equations:

Overall column material balance ➔ $d_i+b_i=f_i$

From which
$$d_i = \frac{f_i}{\left(\frac{b_i}{d_i}+1\right)} \qquad b_i = \frac{f_i}{\left(\frac{d_i}{b_i}+1\right)}$$

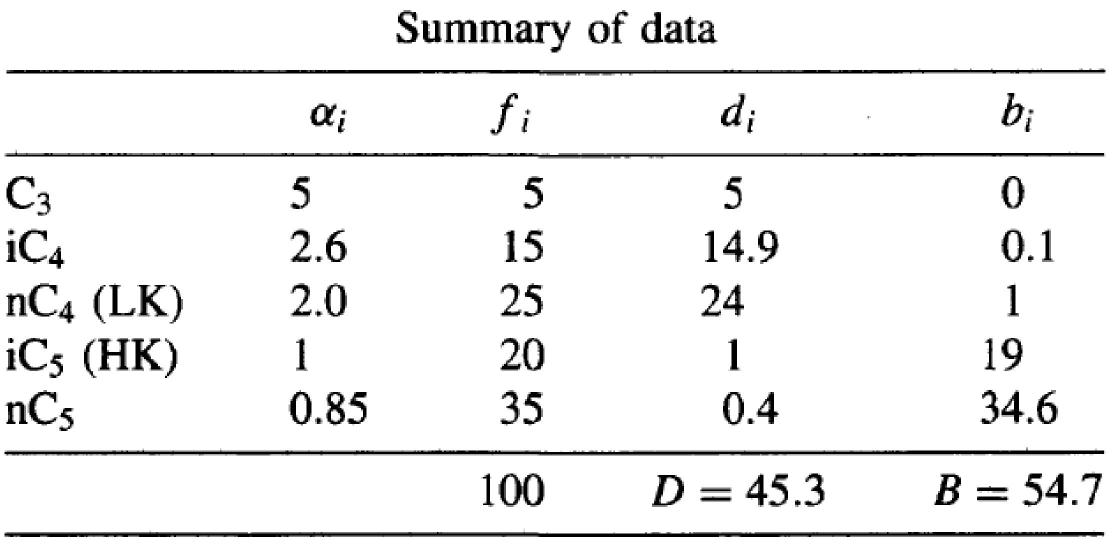
	α_i	f_i	d_i/b_i	d_i	b_i
C_3	5	5	40,000	5	0
iC_4	2.6	15	150	14.9	0.1
nC_4	2.0	25	21	24	1
iC_5	1.0	20	0.053	1	19
nC_5	0.85	35	0.011	0.4	34.6

Example 11.7 - Erbar-Maddox method

For the separation specified in Example 11.5, evaluate the effect of changes in reflux ratio on the number of stages required.

Solution

The relative volatilities estimated in Example 11.5, and the component distributions calculated in Example 11.6 will be used for this example.



Summary of data

	α_i	f_i	d_i	b_i
C_3	5	5	5	0
iC_4	2.6	15	14.9	0.1
nC_4 (LK)	2.0	25	24	1
iC_5 (HK)	1	20	1	19
nC_5	0.85	35	0.4	34.6
		100	$D = 45.3$	$B = 54.7$

Example 11.7 - solution

Minimum number of stages; Fenske equation, equation 11.58:

$$N_m = \frac{\log\left[\left(\frac{24}{1}\right)\left(\frac{19}{1}\right)\right]}{\log 2} = \underline{\underline{8.8}}$$

Minimum reflux ratio; Underwood equations 11.60 and 11.61.

As the feed is at its boiling point *q = 1*

$$\sum \frac{\alpha_i x_{i,f}}{\alpha_i - \theta} = 0$$

Trial and error calculation

				Try	
$x_{i,f}$	α_i	$\alpha_i x_{i,f}$	$\theta = 1.5$	$\theta = 1.3$	$\theta = 1.35$
0.05	5	0.25	0.071	0.068	0.068
0.15	2.6	0.39	0.355	0.300	0.312
0.25	2.0	0.50	1.000	0.714	0.769
0.20	1	0.20	−0.400	−0.667	−0.571
0.35	0.85	0.30	−0.462	−0.667	−0.600
			$\Sigma = 0.564$	−0.252	0.022 close enough

$\theta = 1.35$

Example 11.7 solution

Underwood equation (11.60)

$$\sum \frac{\alpha_i x_{i,d}}{\alpha_i - \theta} = R_m + 1$$

$\theta = 1.35$

$x_{i,d}$	α_i	$\alpha_i x_{i,d}$	$\alpha_i x_{i,d}/(\alpha_i - \theta)$
0.11	5	0.55	0.15
0.33	2.6	0.86	0.69
0.53	2.0	1.08	1.66
0.02	1	0.02	−0.06
0.01	0.85	0.01	−0.02
			$\Sigma = 2.42$

$$R_m + 1 = 2.42$$

$$R_m = \underline{\underline{1.42}}$$

$$\rightarrow \quad \frac{R_m}{(R_m + 1)} = \frac{1.42}{2.42} = 0.59$$

Case of R=2.0: $\frac{R}{(R+1)} = \frac{2}{3} = 0.66$

From E-M diagram (Fig. 11.11):

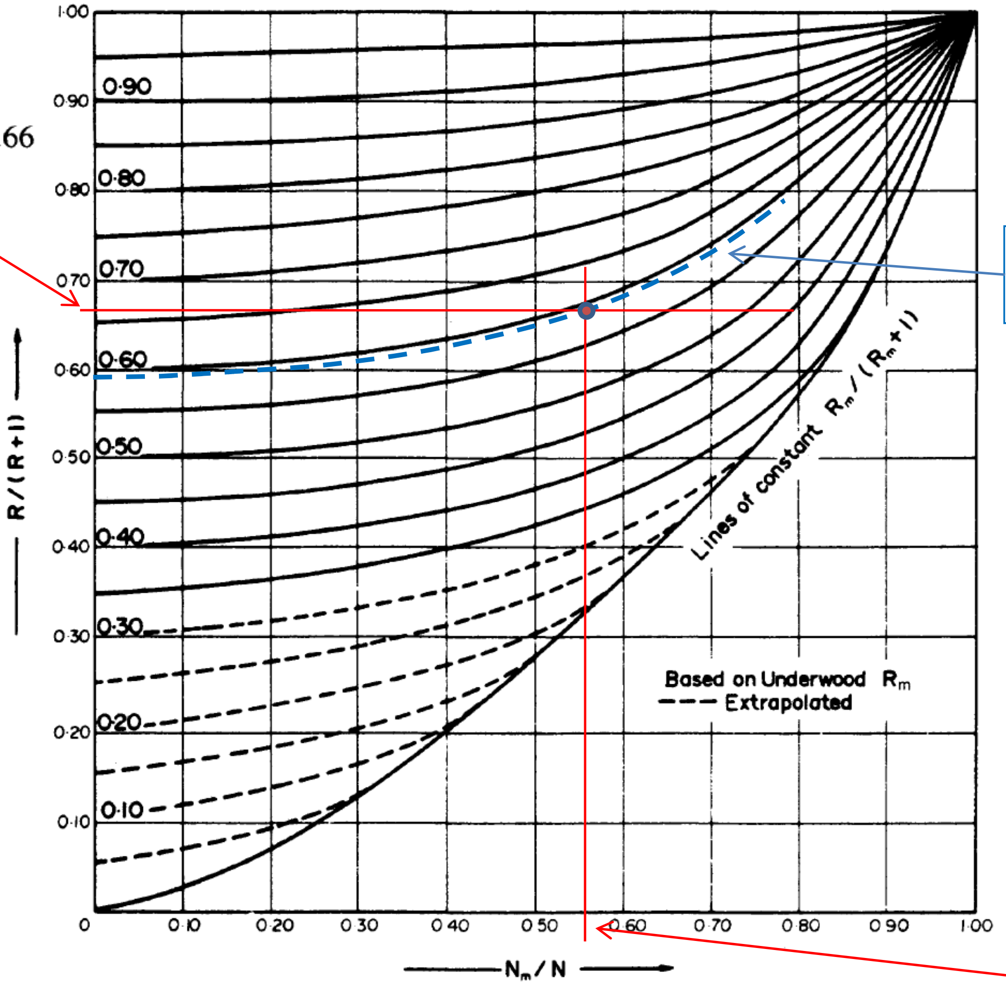
$$\downarrow$$

$$\frac{N_m}{N} = 0.56 \qquad N = \frac{8.8}{0.56} = \underline{\underline{15.7}}$$

for other reflux ratios

R	2	3	4	5	6
N	15.7	11.9	10.7	10.4	10.1

Example 11.7 - solution



$$\frac{R}{(R+1)} = \frac{2}{3} = 0.66$$

$$\frac{R_m}{(R_m+1)} = \frac{1.42}{2.42} = 0.59$$

$$\frac{N_m}{N} = 0.56$$

Example 11.8 - Feed point estimation

Estimate the position of the feed point for the separation considered in Example 11.7, for a reflux ratio of 3.

Solution

Use the **Kirkbride equation** (11.62):

$$\log\left[\frac{N_r}{N_s}\right] = 0.206\log\left[\left(\frac{B}{D}\right)\left(\frac{x_{f,\mathrm{HK}}}{x_{f,\mathrm{LK}}}\right)\left(\frac{x_{b,\mathrm{LK}}}{x_{d,\mathrm{HK}}}\right)^2\right]$$

Product distributions taken from Example 11.6:

$$x_{b,\mathrm{LK}} = \frac{1}{54.7} = 0.018 \qquad x_{d,\mathrm{HK}} = \frac{1}{45.3} = 0.022$$

$$\log\left(\frac{N_r}{N_s}\right) = 0.206\log\left[\frac{54.7}{45.3}\left(\frac{0.20}{0.25}\right)\left(\frac{0.018}{0.022}\right)^2\right] \qquad \log\left(\frac{N_r}{N_s}\right) = 0.206\log(0.65) \rightarrow \boxed{\frac{N_r}{N_s} = \underline{\underline{0.91}}}$$

for $R = 3$, $N = 12$

number of stages, excluding the reboiler $= \underline{\underline{11}}$

$$N_r + N_s = 11$$

$$N_s = 11 - N_r = 11 - 0.91N_s \rightarrow \boxed{N_s = \frac{11}{1.91} = 5.76, \text{ say } \underline{\underline{6}}}$$

Summary of empiric methods

Data: feed composition and thermal condition, operating pressure.

1. Choose ***LK*** and ***HK*** and fix their distribution on top and bottom product
2. Estimate overall top and bottom flow rate compositions (assume light non-key components in top and heavy non-key component in bottom as first attempt)

3. Estimate dew and bubble points on top, bottom and feed

$$\text{Bubble point} \quad \sum y_i = \sum K_i x_i = 1.0 \qquad (11.5a)$$

$$\text{Dew point} \quad \sum x_i = \sum \frac{y_i}{K_i} = 1.0 \qquad (11.5b)$$

(K_i taken from De Priest chart).

Summary of empiric method - cnt

4. Estimate relative volatility with respect to the HK of all comps. at the T_{dew} (top) and $T_{boiling}$ (bottom) : $\alpha_i = \frac{K_i}{K_{\mathrm{HK}}}$

   and calculate average volatilities $\alpha_i = (\alpha_{i,top} * \alpha_{i,bot})^{0.5}$

5. Recalculate overall top and bottom flow rate compositions on the basis of the Hengstebeck and Geddes equation:

$$\log\left(\frac{d_i}{b_i}\right) = A + C\log\alpha_i$$

by plotting on log-log diagrams knew points relevant to LK & HK (assume first trial relative volatilities)

Please note that $A=\log(d_{HK}/b_{HK})$ and $C=[\log(d_{LK}/b_{LK}) - A]/\log\alpha_{LK}$

Check first attempt composition and go back to step 3 to update T_{dew} and $T_{boiling}$ and recalculate relative volatilities if necessary.

Summary of empiric method - cnt

6. Estimate the minimum number of stages for the separation ***N_m***

Fenske eq.:

$$N_m = \frac{\log\left[\frac{x_{LK}}{x_{HK}}\right]_d \left[\frac{x_{HK}}{x_{LK}}\right]_b}{\log \alpha_{LK}}$$

7. Estimate the minimum reflux ratio ***R_m***

Underwood eq.:

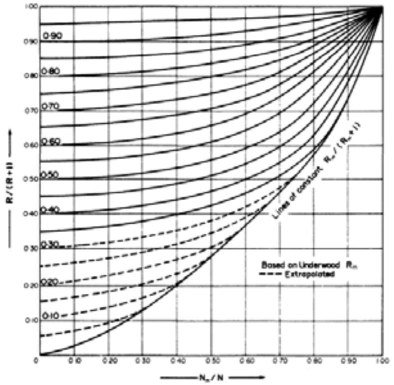
$$\sum \frac{\alpha_i x_{i,d}}{\alpha_i - \theta} = R_m + 1$$

where θ is the root of eq.: $$\sum \frac{\alpha_i x_{i,f}}{\alpha_i - \theta} = 1 - q$$ $q = (L_R - L_S)/F$

Please note that $1 < \theta < \alpha_{LK}$

Summary of empiric method - cnt

8. Choose an operative reflux ratio *R* and calculate the number of (ideal) stages ***N*** with the



a) Erbar-Maddox diagram

or

b) with Gilliland Correlation:

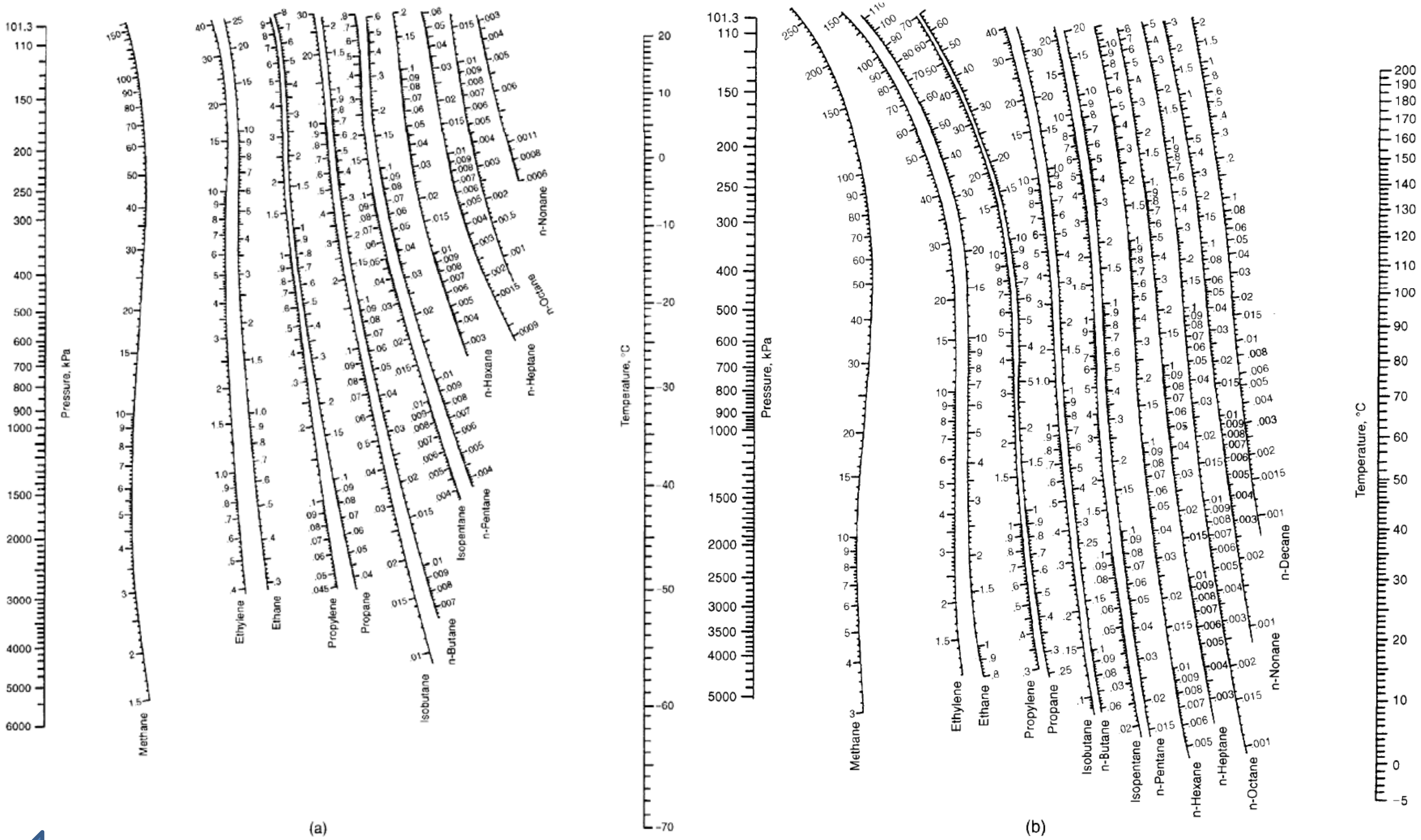
$$\frac{N - N_m}{N+1} = 1 - \exp\left[\left(\frac{1+54.4\Psi}{11+117.2\Psi}\right)\left(\frac{\Psi - 1}{\Psi^{0.5}}\right)\right]$$

$$\Psi = \frac{R - R_m}{R+1}$$

9. Calculate the feed stage position

Kirkbride eq.: $\log\left[\frac{N_r}{N_s}\right] = 0.206 \log\left[\left(\frac{B}{D}\right)\left(\frac{x_{f,\mathrm{HK}}}{x_{f,\mathrm{LK}}}\right)\left(\frac{x_{b,\mathrm{LK}}}{x_{d,\mathrm{HK}}}\right)^2\right]$

De Priester charts - values for hydrocarbons



Low temperature **High temperature**

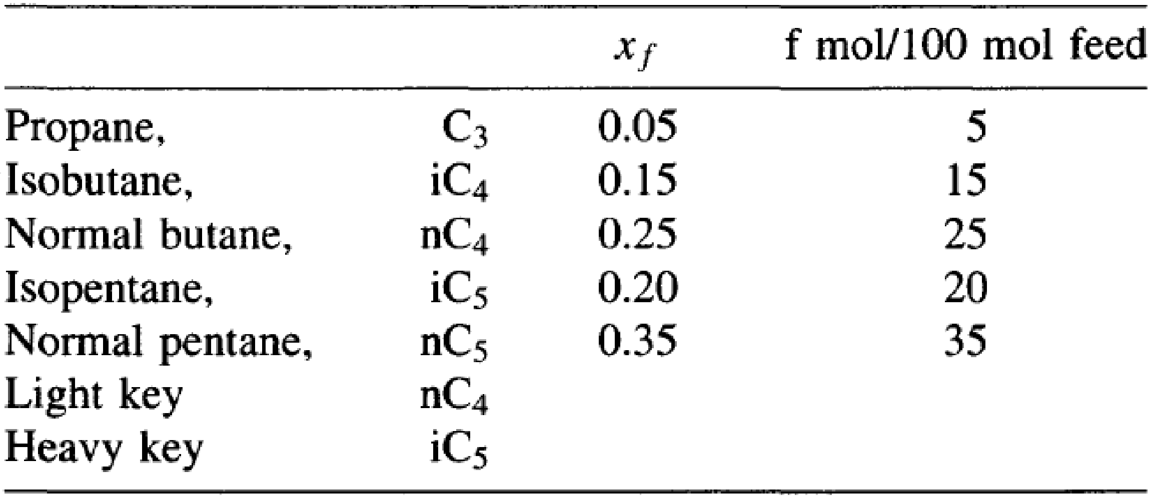
Exact stage by stage calculation methods

Example 11.9 – Stage by stage calculation

This example illustrates the complexity and trial and error nature of stage-by-stage calculation.

The same problem specification has been used in earlier examples to illustrate the shortcut design methods.

A butane-pentane splitter is to operate at 8.3 bar with the following feed composition:



			x_f	f mol/100 mol feed
	Propane,	C_3	0.05	5
	Isobutane,	iC_4	0.15	15
LK	Normal butane,	nC_4	0.25	25
HK	Isopentane,	iC_5	0.20	20
	Normal pentane,	nC_5	0.35	35
	Light key	nC_4		
	Heavy key	iC_5		

Specification: not more than 1 mol of the light key in the bottom product and not more than 1 mol of the heavy key in the top product, reflux ratio of 2.5. Make a stage-by-stage calculation to determine the product composition and number of stages required.

Example 11.9 – solution

Only sufficient trial calculations will be made to illustrate the method used.

Basis 100 mol feed.

Estimation of dew and bubble points:

$$\text{Bubble point} \quad \sum y_i = \sum K_i x_i = 1.0$$

$$\text{Dew point} \quad \sum x_i = \sum \frac{y_i}{K_i} = 1.0$$

The K values, taken from the De Priester charts, are plotted in Figure for easy interpolation.

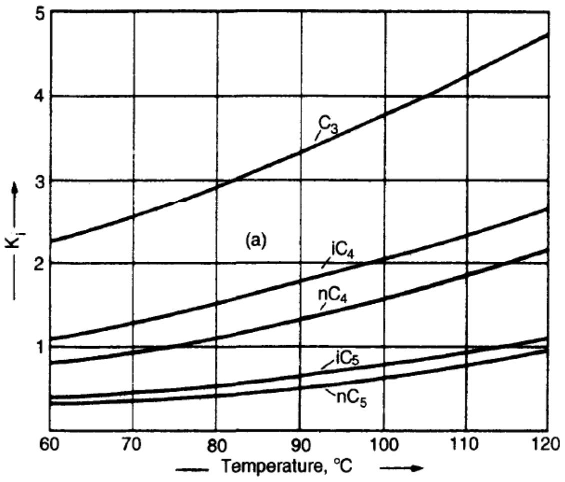


Figure (*a*). *K*-values at 8.3 bar

Example 11.9 – solution

To estimate the dew and bubble points, assume that nothing heavier than the heavy key appears in the tops, and nothing lighter than the light key in the bottoms.

Bottom & top composition
(first attempt assumption)

	d	x_d	b	x_b
C_3	5	0.11	0	—
C_4	15	0.33	0	—
nC_4	24	0.54	1	0.02
iC_5	1	0.02	19	0.34
nC_5	0	—	35	0.64
	45		55	

Bubble-point calculation, **bottoms**

		Try 100°C		Try 120°C	
	x_b	K_i	K_ix_i	K_i	K_ix_i
C_3	—	—	—	—	—
iC_4	—	—	—	—	—
nC_4	0.02	1.85	0.04	2.1	0.04
iC_5	0.34	0.94	0.32	1.1	0.37
nC_5	0.64	0.82	0.52	0.96	0.61
		$\Sigma K_ix_i = 0.88$			1.02
		temp. too low			close enough

Dew-point calculation, **tops**

		Try 70°C		Try 60°C	
	x_d	K_i	y_i/K_i	K_i	y_i/K_i
C_3	0.11	2.6	0.04	2.20	0.24
iC_4	0.33	1.3	0.25	1.06	0.35
nC_4	0.54	0.9	0.60	0.77	0.42
iC_5	0.02	0.46	0.04	0.36	0.01
nC_5	—	—	—	—	—
		$\Sigma y_i/K_i = 0.94$			1.02
		temp. too high			close enough

Bubble-point calculation, **feed (liquid)**

		Try 80°C		Try 90°C		Try 85°C	
	x_f	K_i	x_iK_i	K_i	x_iK_i	K_i	x_iK_i
C_3	0.05	2.9	0.15	3.4	0.17	3.15	0.16
iC_4	0.15	1.5	0.23	1.8	0.27	1.66	0.25
nC_4	0.25	1.1	0.28	1.3	0.33	1.21	0.30
iC_5	0.20	0.5	0.11	0.66	0.13	0.60	0.12
nC_5	0.35	0.47	0.16	0.56	0.20	0.48	0.17
			0.93		1.10		1.00
			temp. too low		temp. too high		satisfactory

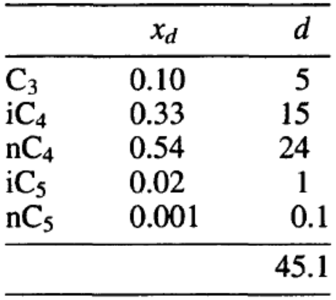
Example 11.9 - solution

Top down calculations, assume total condensation with no sub-cooling

$$y_1 = x_d - x_0$$

It is necessary to estimate the composition of the "non-keys" so that they can be included in the stage calculations.

First trial top composition:

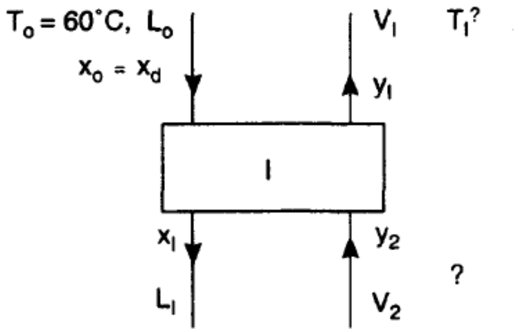


	x_d	d
C_3	0.10	5
iC_4	0.33	15
nC_4	0.54	24
iC_5	0.02	1
nC_5	0.001	0.1
		45.1

In each stage calculation it will necessary to estimate the stage temperatures to determine the *K values and liquid and vapor enthalpies*. The temperature range from top to bottom of the column will be approximately 120 — 60 = 60°C. An *approximate* calculation (Example 11.7) has shown that around 14 ideal stages will be needed; so the temperature change from stage to stage can be expected to be around 4 to 5°C.

Example 11.9 – Stage 1

Estimation of stage temperature and outlet liquid composition *(x_{1i})*,
Impose equilibrium between V_1 and L_1:



$$L_0 = R \times D = 2.5 \times 45.1 = 112.8$$
$$V_1 = (R+1)D = 3.5 \times 45.1 = 157.9$$

	y_1	Try $T_1 = 66°C$		Try $T_1 = 65°C$		
		K_i	y_i/K_i	K_i	y_i/K_i	$x_1 = y_i/K_i$ Normalised
C_3	0.10	2.40	0.042	2.36	0.042	0.042
iC_4	0.33	1.20	0.275	1.19	0.277	0.278
nC_4	0.54	0.88	0.614	0.86	0.628	0.629
iC_5	0.02	0.42	0.048	0.42	0.048	0.048
nC_5	0.001	0.32	0.003	0.32	0.003	0.003
		$\Sigma y_i/K_i = 0.982$ too low			0.998 close enough	

Example 11.9 – Stage 1

Summary of stage equations:

$$L_0 + V_2 = L_1 + V_1 \qquad \text{(i)}$$

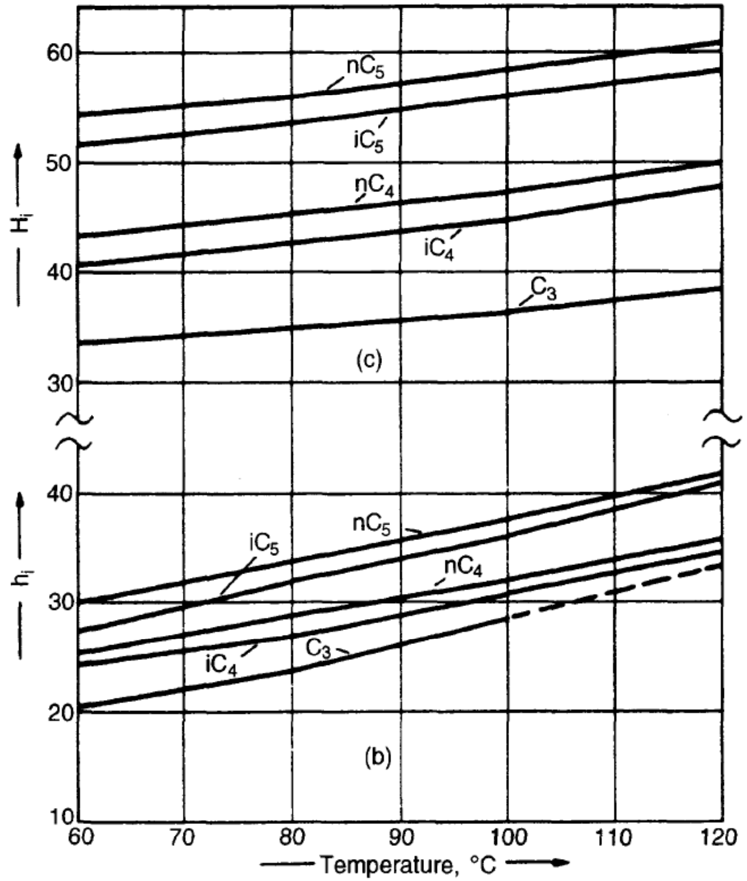
$$L_0 x_0 + V_2 y_2 = L_1 x_1 + V_1 y_1 \qquad \text{(ii)}$$

$$h_0 L_0 + H_2 V_2 = h_1 L_1 + H_1 V_1 \qquad \text{(iii)}$$

$$h = f(x, T) \qquad \text{(iv)}$$

$$H = f(x, T) \qquad \text{(v)}$$

$$y_i = K_i x_i \qquad \text{(vi)}$$



Enthalpy [kJ/kmol] diagram of liquid and vapor hydrocarbons

Example 11.9 – Stage 1

Before a heat balance can be made to estimate L_1 and V_2 an estimate of y_1 and T_1 is needed. V_2 is dependent on the liquid and vapor flows, so as a first trial assume that these are constant and equal to L_0 and V_1; then, from equations (i) and (ii):

$$y_2 = \left(\frac{L_0}{V_1}\right)(x_1 - x_0) + y_1$$

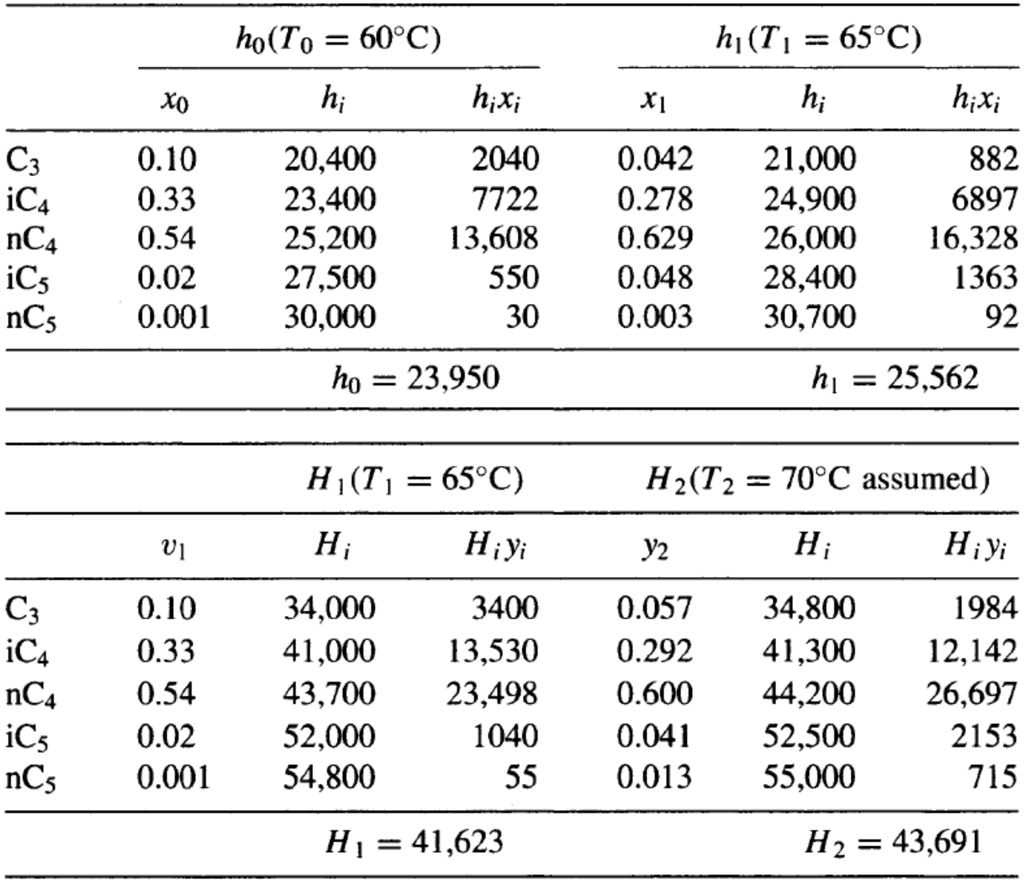
Approximate mass balance over the stage

$$\frac{L_0}{V_1} = \frac{112.8}{157.9} = 0.71$$

	x_1	x_0	$y_2 = 0.71(x_1 - x_0) + y_1$	y_2 Normalised
C_3	0.042	0.10	0.057	0.057
iC_4	0.278	0.33	0.294	0.292
nC_4	0.629	0.54	0.604	0.600
iC_5	0.048	0.02	0.041	0.041
nC_5	0.003	0.001	0.013	0.013
			1.009 close enough	

Example 11.9 – Stage 1

Enthalpy data from figure:



	$h_0(T_0 = 60°C)$			$h_1(T_1 = 65°C)$		
	x_0	h_i	$h_i x_i$	x_1	h_i	$h_i x_i$
C_3	0.10	20,400	2040	0.042	21,000	882
iC_4	0.33	23,400	7722	0.278	24,900	6897
nC_4	0.54	25,200	13,608	0.629	26,000	16,328
iC_5	0.02	27,500	550	0.048	28,400	1363
nC_5	0.001	30,000	30	0.003	30,700	92
		$h_0 = 23{,}950$			$h_1 = 25{,}562$	

	$H_1(T_1 = 65°C)$			$H_2(T_2 = 70°C \text{ assumed})$		
	v_1	H_i	$H_i y_i$	y_2	H_i	$H_i y_i$
C_3	0.10	34,000	3400	0.057	34,800	1984
iC_4	0.33	41,000	13,530	0.292	41,300	12,142
nC_4	0.54	43,700	23,498	0.600	44,200	26,697
iC_5	0.02	52,000	1040	0.041	52,500	2153
nC_5	0.001	54,800	55	0.013	55,000	715
		$H_1 = 41{,}623$			$H_2 = 43{,}691$	

Example 11.9 – Stage 1

Energy balance (equation iii)

$$23{,}950 \times 112.8 + 43{,}691 V_2 = 25{,}562 L_1 + 41{,}623 \times 157.9$$

$$43{,}691 V_2 = 255{,}626 L_1 + 3{,}870{,}712$$

Material balance (equation i)

$$112.8 + V_2 = L_1 + 157.9$$

substituting

$$43{,}691(L_1 + 45.1) = 25{,}562 L_1 + 3{,}870{,}712$$

$$L_1 = 104.8$$

$$V_2 = 104.8 + 45.1 = 149.9$$

$$\frac{L_1}{V_2} = 0.70$$

Example 11.9 – Stage 1

Energy balance (equation iii)

$$23{,}950 \times 112.8 + 43{,}691 V_2 = 25{,}562 L_1 + 41{,}623 \times 157.9$$

$$43{,}691 V_2 = 255{,}626 L_1 + 3{,}870{,}712$$

Material balance (equation i)

$$112.8 + V_2 = L_1 + 157.9$$

substituting

$$43{,}691(L_1 + 45.1) = 25{,}562 L_1 + 3{,}870{,}712$$

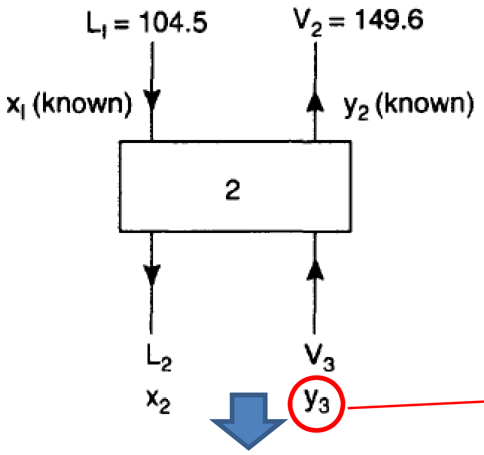
$$L_1 = 104.8$$

$$V_2 = 104.8 + 45.1 = 149.9$$

$$\frac{L_1}{V_2} = 0.70$$

Could revise calculated values for y_2 but L_1/V_2 is close enough to assumed value of *0.71*, so there would be no significant difference from first estimate.

Example 11.9 - Stage 2



As a first trial take L/V as $L_1/V_1 = 0.70$

$$\boxed{y_3 = \frac{L}{V}(x_2 - x_1) + y_2}$$

	x_2	x_1	$y_3 = 0.70(x_2 - x_1) + y_2$	y_3 Normalised
C_3	0.022	0.042	0.044	0.043
iC_4	0.222	0.277	0.256	0.251
nC_4	0.630	0.628	0.613	0.601
iC_5	0.093	0.048	0.072	0.072
nC_5	0.033	0.003	0.035	0.034
			1.020	

Estimation of stage temperature and outlet liquid composition *(x_2)*.

$T_2 = 70°C$ (use assumed value as first trial)				
	y_2	K_i	$x_2 = y_2/K_i$	x_2 Normalised
C_3	0.057	2.55	0.022	0.022
iC_4	0.292	1.30	0.226	0.222
nC_4	0.600	0.94	0.643	0.630
iC_5	0.041	0.43	0.095	0.093
nC_5	0.013	0.38	0.034	0.033
			1.020 close enough to 1.0	

Enthalpy data from Figures (*b*) and (*c*)

	$h_2(T_2 = 70°C)$			$H_3(T_3 = 75°C$ assumed)		
	x_2	h_i	$h_i x_2$	y_3	H_i	$H_i y_3$
C_3	0.022	21,900	482	0.043	34,600	1488
iC_4	0.222	25,300	5617	0.251	41,800	10,492
nC_4	0.630	27,000	17,010	0.601	44,700	26,865
iC_5	0.093	29,500	2744	0.072	53,000	3816
nC_5	0.033	31,600	1043	0.035	55,400	1939
	$h_2 = 26,896$			$H_3 = 44,600$		

Example 11.9 - Stage 2

Energy balance

$$25{,}562 \times 104.8 + 44{,}600 V_3 = 4369 \times 149.9 + 26{,}896 L_2$$

Material balance

$$104.8 + V_3 = 149.9 + L_2$$

$$L_2 = 105.0$$

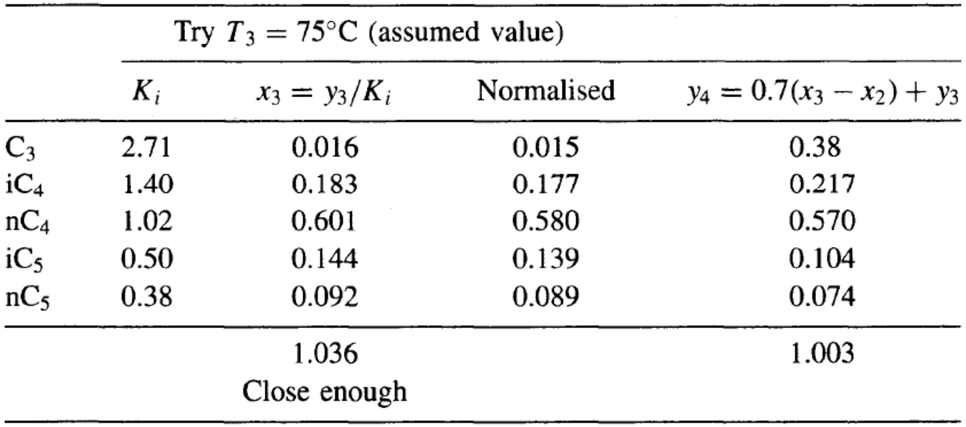
$$V_3 = 150.1$$

$$\frac{L_2}{V_3} = 0.70 \text{ checks with assumed value.}$$

Example 11.9 - Stages 3-4

As the calculated liquid and vapor flows are not changing much from stage to stage the calculation will be continued with the value of *L/V taken as constant at 0.7.*

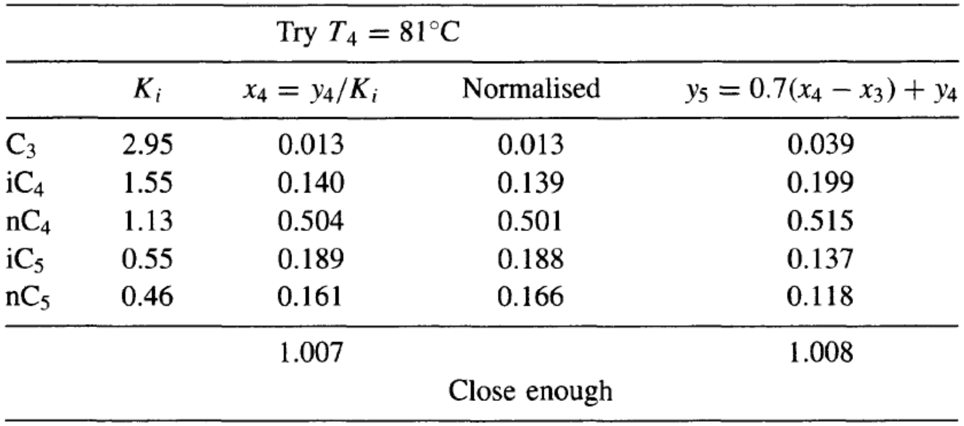
Stage 3



Try $T_3 = 75°C$ (assumed value)

	K_i	$x_3 = y_3/K_i$	Normalised	$y_4 = 0.7(x_3 - x_2) + y_3$
C_3	2.71	0.016	0.015	0.38
iC_4	1.40	0.183	0.177	0.217
nC_4	1.02	0.601	0.580	0.570
iC_5	0.50	0.144	0.139	0.104
nC_5	0.38	0.092	0.089	0.074
		1.036 Close enough		1.003

Stage 4



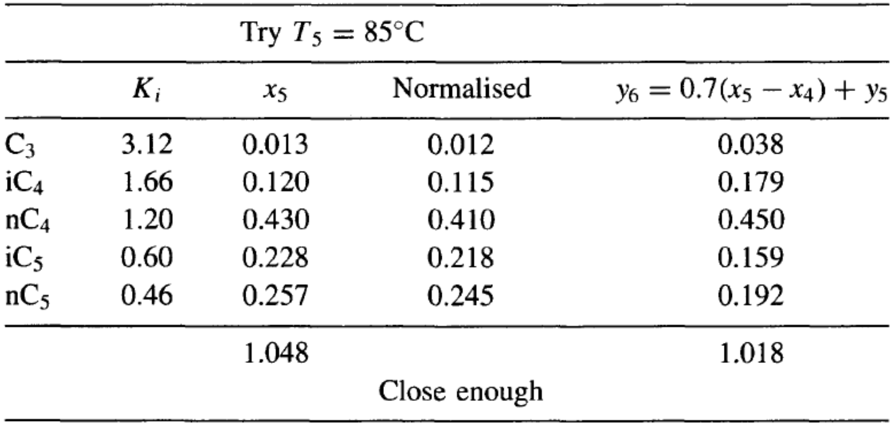
Try $T_4 = 81°C$

	K_i	$x_4 = y_4/K_i$	Normalised	$y_5 = 0.7(x_4 - x_3) + y_4$
C_3	2.95	0.013	0.013	0.039
iC_4	1.55	0.140	0.139	0.199
nC_4	1.13	0.504	0.501	0.515
iC_5	0.55	0.189	0.188	0.137
nC_5	0.46	0.161	0.166	0.118
		1.007		1.008

Close enough

Example 11.9 - Stages 5-6

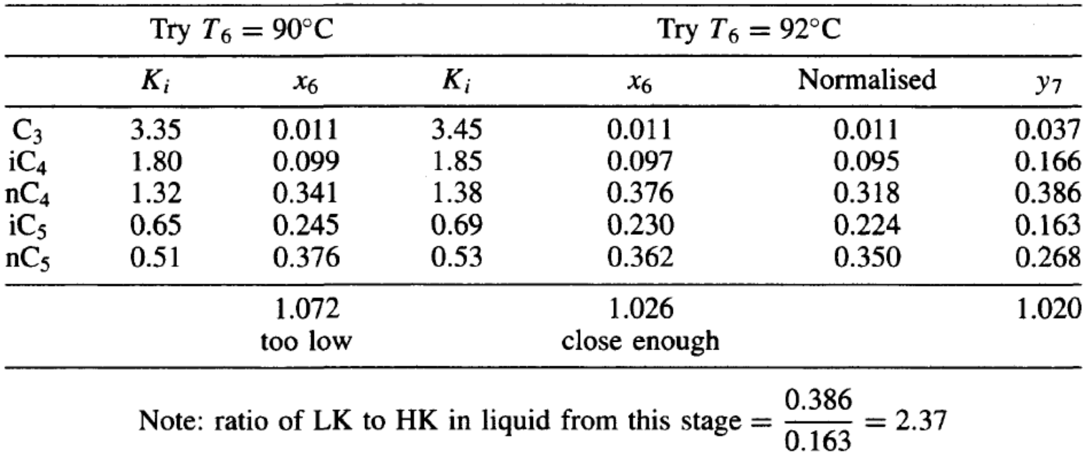
Stage 5



Try $T_5 = 85°C$

	K_i	x_5	Normalised	$y_6 = 0.7(x_5 - x_4) + y_5$
C_3	3.12	0.013	0.012	0.038
iC_4	1.66	0.120	0.115	0.179
nC_4	1.20	0.430	0.410	0.450
iC_5	0.60	0.228	0.218	0.159
nC_5	0.46	0.257	0.245	0.192
		1.048		1.018
			Close enough	

Stage 6



	Try $T_6 = 90°C$		Try $T_6 = 92°C$			
	K_i	x_6	K_i	x_6	Normalised	y_7
C_3	3.35	0.011	3.45	0.011	0.011	0.037
iC_4	1.80	0.099	1.85	0.097	0.095	0.166
nC_4	1.32	0.341	1.38	0.376	0.318	0.386
iC_5	0.65	0.245	0.69	0.230	0.224	0.163
nC_5	0.51	0.376	0.53	0.362	0.350	0.268
		1.072 too low		1.026 close enough		1.020

Note: ratio of LK to HK in liquid from this stage $= \dfrac{0.386}{0.163} = 2.37$

Example 11.9 - Stage 7

Feed composition

	x_f	x_7
C_3	0.05	0.10
iC_4	0.15	0.084
nC_4	0.25	0.254
iC_5	0.20	0.217
nC_5	0.35	0.447

Stage 7 composition

	Try $T_6 = 97°C$		
	K_i	x_7	Normalised
C_3	3.65	0.010	0.010
iC_4	1.98	0.084	0.083
nC_4	1.52	0.254	0.251
iC_5	0.75	0.217	0.214
nC_5	0.60	0.447	0.442
		1.012	

$$\text{ratio } \frac{\text{LK}}{\text{HK}} = \frac{0.251}{0.214} = 1.17$$

This is just below the ratio in the feed

$$= \frac{25}{20} = 1.25$$

So, the feed would be introduced at this stage.

But the composition of the non-key components on the plate does not match the feed composition.

So it would be necessary to adjust the assumed top composition and repeat the calculation.

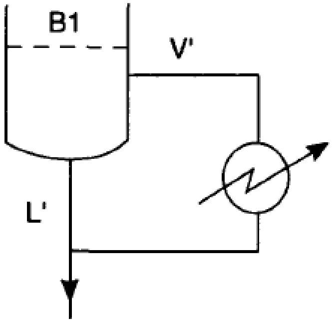
Example 11.9 – Bottom➔up calc.

To illustrate the procedure the calculation will be shown for the reboiler and bottom stage, assuming constant molar overflow.
With the feed at its boiling point and constant molar overflow the base flows can be calculated as follows:

$$V' = V_0 = 157.9$$

$$L' = L_0 + \text{FEED} = 112.8 + 100 = 212.8$$

$$\frac{V'}{L'} = \frac{157.9}{212.8} = 0.74$$



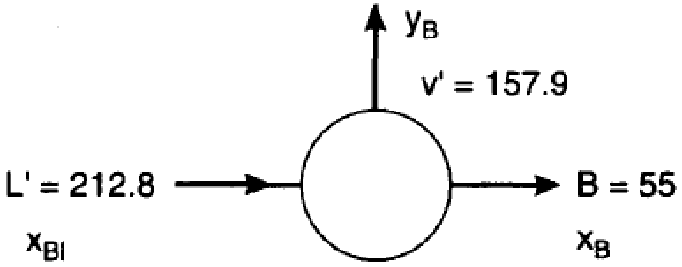
It will be necessary to estimate the concentration of the non-key components in the bottom product; as a first trial take:

C_3	iC_4	nC_4	iC_5	nC_5
0.001	0.001	0.02	0.34	0.64

Example 11.9 - reboiler

Check bubble-point estimate of 120°C

	Try 120°C			Try 118°C	
	x_B	K_i	$y_B = K_i x_B$	K_i	y_B
C_3	0.001	4.73	0.005	4.60	0.005
iC_4	0.001	2.65	0.003	2.58	0.003
nC_4	0.02	2.10	0.042	2.03	0.041
iC_5	0.34	1.10	0.374	1.06	0.360
nC_5	0.64	0.96	0.614	0.92	0.589
			1.038		0.998
			too high		close enough



Material balance

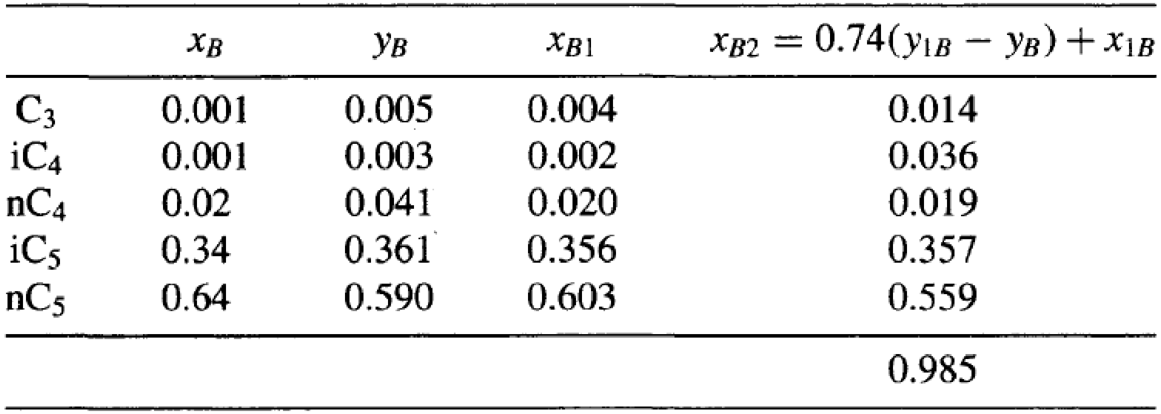
$$x_{B1}L' = y_B V' + x_B B$$

$$x_{B1} = \frac{V'}{L'} y_B + \frac{B}{L'} x_B$$

$$x_{B1} = \frac{157.9}{212.8} y_B + \frac{55}{212.8} x_B$$

$$= 0.74 y_B + 0.26 x_B$$

Example 11.9 – Stage 1 (from bottom)



	x_B	y_B	x_{B1}	$x_{B2} = 0.74(y_{1B} - y_B) + x_{1B}$
C_3	0.001	0.005	0.004	0.014
iC_4	0.001	0.003	0.002	0.036
nC_4	0.02	0.041	0.020	0.019
iC_5	0.34	0.361	0.356	0.357
nC_5	0.64	0.590	0.603	0.559
				0.985

The calculation is continued stage-by-stage up the column to the feed point (stage 7 from the top).

If the vapor composition at the feed point does not mesh with the top-down calculation, the assumed concentration of the non-keys in the bottom product is adjusted and the calculations repeated.

Rigorous computer methods

The application of digital computers has made the rigorous solution of the MESH equations a practical proposition, and computer methods for the design of multicomponent separation columns will be available in most design organizations.

A considerable amount of work has been done over the past twenty or so years to develop efficient and reliable computer-aided design procedures for distillation and other staged processes.

Several different approaches have been taken to develop programs that are efficient in the use of computer time, and suitable for the full range of multicomponent separation processes that are used in the process industries.

A design group will use those methods that are best suited to the processes that it normally handles.

In this section a brief outline will be given of the methods that have been developed.

Rigorous solution procedure: basic steps

The basic steps **in any rigorous solution procedure will be:**

1. Specification of the problem; complete specification is essential for computer methods.

2. Selection of values for the iteration variables; for example, estimated stage temperatures, and liquid and vapour flows (the column temperature and flow profiles).

3. A calculation procedure for the solution of the stage equations.

4. A procedure for the selection of new values for the iteration variables for each set of trial calculations.

5. A procedure to test for convergence; to check if a satisfactory solution has been achieved.

Available computer methods

It is convenient to consider the methods available under the following four headings:

1. Lewis-Matheson method.
2. Thiele-Geddes method.
3. Relaxation methods.
4. Linear algebra methods.

Rating and design methods

With the exception of the Lewis-Matheson method, all the methods listed above require the specification of the number of stages below and above the feed point.

They are therefore not directly applicable to design: where the designer wants to determine the number of stages required for a specified separation.

They are strictly what are referred to as "*rating methods*"; used to determine the performance of existing, or specified, columns.

Given the number of stages they can be used to determine product compositions.

Iterative procedures are necessary to apply rating methods to the design of new columns.

An initial estimate of the number of stages can be made using short-cut methods and the programs used to calculate the product compositions; repeating the calculations with revised estimates till a satisfactory design is obtained.

Lewis-Matheson method

The method proposed by **Lewis and Matheson** (1932) is essentially the application of the Lewis-Sorel method (Section 11.5.1) to the solution of multicomponent problems.

Constant molar overflow is assumed and the material balance and equilibrium relationship equations are solved stage by stage starting at the top or bottom of the column (refer to the Example 11.9 C&R Vol. VI).

Lewis-Matheson method

To define a problem for the Lewis-Matheson method the following variables must be specified, or determined from other specified variables:

1. Feed composition, flow rate and condition.
2. Distribution of the key components.
3. One product flow.
4. Reflux ratio.
5. Column pressure.
6. Assumed values for the distribution of the non-key components.

Lewis-Matheson method

The usual procedure is to start the calculation at the top and bottom of the column and proceed toward the feed point.

The initial estimates of the component distributions in the products are then revised and the calculations repeated until the compositions calculated from the top and bottom starts mesh, and match the feed at the feed point.

Efficient procedures for adjusting the compositions to achieve a satisfactory mesh at the feed point are given by Hengstebeck (1961).

Good descriptions of the Lewis-Matheson method, with examples of manual calculations, are also given in the books by Oliver (1966) and Smith (1963); a simple example is given in C&R Vol. 2, Chapter 11.

Lewis-Matheson method

In some computer applications of the method, where the assumption of constant molar overflow is not made, it is convenient to start the calculations by assuming flow and temperature profiles.

The stage component compositions can then be readily determined and used to revise the profiles for the next iteration.

In general, the Lewis-Matheson method has not been found to be an efficient procedure for computer solutions, other than for relatively straightforward problems.

It is not suitable for problems involving multiple feeds, and side-streams, or where more than one column is needed.

The method is suitable for interactive programs run on programmable calculators and Personal Computers.

As the calculations are carried out one stage at a time, only a relatively small computer memory is needed.

Lewis-Matheson method: details

(C&R Vol. 2)

In a binary system, the relationship between the composition of the vapour y_A and of the liquid x_A in equilibrium may also be expressed in a way, which is particularly useful in distillation calculations.

If the ratio of the partial pressure to the mole fraction in the liquid is defined as the ***volatility***, then:

volatility of A $= P_A/x_A$ and **volatility of B** $= P_B/x_B$

The ratio of these two volatilities is known as the ***relative volatility*** α given by (ideal systems):

$$\alpha_{AB} = P_A x_B / P_B x_A = P^\circ_A / P^\circ_B$$

Being P the total pressure, substituting Py_A *for* P_A, *and* Py_B *for* P_B:

$$\alpha_{AB} = Py_A x_B / Py_B x_A = y_A x_B / y_B x_A$$

$$y_A/y_B = \alpha_{AB}\, x_A/x_B$$

The extension of this concept to multicomponent (ideal) systems with components A, B, C, D, … leads to:

$$y_A/y_B = \alpha_{AB}\, x_A/x_B; \quad y_C/y_B = \alpha_{CB}\, x_C/x_B; \quad y_D/y_B = \alpha_{DB}\, x_D/x_B; \quad \ldots$$

Lewis-Matheson method: details

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

$$y_A + y_B + y_C + y_D + \ldots = 1 \qquad (1)$$

$$y_A/y_B + y_B/y_B + y_C/y_B + y_D/y_B + \ldots = 1/y_B \qquad (2)$$

as $$y_i/y_B = \alpha_{iB}\, x_i/y_B \qquad (3)$$

subst.: $$\alpha_{AB}\, x_A/x_B + \alpha_{BB}\, x_B/x_B + \alpha_{CB}\, x_C/x_B + \alpha_{DB}\, x_D/x_B + \ldots = 1/y_B \qquad (4)$$

$$\sum_{i=1}^{N} \alpha_{iB} x_i = x_B/y_B \qquad (5)$$

expliciting: $$\boxed{y_B = x_B \Big/ \sum_{i=1}^{N} \alpha_{iB} x_i} \qquad (6)$$

similarly: $$\boxed{y_A = x_A \Big/ \sum_{i=1}^{N} \alpha_{iA} x_i \;;\quad y_C = x_C \Big/ \sum_{i=1}^{N} \alpha_{iC} x_i \;;\quad y_D = x_D \Big/ \sum_{i=1}^{N} \alpha_{iD} x_i \;;} \qquad (7)$$

Lewis-Matheson method: example

A mixture of ortho, meta, and para-mononitrotoluenes containing

60 % mol	ortho-mononitrotoluene
4% mol	meta-mononitrotoluene
36% mol	para-mononitrotoluene

Is to be continuously distilled to give a top product of **x_{do}=98%** mol ortho, and the bottom is to contain **x_{wo}=12.5%** mol ortho.

The mixture is to be distilled at a bottom temperature of **T_B=410K** requiring a pressure in the boiler of about **P=6.0 kN/m^2**.

If a reflux ratio of **R=5** is used, **how many ideal plates** will be required and what will be the approximate compositions of the product streams?

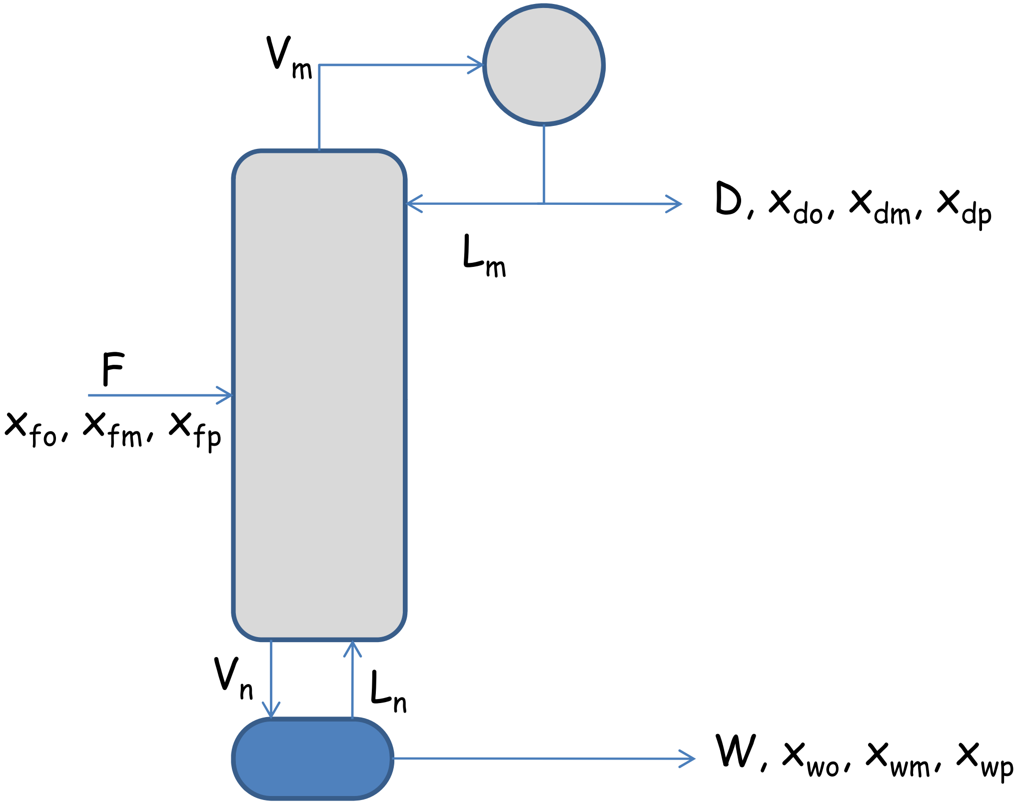
The **volatility** of ortho relative to the para isomer may be taken as **α_{op}=1.70** and of the meta as **α_{om}=1.16** over the temperature range of 380–415 K.

Column scheme

Specs:

$x_{fo},$ = 0.4
$x_{fm},$ =0.04
x_{fp} =0.36
x_{do} =0.98
x_{wo} =0.125

P = 6 kN/m^2
T_B = 410 K
R =L_m/D = 5



Lewis-Matheson method: example

Solution

As a first estimate, it is supposed that the distillate contains 0.6% meta and 1.4% para.

A material balance then gives the composition of the bottoms.

For 100 kmol of feed with D *and* W *kmol of product and bottoms, respectively and* x_{do} *and* x_{wo} the mole fraction of the ortho in the distillate and bottoms, then an overall material balance gives:

$$100 = D + W$$

An ortho balance gives:

$$60 = Dx_{do} + Wx_{wo}$$

and:

$$60 = (100 - W)0.98 + 0.125W$$

from which:

$$D = 55.56 \text{ kmol} \quad \text{and} \quad W = 44.44. \text{ kmol}$$

Lewis-Matheson method: example

The compositions and amounts of the streams are then be obtained by the overall mass balance as follows:

Component	Feed		Distillate		Bottoms	
	(kmol)	(mole per cent)	(kmol)	(mole per cent)	(kmol)	(mole per cent)
Ortho o	60	60	54.44	98.0	5.56	12.5
Meta m	4	4	0.33	0.6	3.67	8.3
Para p	36	36	0.79	1.4	35.21	79.2
	100	100	55.56	100	44.44	100

Equations of operating lines

The liquid and vapour streams in the column are obtained (assuming that McCabe and Thiele conditions hold) as follows:

Above the feed-point:

Liquid downflow $L_n = 5D = 277.8\ \text{kmol}$

Vapour up $V_n = 6D = 333.4\ \text{kmol}$

Lewis-Matheson method: example

Equations of operating lines

Below the feed-point, assuming the feed is liquid at its boiling point then:

Liquid downflow **$L_m = L_n + F = (277.8 + 100) = 377.8$ kmol**

Vapour up **$V_m = L_m - W = (377.8 - 44.44) = 333.4$ kmol**

The equations for the **operating lines** may then be written as:

below the feed plate:

$$y_m = \frac{L_m}{V_m}x_{m+1} - \frac{W}{V_m}x_w \qquad (8)$$

ortho:

$$y_{mo} = \left(\frac{377.8}{333.4}\right)x_{m+1} - \left(\frac{44.44}{333.4}\right)x_w$$

meta:
para:

$$\left.\begin{aligned} &= 1.133x_{m+1} - 0.0166 \\ y_{mm} &= 1.133x_{m+1} - 0.011 \\ y_{mp} &= 1.133x_{m+1} - 0.105 \end{aligned}\right\} \qquad \text{(i)}$$

Lewis-Matheson method: example

Equations of operating lines

Above the feed plate:

$$y_n = \frac{L_n}{V_n} x_{n+1} + \frac{D}{V_n} x_d \qquad (9)$$

$$\text{ortho:}\quad y_{no} = \left(\frac{277.8}{333.4}\right) x_{n+1} + \left(\frac{55.56}{333.4}\right) 0.98$$

$$\left.\begin{aligned} &= 0.833x_{n+1} + 0.163 \\ \text{meta:}\quad y_{nm} &= 0.833_{x+1} + 0.001 \\ \text{para:}\quad y_{np} &= 0.833x_{n+1} + 0.002 \end{aligned}\right\} \qquad \text{(ii)}$$

Lewis-Matheson method: example

Composition of liquid on first plate

The temperature of distillation is fixed by safety considerations at 410 K and, from a knowledge of the vapour pressures of the three components, the pressure in the still is found to be about 6 kN/m^2.

The composition of the vapour in the still is found from the relation

$$y_{so} = \alpha_o x_{so} / \Sigma \alpha_s x_s \qquad \text{(eqns. 6-7)}$$

The liquid composition on the first plate is then found from equation (i). As example and for ortho:

$$0.191 = (1.133x_1 - 0.0166) \rightarrow x_1 = 0.183$$

The values of the other compositions are found in this way (see following tables).

Lewis-Matheson method: example

Using the relation $v_{mo} = \alpha_o x_{mo} / \Sigma \alpha x_m$:

Plate compositions below the feed plate

Component	x_s	αx_s	y_s	x_1	αx_1	y_1	x_2
o	0.125	0.211	0.191	0.183	0.308	0.270	0.253
m	0.083	0.096	0.088	0.088	0.102	0.090	0.089
p	0.792	0.792	0.721	0.729	0.729	0.640	0.658
	1	1.099	1	1	1.139	1	1

	αx_2	y_2	x_3	αx_3	y_3	x_4	αx_4
o	0.430	0.357	0.330	0.561	0.450	0.411	0.698
m	0.103	0.086	0.086	0.100	0.080	0.080	0.093
p	0.658	0.557	0.584	0.584	0.470	0.509	0.509
	1.191	1	1	1.245	1	1	1.300

Component	y_4	x_5	αx_5	y_5	x_6	αx_6	y_6
o	0.537	0.488	0.830	0.613	0.556	0.944	0.674
m	0.071	0.072	0.083	0.061	0.063	0.073	0.052
p	0.392	0.440	0.440	0.326	0.381	0.381	0.274
	1	1	1.353	1	1	1.398	1

	x_-
o	0.609
m	0.055
p	0.336
	1

Lewis-Matheson method: example

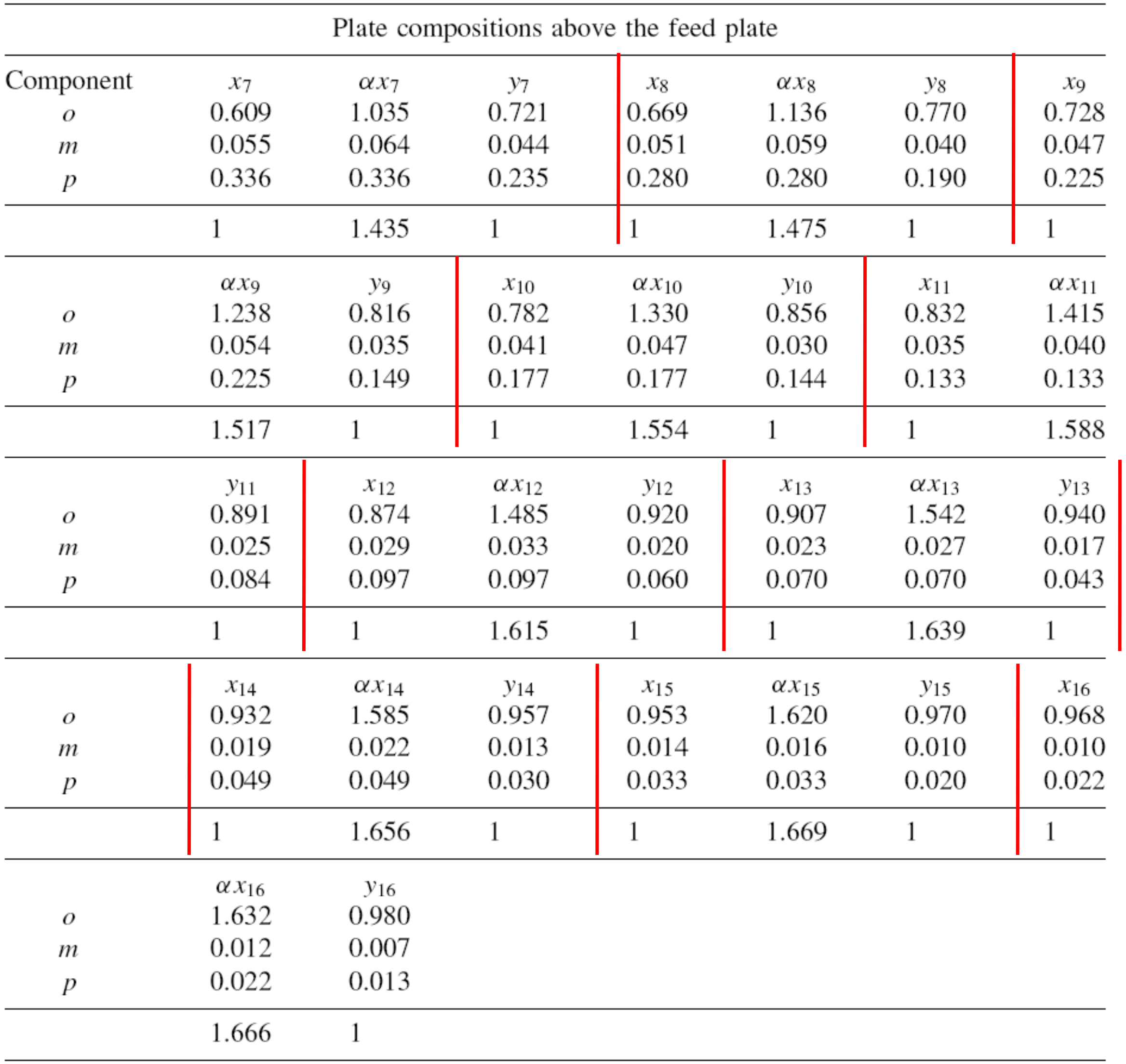


Plate compositions above the feed plate

Component	x_7	αx_7	y_7	x_8	αx_8	y_8	x_9
o	0.609	1.035	0.721	0.669	1.136	0.770	0.728
m	0.055	0.064	0.044	0.051	0.059	0.040	0.047
p	0.336	0.336	0.235	0.280	0.280	0.190	0.225
	1	1.435	1	1	1.475	1	1

	αx_9	y_9	x_{10}	αx_{10}	y_{10}	x_{11}	αx_{11}
o	1.238	0.816	0.782	1.330	0.856	0.832	1.415
m	0.054	0.035	0.041	0.047	0.030	0.035	0.040
p	0.225	0.149	0.177	0.177	0.144	0.133	0.133
	1.517	1	1	1.554	1	1	1.588

	y_{11}	x_{12}	αx_{12}	y_{12}	x_{13}	αx_{13}	y_{13}
o	0.891	0.874	1.485	0.920	0.907	1.542	0.940
m	0.025	0.029	0.033	0.020	0.023	0.027	0.017
p	0.084	0.097	0.097	0.060	0.070	0.070	0.043
	1	1	1.615	1	1	1.639	1

	x_{14}	αx_{14}	y_{14}	x_{15}	αx_{15}	y_{15}	x_{16}
o	0.932	1.585	0.957	0.953	1.620	0.970	0.968
m	0.019	0.022	0.013	0.014	0.016	0.010	0.010
p	0.049	0.049	0.030	0.033	0.033	0.020	0.022
	1	1.656	1	1	1.669	1	1

	αx_{16}	y_{16}
o	1.632	0.980
m	0.012	0.007
p	0.022	0.013
	1.666	1

Lewis-Matheson method: example

The liquid on plate 7 has a composition with the ratio of the concentrations of ortho and para about that in the feed, and the feed will therefore be introduced on this plate.

Above this plate the same method is used but the operating equations are equation (ii).

The vapour from the 16th plate has the required concentration of the ortho isomer, and the values the meta and para are sufficiently near to take this as showing that 16 ideal plates will be required.

Thiele-Geddes method

Like the Lewis-Matheson method, the original method of **Thiele and Geddes** (1933) was developed for manual calculation. It has subsequently been adapted by many workers for computer applications.

The variables specified in the basic method, or that must be derived from other specified variables, are:

1. Reflux temperature.
2. Reflux flow rate.
3. Distillate rate.
4. Feed flows and condition.
5. Column pressure.
6. Number of equilibrium stages above and below the feed point.

Thiele-Geddes procedure

The method starts with an assumption of the column temperature and flow profiles.

The stage equations are then solved to determine the stage component compositions and the results used to revise the temperature profiles for subsequent trial calculations.

Efficient convergence procedures have been developed for the Thiele-Geddes method.

The so-called "theta method", described by Lyster *etal. (1959) and Holland (1963), is recommended.*

The Thiele-Geddes method can be used for the solution of complex distillation problems, and for other multi-component separation processes.

Relaxation methods

With the exception of this method, all the methods described solve the stage equations for the steady-state design conditions.

In an operating column other conditions will exist at start-up, and the column will approach the "design" steady-state conditions after a period of time.

The stage material balance equations can be written in a finite difference form, and procedures for the solution of these equations will model the unsteady-state behaviour of the column.

Relaxation methods are not competitive with the "steady-state" methods in the use of computer time, because of slow convergence. However, because they model the actual operation of the column, convergence should be achieved for all practical problems.

The method has the potential of development for the study of the transient behaviour of column designs, and for the analysis and design of batch distillation columns.

Linear algebra methods

The Lewis-Matheson and Thiele-Geddes methods use a stage-by-stage procedure to solve the equations relating the component compositions to the column temperature and flow profiles.

However, the development of high-speed digital computers with large memories makes possible the simultaneous solution of the complete set of MESH equations that describe the stage compositions throughout the column.

If the equilibrium relationships and flow-rates are known (or assumed) the set of material balance equations for each component is linear in the component compositions.

Whit the aim of a numerical method these equations are solved simultaneously and the results used to provide improved estimates of the temperature and flow profiles.

Linear algebra methods

The set of equations can be expressed in matrix form and solved using the standard inversion routines available in modern computer systems. Convergence can usually be achieved after a few iterations.

It is possible to include and couple to the distillation program, some thermodynamic method for estimation of the liquid-vapour relationships (activity coefficients) as the UNIFAC method (see Chapter 8, Section 16.3).

This makes the program particularly useful for the design of columns for new processes, where experimental data for the equilibrium relationships are unlikely to be available.