1. The fractionating process

The operation of a typical fractionating column may be followed by reference to Figure 11.11. The column consists of a cylindrical structure divided into sections by a series of perforated trays which permit the upward flow of vapour. The liquid reflux flows across each tray, over a weir and down a downcomer to the tray below. The vapour rising from the top tray passes to a condenser and then through an accumulator or reflux drum and a reflux divider, where part is withdrawn as the overhead product D, and the remainder is returned to the top tray as reflux R.

The liquid in the base of the column is frequently heated, either by condensing steam or by a hot oil stream, and the vapour rises through the perforations to the bottom tray. A more commonly used arrangement with an external reboiler is shown in Figure 11.11 where the liquid from the still passes into the reboiler where it flows over the tubes and weir and leaves as the bottom product by way of a bottoms cooler, which preheats the incoming feed. The vapour generated in the reboiler is returned to the bottom of the column with a composition ys, and enters the bottom tray where it is partially condensed and then revaporised to give vapour of composition y1. This operation of partial condensation of the rising vapour and partial vaporisation of the reflux liquid is repeated on each tray. Vapour of composition yt from the top tray is condensed to give the top product D and the reflux R, both of the same composition yt . The feed stream is introduced on some intermediate tray where the liquid has approximately the same composition as the feed. The part of the column above the feed point is known as the rectifying section and the lower portion is known as the stripping section. The vapour rising from an ideal tray will be in equilibrium with the liquid leaving, although in practice a smaller degree of enrichment will occur.

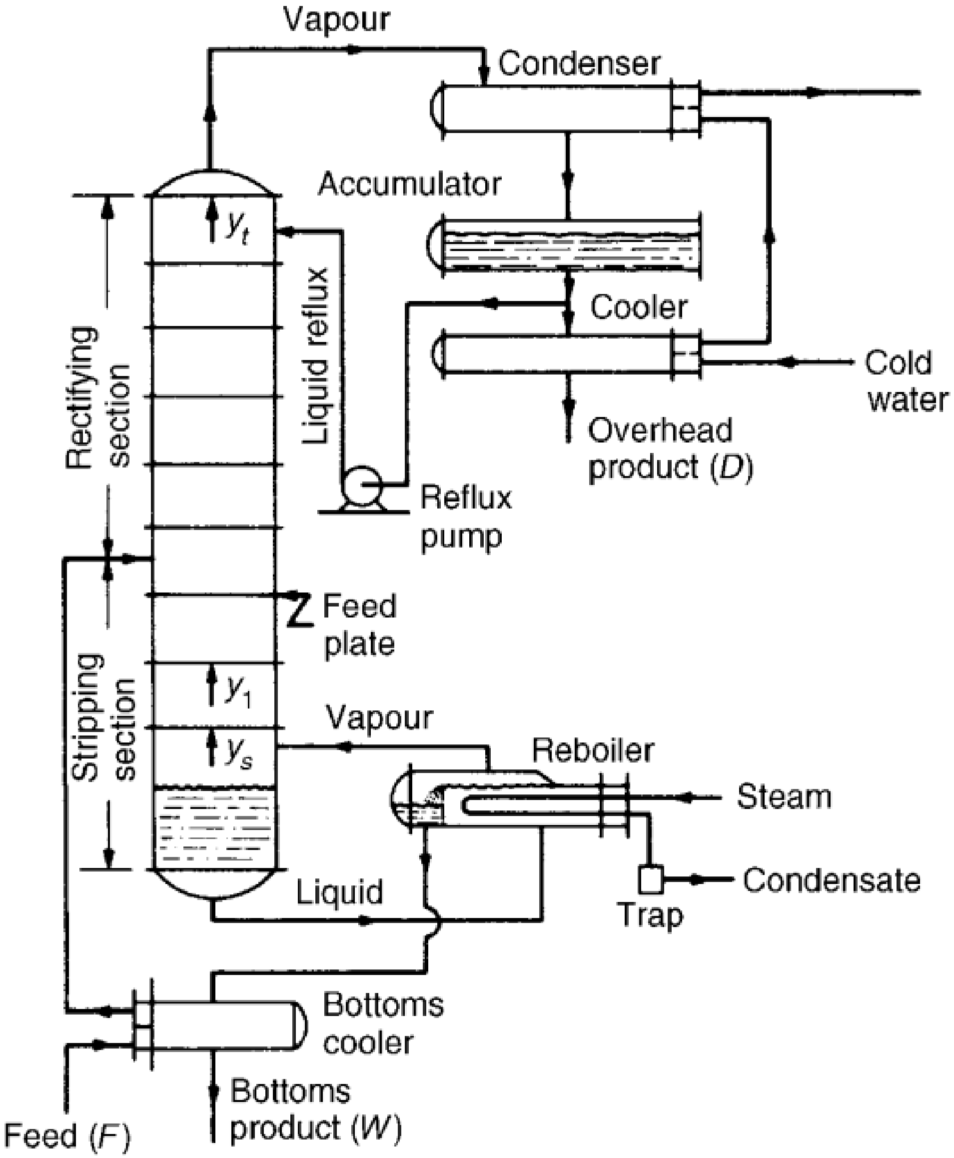

Figure 11.11. Continuous fractionating column with rectifying and stripping sections

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In analysing the operation on each tray it is important to note that the vapour rising to it, and the reflux flowing down to it, are not in equilibrium, and adequate rates of mass and heat transfer are essential for the proper functioning of the tray. The tray as described is known as a sieve tray and it has perforations of up to about 12 mm diameter, although there are several alternative arrangements for promoting mass transfer on the tray, such as valve units, bubble caps and other devices described in Section 11.10.1. In all cases the aim is to promote good mixing of vapour and liquid with a low drop in pressure across the tray.

On each tray the system tends to reach equilibrium because:

(a) Some of the less volatile component condenses from the rising vapour into the liquid thus increasing the concentration of the more volatile component (MVC) in the vapour.

(b) Some of the MVC is vaporised from the liquid on the tray thus decreasing the concentration of the MVC in the liquid.

The number of molecules passing in each direction from vapour to liquid and in reverse is approximately the same since the heat given out by one mole of the vapour on condensing is approximately equal to the heat required to vaporise one mole of the liquid. The problem is thus one of equimolecular counterdiffusion If the molar heats of vaporisation are approximately constant, the flows of liquid and vapour in each part of the column will not vary from tray to tray.

2. Number of plates required in a distillation column

In order to develop a method for the design of distillation units to give the desired fractionation, it is necessary, in the first instance, to develop an analytical approach which enables the necessary number of trays to be calculated. First the heat and material flows over the trays, the condenser, and the reboiler must be established. Thermodynamic data are required to establish how much mass transfer is needed to establish equilibrium between the streams leaving each tray. The required diameter of the column will be dictated by the necessity to accommodate the desired flowrates, to operate within the available drop in pressure, while at the same time effecting the desired degree of mixing of the streams on each tray.

Four streams are involved in the transfer of heat and material across a plate, as shown in Figure 11.12 in which plate n receives liquid L_{n+1} from plate n + 1 above, and vapour V_{n-1} from plate n − 1 below. Plate n supplies liquid L_n to plate n − 1, and vapour V_n to plate n + 1.

The action of the plate is to bring about mixing so that the vapour V_n, of composition yn, approaches equilibrium with the liquid Ln, of composition xn. The streams L_{n+1} and V_{n-1} cannot be in equilibrium and, during the interchange process on the plate, some of the more volatile component is vaporised from the liquid L_{n+1}, decreasing its concentration to x_n, and some of the less volatile component is condensed from V_{n-1}, increasing the vapour concentration to y_n. The heat required to vaporise the more volatile component from the liquid is supplied by partial condensation of the vapour V_{n-1}. Thus the resulting effect is that the more volatile component is passed from the liquid running down the

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column to the vapour rising up, whilst the less volatile component is transferred in the opposite direction.

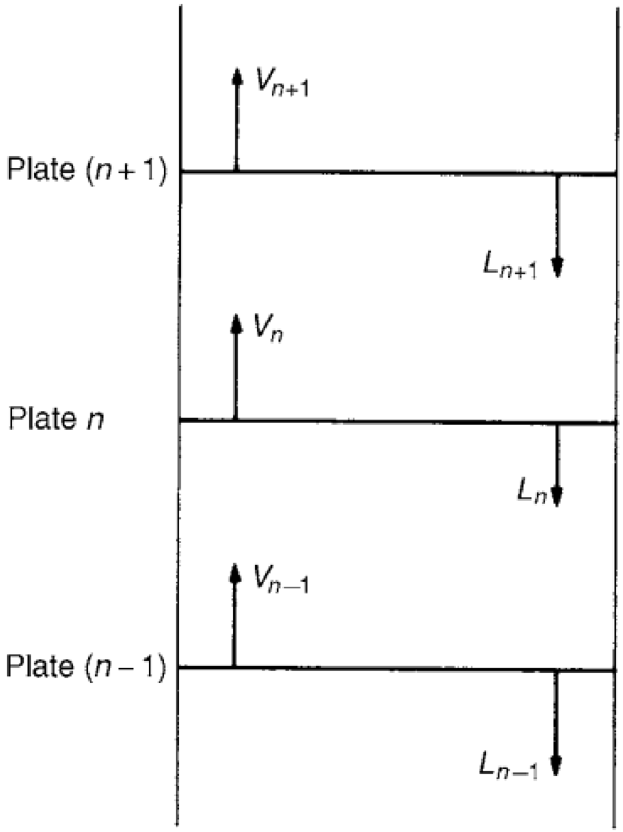

Figure 11.12. Material balance over a plate

Heat balance over a plate

A heat balance across plate n may be written as:

$$L_{n+1}H^L_{n+1} + V_{n-1}H^V_{n-1} = V_n H^V_n + L_n H^L_n + \text{losses} + \text{heat of mixing}$$

where: H^L_n is the enthalpy per mole of the liquid on plate n, and H^V_n is the enthalpy per mole of the vapour rising from plate n.

This equation is difficult to handle for the majority of mixtures, and some simplifying assumptions are usually made. Thus, with good lagging, the heat losses will be small and may be neglected, and for an ideal system the heat of mixing is zero. For such mixtures, the molar heat of vaporisation may be taken as constant and independent of the composition. Thus, one mole of vapour V_{n-1} on condensing releases sufficient heat to liberate one mole of vapour Vn. It follows that Vn = V_{n-1}, so that the molar vapour flow is constant up the column unless material enters or is withdrawn from the section. The temperature change from one plate to the next will be small, and H^L_n may be taken as equal to H^L_{n+1}. Applying these simplifications to equation 11.32, it is seen that Ln = L_{n+1}, so that the moles of liquid reflux are also constant in this section of the column. Thus V_n and L_n are constant over the rectifying section, and Vm and Lm are constant over the stripping section.

3. The importance of the reflux ratio

Influence on the number of plates required

The ratio *Ln/D*, that is the ratio of the top overflow to the quantity of product, is denoted by *R*, and this enables the equation of the operating line to be expressed in another way, which is often more convenient.

$$y_n = \left(\frac{L_n}{L_n + D}\right) x_{n+1} + \left(\frac{D}{L_n + D}\right) x_d$$
$$= \left(\frac{R}{R+1}\right) x_{n+1} + \left(\frac{x_d}{R+1}\right)$$

Any change in the reflux ratio *R* will therefore modify the slope of the operating line and, as may be seen from Figure 11.15, this will alter the number of plates required for a given separation. If R is known, the top line is most easily drawn by joining point A (x_d, x_d) to B $(0, x_d/(R+1))$ as shown in Figure 11.17. This method avoids the calculation of the actual flow rates L_n and V_n, when the number of plates only is to be estimated.

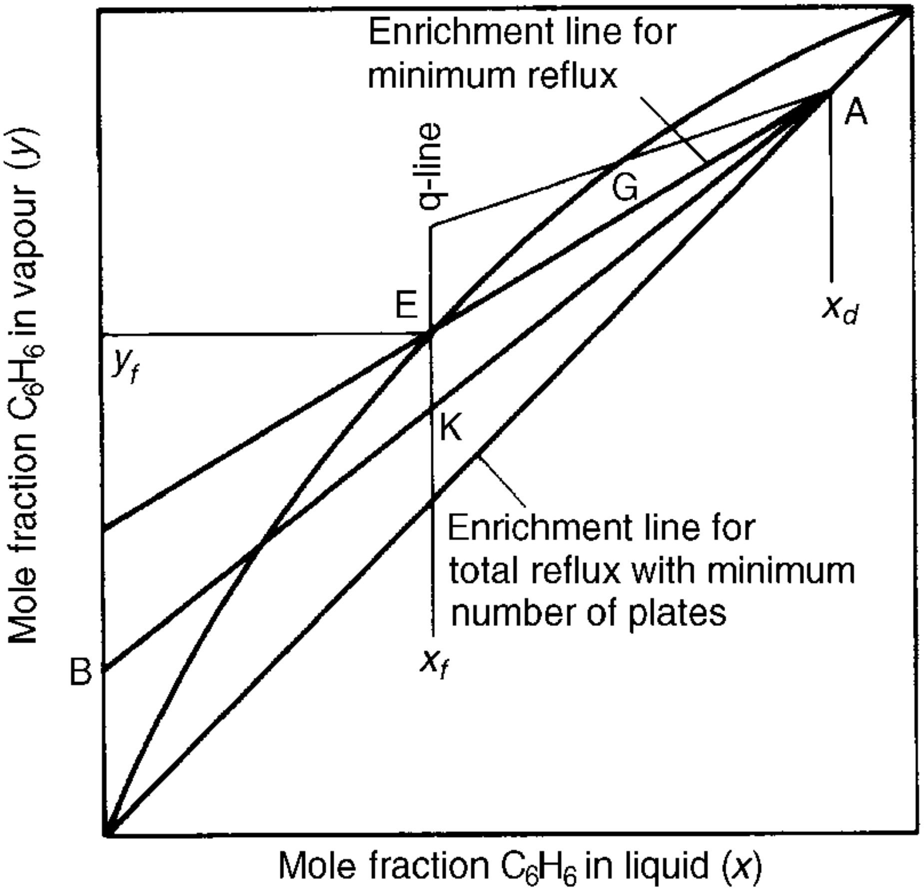

Figure 11.17. Influence of reflux ratio on the number of plates required for a given separation

If no product is withdrawn from the still, that is $D = 0$, then the column is said to operate under conditions of total reflux and, as seen from equation 11.47, the top

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operating line has its maximum slope of unity, and coincides with the line $x = y$. If the reflux ratio is reduced, the slope of the operating line is reduced and more stages are required to pass from xf to xd , 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 xd to xf . This arises from the fact that under these conditions the steps become very close together at liquid compositions near to xf , 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 Rm. Any small increase in R beyond Rm 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 Rm, represents an impossible condition, since it is impossible to pass beyond point G towards xf. 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 (xf , yf). 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

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

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 *(xc, yc)*. Thus:

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

or, since $y_c = x_f$:

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