

from x_f to x_d , as shown by the line AK in Figure 11.17. Further reduction in R will eventually bring the operating line to AE, where an infinite number of stages is needed to pass from x_d to x_f . This arises from the fact that under these conditions the steps become very close together at liquid compositions near to x_f , and no enrichment occurs from the feed plate to the plate above. These conditions are known as *minimum reflux*, and the reflux ratio is denoted by R_m . Any small increase in R beyond R_m will give a workable system, although a large number of plates will be required. It is important to note that any line such as AG, which is equivalent to a smaller value of R than R_m , represents an impossible condition, since it is impossible to pass beyond point G towards x_f . Two important deductions may be made. Firstly that the minimum number of plates is required for a given separation at conditions of total reflux, and secondly that there is a minimum reflux ratio below which it is impossible to obtain the desired enrichment, however many plates are used.

Calculation of the minimum reflux ratio

Figure 11.17 represents conditions where the q -line is vertical, and the point E lies on the equilibrium curve and has co-ordinates (x_f, y_f) . The slope of the line AE is then given by:

$$\left(\frac{R_m}{R_m + 1} \right) = \left(\frac{x_d - y_f}{x_d - x_f} \right)$$

or:

$$R_m = \left(\frac{x_d - y_f}{y_f - x_f} \right) \quad (11.49)$$

If the q -line is horizontal as shown in Figure 11.18, the enrichment line for minimum reflux is given by AC, where C has coordinates (x_c, y_c) . Thus:

$$\left(\frac{R_m}{R_m + 1} \right) = \left(\frac{x_d - y_c}{x_d - x_c} \right)$$

or, since $y_c = x_f$:

$$R_m = \left(\frac{x_d - y_c}{y_c - x_c} \right) = \left(\frac{x_d - x_f}{x_f - x_c} \right) \quad (11.50)$$

Underwood and Fenske equations

For ideal mixtures, or where over the concentration range concerned the relative volatility may be taken as constant, R_m may be obtained analytically from the physical properties of the system as discussed by UNDERWOOD⁽²⁸⁾. Thus, if x_{nA} and x_{nB} are the mole fractions of two components **A** and **B** in the liquid on any plate n , then a material balance over the top portion of the column above plate n gives:

$$V_n y_{nA} = L_n x_{(n+1)A} + D x_{dA} \quad (11.51)$$

and:

$$V_n y_{nB} = L_n x_{(n+1)B} + D x_{dB} \quad (11.52)$$

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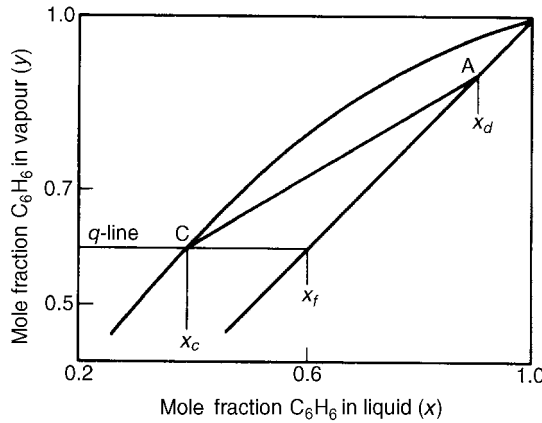


Figure 11.18. Minimum reflux ratio with feed as saturated vapour

Under conditions of minimum reflux, a column has to have an infinite number of plates, or alternatively the composition on plate n is equal to that on plate $n + 1$. Dividing equation 11.51 by equation 11.52 and using the relations $x_{(n+1)A} = x_{nA}$ and $x_{(n+1)B} = x_{nB}$, then:

$$\frac{\alpha x_{nA}}{x_{nB}} = \frac{y_{nA}}{y_{nB}} = \frac{L_n x_{nA} + D x_{dA}}{L_n x_{nB} + D x_{dB}}$$

Thus:

$$R_m = \left(\frac{L_n}{D}\right)_{\min} = \frac{1}{\alpha - 1} \left[\frac{x_{dA}}{x_{nA}} - \alpha \left(\frac{x_{dB}}{x_{nB}}\right) \right] \tag{11.53}$$

In this analysis, α is taken as the volatility of **A** relative to **B**. There is, in general, therefore a different value of R_m for each plate. In order to produce any separation of the feed, the minimum relevant value of R_m is that for the feed plate, so that the minimum reflux ratio for the desired separation is given by:

$$R_m = \frac{1}{(\alpha - 1)} \left[\frac{x_{dA}}{x_{fA}} - \alpha \frac{x_{dB}}{x_{fB}} \right] \tag{11.54}$$

For a binary system, this becomes:

$$R_m = \frac{1}{(\alpha - 1)} \left[\frac{x_{dA}}{x_{fA}} - \alpha \frac{(1 - x_{dA})}{(1 - x_{fA})} \right] \tag{11.55}$$

This relation may be obtained by putting $y = \alpha x/[1 + (\alpha - 1)x]$ from equation 11.15, in equation 11.49 to give:

$$R_m = \frac{x_d - \left(\frac{\alpha x_f}{1 + (\alpha - 1)x_f}\right)}{\left(\frac{\alpha x_f}{1 + (\alpha - 1)x_f}\right) - x_f} = \frac{1}{(\alpha - 1)} \left[\frac{x_d}{x_f} - \frac{\alpha(1 - x_d)}{(1 - x_f)} \right] \tag{11.56}$$

The number of plates at total reflux. Fenske's method

For conditions in which the relative volatility is constant, FENSKE⁽²⁹⁾ derived an equation for calculating the required number of plates for a desired separation. Since no product is withdrawn from the still, the equations of the two operating lines become:

$$y_n = x_{n+1} \quad \text{and} \quad y_m = x_{m+1} \quad (11.57)$$

If for two components **A** and **B**, the concentrations in the still are x_{sA} and x_{sB} , then the composition on the first plate is given by:

$$\left(\frac{x_A}{x_B}\right)_1 = \left(\frac{y_A}{y_B}\right)_s = \alpha_s \left(\frac{x_A}{x_B}\right)_s$$

where the subscript outside the bracket indicates the plate, and s the still.

For plate 2:
$$\left(\frac{x_A}{x_B}\right)_2 = \left(\frac{y_A}{y_B}\right)_1 = \alpha_1 \left(\frac{x_A}{x_B}\right)_1 = \alpha_1 \alpha_s \left(\frac{x_A}{x_B}\right)_s$$

and for plate n :

$$\left(\frac{x_A}{x_B}\right)_n = \left(\frac{y_A}{y_B}\right)_{n-1} = \alpha_1 \alpha_2 \alpha_3 \dots \alpha_{n-1} \alpha_s \left(\frac{x_A}{x_B}\right)_s$$

If an average value of α is used, then:

$$\left(\frac{x_A}{x_B}\right)_n = \alpha_{av}^n \left(\frac{x_A}{x_B}\right)_s$$

In most cases total condensation occurs in the condenser, so that:

$$\begin{aligned} \left(\frac{x_A}{x_B}\right)_d &= \left(\frac{y_A}{y_B}\right)_n = \alpha_n \left(\frac{x_A}{x_B}\right)_n = \alpha_{av}^{n+1} \left(\frac{x_A}{x_B}\right)_s \\ n + 1 &= \frac{\log \left[\left(\frac{x_A}{x_B}\right)_d \left(\frac{x_B}{x_A}\right)_s \right]}{\log \alpha_{av}} \end{aligned} \quad (11.58)$$

and n is the required number of theoretical plates in the column.

It is important to note that, in this derivation, only the relative volatilities of two components have been used. The same relation may be applied to two components of a multicomponent mixture, as is seen in Section 11.7.6.

Example 11.9

For the separation of a mixture of benzene and toluene, considered in Example 11.7, $x_d = 0.9$, $x_w = 0.1$, and $x_f = 0.4$. If the mean volatility of benzene relative to toluene is 2.4, what is the number of plates required at total reflux?

Solution

The number of plates at total reflux is given by:

$$n + 1 = \frac{\log \left[\left(\frac{0.9}{0.1} \right) \left(\frac{0.9}{0.1} \right) \right]}{\log 2.4} = 5.0 \quad (\text{equation 11.58})$$

Thus the number of theoretical plates in the column is 4, a value which is independent of the feed composition.

If the feed is liquid at its boiling point, then the minimum reflux ratio R_m is given by:

$$\begin{aligned} R_m &= \frac{1}{\alpha - 1} \left[\frac{x_d}{x_f} - \alpha \frac{(1 - x_d)}{(1 - x_f)} \right] \quad (\text{equation 11.56}) \\ &= \frac{1}{2.4 - 1} \left[\frac{0.9}{0.4} - \frac{(2.4 \times 0.1)}{0.6} \right] \\ &= \underline{\underline{1.32}} \end{aligned}$$

Using the graphical construction shown in Figure 11.18, with $y_f = 0.61$, the value of R_m is:

$$R_m = \frac{x_d - y_f}{y_f - x_f} = \frac{(0.9 - 0.61)}{(0.61 - 0.4)} = \underline{\underline{1.38}}$$

Selection of economic reflux ratio

The cost of a distillation unit includes the capital cost of the column, determined largely by the number and diameter of the plates, and the operating costs, determined by the steam and cooling water requirements. The depreciation charges may be taken as a percentage of the capital cost, and the two together taken as the overall charges. The steam required will be proportional to V_m , which may be taken as V_n where the feed is liquid at its boiling point. From a material balance over the top portion of the column, $V_n = D(R + 1)$, and hence the steam required per mole of product is proportional to $(R + 1)$. This will be a minimum when R equals R_m , and will steadily rise as R is increased. The relationship between the number of plates n and the reflux ratio R , as derived by GILLILAND⁽³⁰⁾, is discussed in Section 11.7.7.

The reduction in the required number of plates as R is increased beyond R_m will tend to reduce the cost of the column. For a column separating a benzene–toluene mixture, for example, where $x_f = 0.79$, $x_d = 0.99$ and $x_w = 0.01$, the numbers of theoretical plates as given by the McCabe–Thiele method for various values of R are given as follows. The minimum reflux ratio for this case is 0.81.

| | | | | | |
|------------------|----------|-----|-----|-----|-----|
| Reflux ratio R | 0.81 | 0.9 | 1.0 | 1.1 | 1.2 |
| Number of plates | ∞ | 25 | 22 | 19 | 18 |

Thus, an increase in R , at values near R_m , gives a marked reduction in the number of plates, although at higher values of R , further increases have little effect on the number of plates. Increasing the reflux ratio from R_m therefore affects the capital and operating costs of a column as follows: