5. **Multicomponent Distillation – General Considerations:**

- Calculation of stages and reflux is more complex than binary mixtures.
- The separation between the top and bottom products is specified by setting limits on two "key" components, between which it is desired to make the separation.
- Normal procedure used in calculation involves solution of MESH equations stage-by-stage, from top and bottom of the column towards the feed point.
- For calculations to be exact, the compositions obtained from both the bottom-up and top-down calculations must mesh at the feed point and match the feed composition.
- For a completely rigorous solution the assumed top and bottom compositions must be adjusted and the calculations repeated until a satisfactory mesh at the feed point is obtained.
- The greater the number of components, the more difficult is the problem.
- Various "short-cut" methods were developed to simplify the task of designing multicomponent columns.
- 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).
  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 and the Erbar-Maddox correlation.

5.1 **Selection of Key Components:**

- Before starting the designing part, we must select the key components between which the separation is desired.
- *Light Key* will be the component that is desired to be *taken as the bottom product*.
- *Heavy Key* will be the component that is desired to be *taken as the top product*.
- 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; usually the keys are found to be adjacent.
- The "non-key" components 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.
5.2 **Number and Sequencing of Columns:**
- Number of columns required in a multicomponent distillation usually depends on number of components involved in the entering feed.
- For $N$ number of components, there are $(N-1)$ columns required for separation of each component. 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.
- The order in which the components are separated will determine the capital and operating costs.
- To determine optimum sequence for a multicomponent separation a general guideline is given as follows:
  - Remove the components one at a time; as in the benzene-toluene-xylene example,
  - Remove any components that are present in large excess early in the sequence.
  - With difficult separations, involving close boiling components, postpone the most difficult separation to late in the sequence.

6. **Multicomponent Distillation – Short Cut Methods for Stage and Reflux Calculations:**

6.1 **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 and then the number of stages can be calculated using McCabe-Thiele Method or by any other method that are used for binary 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:

$$v_{n+1,i} = l_{n,i} + d_i$$  \hspace{1cm} (11.42)
for the stripping section:

\[ l'_{n+1,i} = v'_{n,i} + b_i \]  \hspace{1cm} (11.44)

\[ v'_{n,i} = K_{n,i} \frac{V'}{L'} l'_{n,i} \]  \hspace{1cm} (11.45)

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 to be constant.

- 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 makes use of the fact 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.

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.

For rectifying section

\[ L_e = L - \Sigma l_i \]  \hspace{1cm} (11.46)

\[ V_e = V - \Sigma v_i \]  \hspace{1cm} (11.47)

For stripping section

\[ L'_e = L' - \Sigma l'_i \]  \hspace{1cm} (11.48)

\[ V'_e = V' - \Sigma v'_i \]  \hspace{1cm} (11.49)
where $V_e$ and $L_e$ are the estimated flow rates of the combined keys, $l_i$ and $v_i$ are the limiting liquid and vapour rates of components lighter than the keys in the rectifying section, $l'_i$ and $v'_i$ are the limiting liquid and vapour rates of components heavier than the keys in the rectifying section,

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

\[
\begin{align*}
l_i &= \frac{d_i}{\alpha_i - 1} \quad \text{(11.50)} \\
v_i &= l_i + d_i \quad \text{(11.51)} \\
v'_i &= \frac{\alpha_i b_i}{\alpha_{LK} - \alpha_i} \quad \text{(11.52)} \\
l'_i &= v'_i + b_i \quad \text{(11.53)}
\end{align*}
\]

where $\alpha_i = \text{relative volatility of component } i, \text{ relative to the heavy key (HK)},$ $\alpha_{LK} = \text{relative volatility of the light key (LK), relative to the heavy key}.$

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 vapour and liquid concentrations of the light key.

**Example-1:** 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 of 2.5. The feed is at its boiling point.
#### Solution:

Relative volatilities are given by the equation:

\[
\alpha_i = \frac{K_i}{K_{HK}}
\]

Equilibrium constants were taken from the Depriester charts (Figure 8.3, pp. no. 346) therefore, relative volatilities –

<table>
<thead>
<tr>
<th></th>
<th>Top</th>
<th>Bottom</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temp. °C</td>
<td>65</td>
<td>120</td>
<td></td>
</tr>
<tr>
<td>C₃</td>
<td>5.5</td>
<td>4.5</td>
<td>5.0</td>
</tr>
<tr>
<td>iC₄</td>
<td>2.7</td>
<td>2.5</td>
<td>2.6</td>
</tr>
<tr>
<td>(LK) nC₄</td>
<td>2.1</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>(HK) iC₅</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>nC₅</td>
<td>0.84</td>
<td>0.85</td>
<td>0.85</td>
</tr>
</tbody>
</table>

Calculations of non-key flows form equations 11.50, 11.51, 11.52, 11.53

<table>
<thead>
<tr>
<th>αᵢ</th>
<th>dᵢ</th>
<th>( l_i = d_i/(α_i - 1) )</th>
<th>( v_i = l_i + d_i )</th>
</tr>
</thead>
<tbody>
<tr>
<td>C₃</td>
<td>5</td>
<td>1.3</td>
<td>6.3</td>
</tr>
<tr>
<td>iC₄</td>
<td>2.6</td>
<td>15</td>
<td>9.4</td>
</tr>
</tbody>
</table>

\[ \Sigma l_i = 10.7 \quad \Sigma v_i = 30.7 \]

\[
\alpha_i \quad \quad b_i \quad \quad v_i' = \alpha_i b_i/\alpha_i \\
\quad \quad v_i'' = v_i' + b_i
\]

| nC₅ | 0.85| 35 | 25.9 | 60.9 |

\[ \Sigma v_i' = 25.9 \quad \Sigma l_i' = 60.9 \]

Flow of combined keys:

\[ L_e = 2.5 \times 45 - 10.7 = 101.8 \quad \quad (11.46) \]
\[ V_e = (2.5 + 1)45 - 30.7 = 126.8 \quad \quad (11.47) \]
\[ V_e' = (2.5 + 1)45 - 25.9 = 131.6 \quad \quad (11.49) \]
\[ L_e' = (2.5 + 1)45 + 55 - 60.9 = 151.6 \quad \quad (11.48) \]
Slope of top operating line

\[
\frac{L_e}{V_e} = \frac{101.8}{126.8} = 0.8
\]

Slope of bottom operating line

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

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

\[
x_d = \frac{24}{24 + 1} = 0.96
\]

\[
x_f = \frac{25}{25 + 20} = 0.56
\]

\[
y = \frac{2x}{1 + (2 - 1)x} = \frac{2x}{1 + x}
\]  \hspace{1cm} (11.23)

<table>
<thead>
<tr>
<th>x</th>
<th>0</th>
<th>0.20</th>
<th>0.40</th>
<th>0.60</th>
<th>0.80</th>
<th>1.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>y</td>
<td>0</td>
<td>0.33</td>
<td>0.57</td>
<td>0.75</td>
<td>0.89</td>
<td>1.0</td>
</tr>
</tbody>
</table>

The McCabe-Thiele diagram is shown in Figure 11.10. Twelve stages required; feed on seventh from base.

![McCabe-Thiele Diagram for the solved example](image)
7. **Empirical Correlations:**

- The two most frequently used empirical methods for estimating the stage requirements for multicomponent distillations are the correlations given 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).
- The Erbar-Maddox correlation is now generally considered to give more reliable predictions. It 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.
- The number of stages at total reflux and the minimum reflux ratio are needed in order to use Erbar-Maddox correlation.

![Figure 11.11: Erbar-Maddox Correlation](image)

Figure 11.11: Erbar-Maddox Correlation
**Minimum Number of Stages (Fenske Equation):**

Fenske equation is used to calculate the minimum number of stages at total reflux. The equation is given as –

\[
N_m = \frac{\log \left[ \frac{x_{LK}}{x_{HK}} \right]_d}{\log \alpha_{LK}} \quad \text{------ (11.58)}
\]

- where \( \alpha_{LK} \) 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.
- If there is 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, therefore, this method will only give best results only if relative volatility is found to almost constant.

**Minimum Reflux Ratio:**

Colburn (1941) and Underwood (1948) have derived equations for estimating the minimum reflux ratio for multicomponent distillations. Underwood equation is most widely used and can be stated in the form:

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

where \( \alpha_i \) = 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, \( \theta \) is the root of the following equation:

\[
\sum \frac{\alpha_i x_{i,f}}{\alpha_i - \theta} = 1 - q 
\]

\text{------ (11.61)}
where \( x_{if} \) = the concentration of component \( i \) in the feed, and \( q \) depends on the condition of the feed.

**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):

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

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,
\( x_{f,HK} \) = concentration of the heavy key in the feed,
\( x_{f,LK} \) = concentration of the light key in the feed,
\( x_{d,HK} \) = concentration of the heavy key in the top product,
\( x_{b,LK} \) = concentration of the light key if in the bottom product.

**Distribution of Non-Key Components:**
Hengstebeck and Geddes (1958) have shown that the Fenske equation can be written in the form:

\[
\log \left( \frac{d_i}{b_i} \right) = A + C \log \alpha_i \tag{11.63}
\]

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-2:** Use the Geddes-Hengstebeck method to check the component distributions for the separation specified in Example-1.

<table>
<thead>
<tr>
<th>Component</th>
<th>( \alpha_i )</th>
<th>Feed ( (f_i) )</th>
<th>Distillate ( (d_i) )</th>
<th>Bottoms ( (b_i) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>C(_3)</td>
<td>5</td>
<td>5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>iC(_4)</td>
<td>2.6</td>
<td>15</td>
<td></td>
<td></td>
</tr>
<tr>
<td>nC(_4)  (LK)</td>
<td>2.0</td>
<td>25</td>
<td>24</td>
<td>1</td>
</tr>
<tr>
<td>iC(_5)  (HK)</td>
<td>1.0</td>
<td>20</td>
<td>1</td>
<td>19</td>
</tr>
<tr>
<td>nC(_5)</td>
<td>0.85</td>
<td>35</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Solution:**
Relative volatilities will be the same as calculated in Example-1.

For calculating split in components, following expression is used –

\[
\frac{d_i}{b_i} = \alpha_i^{N_m} \left[ \frac{d_r}{b_r} \right]
\]  

For the light key,

\[
\frac{d_i}{b_i} = \frac{24}{1} = 24
\]

For the heavy key,

\[
\frac{d_i}{b_i} = \frac{1}{19} = 0.053
\]

These are plotted in figure 11.12. The distribution of the non-keys are read from Figure 11.12 at the appropriate relative volatility and the component flows calculated from the following equations:

Overall column material balance –

\[
f_i = d_i + b_i
\]

which on some manipulation gives:

\[
d_i = \frac{f_i}{\left( \frac{b_i}{d_i} + 1 \right)}
\]

\[
b_i = \frac{f_i}{\left( \frac{d_i}{b_i} + 1 \right)}
\]
on calculation, the calculated value comes out to be –

<table>
<thead>
<tr>
<th>$\alpha_i$</th>
<th>$f_i$</th>
<th>$d_i/b_i$</th>
<th>$d_i$</th>
<th>$b_i$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$C_3$</td>
<td>5</td>
<td>40,000</td>
<td>5</td>
<td>0</td>
</tr>
<tr>
<td>i$C_4$</td>
<td>2.6</td>
<td>150</td>
<td>14.9</td>
<td>0.1</td>
</tr>
<tr>
<td>n$C_4$</td>
<td>2.0</td>
<td>21</td>
<td>24</td>
<td>1</td>
</tr>
<tr>
<td>i$C_5$</td>
<td>1.0</td>
<td>0.053</td>
<td>1</td>
<td>19</td>
</tr>
<tr>
<td>n$C_5$</td>
<td>0.85</td>
<td>0.011</td>
<td>0.4</td>
<td>34.6</td>
</tr>
</tbody>
</table>

which is closed enough to the assumed value.