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## Rectifying Column Calculations<sup>1</sup>

### With Particular Reference to *N* Component Mixtures

By E. V. Murphree

THE SOLVAY PROCESS CO., SYRACUSE, N. Y.

THE mathematical theory of the rectifying columns as applied to binary mixtures was first developed by Sorel.<sup>2</sup> He used as the basis of his calculations an ideal column in which the vapor and liquor on each plate were in equilibrium. By making heat and material balances for each plate he was able to calculate the liquor composition on each plate of his ideal column as determined by his initial conditions. Except for calculation simplifications this is the method generally used today.<sup>3</sup> Lewis<sup>4</sup> has developed an approximate method of calculation for cases in which the rate of change of composition of the liquor from plate to plate of the ideal column is small. His method considerably simplifies the calculations. It should only be used, however, when the rate of change is small.

In actual columns, equilibrium between vapor and liquor is seldom realized, and consequently the number of plates needed to perform a given separation in an actual column is usually greater than the number calculated for the ideal column. The ratio of the number of ideal plates to actual plates or the number of ideal plates equivalent to one actual plate for a given column is called the plate efficiency of that column. This is an average value.

The plate efficiency for many cases gives a satisfactory basis for the comparison and design of fractionating columns. It is probably a function of several variables, the most important of which seem to be the type of plate, the substances being fractionated, and in some cases the temperature. Peters<sup>5</sup> first showed the variation of plate efficiency with the material being separated. The author has seen tests on a given type of column which gave plate efficiencies varying from less than 0.1 up to 0.8, depending on the particular substances being fractionated.

In certain types of rectification the concept of the ideal column does not offer a satisfactory basis of calculation. When the plate efficiency is small, it is rather unsatisfactory not to be able to trace the liquor from plate to plate of the actual column. This is especially true when liquor or vapor is added or withdrawn from the column on other than the termi-

nal plates. A considerably more serious objection arises when the mixture being distilled contains more than two volatile components. The  $\text{NH}_3\text{-CO}_2\text{-H}_2\text{O}$  system is an example of a system of industrial importance which contains three volatile components. The difficulties that arise in using the concept of the ideal column and of plate efficiency for such systems can best be brought out by considering rectification from a different viewpoint.

Rectification consists essentially in the transfer of material between a gas phase and a liquid phase. It is therefore a special case of absorption. The less volatile components are absorbed and the more volatile liberated. The equations for the rates of material transfer are of the following type:<sup>6</sup>

The concept of the theoretical plate does not offer a satisfactory basis of calculation for rectifying columns when the mixture being rectified contains more than two components, and even for some calculations on binary mixtures its use is not satisfactory.

A method of calculation for actual plates which can be used for mixtures of any number of components is developed from the absorption equations.

A means of expressing the efficiencies of rectification of the volatile components is given.

$$\frac{dW}{d\Theta} = k_g A (P_i - P_o) = k_c A (C_L - C_i) \quad (1)$$

where  $\Theta$  = time of contact of liquor and vapor  
 $W$  = quantity of component one transferred to gas phase for an area of contact of  $A$

$k_g$  = conductivity of gas film for component one

$k_c$  = conductivity of liquid film for component one

$A$  = area of contact of liquid and vapor

$P_i$  = partial pressure of component one at vapor-liquid interface

$P_o$  = partial pressure of component one in main body of gas

$C_L$  = concentration of component one in main body of liquid

$C_i$  = concentration of component one at vapor-liquid interface

When the liquid film resistance is negligible in comparison with the gas film resistance or when the partial pressure is a linear function of the concentration, Equation 1 reduces to:

$$\frac{dW}{d\Theta} = K_g A (P_L - P_o) \quad (2)$$

where  $K_g$  = an over-all conductivity for component one  
 $P_L$  = partial pressure of component one in equilibrium with liquid

For cases in which the gas film resistance is negligible in comparison with the liquid film resistance Equation 1 reduces to

$$\frac{dW}{d\Theta} = K_c A (C_L - C_o) \quad (3)$$

where  $K_c$  = an over-all conductivity for component one  
 $C_o$  = concentration of liquid in equilibrium with the vapor

<sup>1</sup> Received March 19, 1925.

<sup>2</sup> "La Rectification de l'Alcool," 1893, Paris.

<sup>3</sup> Lewis, *THIS JOURNAL*, **1**, 522 (1909).

<sup>4</sup> *Ibid.*, **14**, 492 (1922).

<sup>5</sup> *Ibid.*, **14**, 476 (1922).

<sup>6</sup> Lewis and Whitman, *THIS JOURNAL*, **16**, 1215 (1924).

The use of the general equation in rectifying column problems would cause the calculations to become very much involved, and is therefore not considered feasible for practical purposes. It is believed that Equation 2 or 3 should form the basis of calculation in all cases, although their use may introduce some error. As all rectifying column calculations are essentially extrapolations of plant data, serious error should not be introduced in this way.

In most cases of rectification of commercial importance the mutual solubilities of the components are high, and hence the liquid film resistance is low, or else when low the partial pressures of the components may be expressed as a linear function of the concentration over the distillation range. For these cases it is believed that Equation 2 represents the material transfer with sufficient accuracy.

When the mutual solubilities of some of the components are low, which tends to make the liquid film resistance for these components high, and when the partial pressures of these components cannot be represented as a linear function of the concentration, it is believed that the rate of material transfer should be represented by an equation of the type of (3). The rate of material transfer for the other components should, as before, be represented by Equation 2.

In order for the vapor to be in equilibrium with the liquor it leaves for a finite time of contact,  $K_o$  and  $K_c$  would have to be infinity for all the components. This means that the maximum amounts of the components would be transferred if equilibrium were reached, and that actually less are transferred.

It is interesting to note that equations of the above type equal in number to the number of components, together with a heat balance, would sharply define the temperature of the liquor on any plate.

The absorption equations show clearly why the plate efficiency varies with the substances being rectified. Both  $k_o$  (and  $k_c$ ) and the plate efficiency are measures of the approach to equilibrium; if one varies, the other would be expected to vary. It is known that  $K_o$  (and  $K_c$ ) vary for different substances,<sup>7,8</sup> and possibly with different solvents for a given substance, and therefore the plate efficiency would be expected to do the same. For three-component mixtures the approach to equilibrium would not in general be equal for the two volatile components, and hence the theoretical plate cannot be used as a basis of calculation.

The following method of calculation can be applied only where the rate of material transfer is governed by an equation of the type of (2), although, as will be shown later, the treatment may be readily modified for cases in which the rate of material transfer is governed by equations similar to (3).

#### Development of Method of Calculation when Equation 2 Applies

The absorption equations offer a basis of calculation for rectifying columns when any number of components are present. In using the absorption equations it is desirable to use a certain amount of latent heat as a basis of calculation. This can most conveniently be done by taking the molal latent heat of vaporization for one of the components as a basis and assigning artificial molecular weights to the other components, so that the molal latent heats of these artificial mols will be equal to the molal latent heat taken as a standard.

An imaginary column that is at the same temperature throughout will first be considered. In such a column on the mol basis mentioned above, the number of mols of liquor and vapor would be constant from plate to plate.

<sup>7</sup> Haslam, Hershey, and Keen, *THIS JOURNAL*, **16**, 1224 (1924).

<sup>8</sup> Whitman and Davis, *Ibid.*, **16**, 1233 (1924).

#### NOMENCLATURE

- $P_n$  = total pressure of vapor above  $n$ th plate  
 $p'_n, p''_n, p'''_n$  = partial pressures of components one, two, three in equilibrium with liquor on  $n$ th plate. The more volatile components are taken as two and three and the less volatile as one.  
 $a_n$  = mol fraction of component one in liquor on  $n$ th plate  
 $b_n$  = mol fraction of component two in liquor on  $n$ th plate  
 $c_n$  = mol fraction of component three in liquor on  $n$ th plate  
 $w_n$  = mol fraction of component one in vapor above  $n$ th plate  
 $y_n$  = mol fraction of component two in vapor above  $n$ th plate  
 $z_n$  = mol fraction of component three in vapor above  $n$ th plate  
 $V_n$  = mols of vapor leaving  $n$ th plate per unit of product or whatever is taken as the basis of calculation  
 $O_n$  = mols of liquor leaving the  $n$ th plate on the same basis as  $V_n$   
 $t_n$  = temperature of liquor on  $n$ th plate  
 $\Theta_n$  = time of contact of liquor and vapor on  $n$ th plate  
 $A_n$  = area of contact per mol vapor on  $n$ th plate  
 $Kg'_n, Kg''_n, Kg'''_n$  = coefficients of material transfer on the  $n$ th plate for components one, two, and three  
 $m_n$  = molal heat capacity of liquor from  $n$ th plate  
 $L$  = molal latent heat of vaporization which on the basis taken is the same for all components  
 $M$  = average value of  $e^{-Kg''_n A_n P_n \Theta_n}$   
 $N$  = average value of  $e^{-Kg'''_n A_n P_n \Theta_n}$

In the following calculations it will be assumed that three components are present, but the method is capable of extension to mixtures of any number of components.

Equation 2 for component two may be written in the following form:

$$\frac{d(Y_n V_n)}{d\Theta_n} = Kg''_n A_n V_n (p''_n - y_n P_n)$$

$$\text{or } \frac{V_n dy_n}{d\Theta_n} + y_n \frac{dV_n}{d\Theta_n} = Kg''_n A_n V_n (p''_n - y_n P_n) \quad (4)$$

Similar equations hold for components one and three. For an isothermal column  $V_n$  is constant, and hence  $\frac{dV_n}{d\Theta_n}$  is zero.

If the composition of the vapor going to the  $n$ th plate of this isothermal column is known, the composition of the vapor leaving the  $n$ th plate may be found by integrating Equation 4.

$$\frac{P_n dy}{p''_n - y P_n} = Kg''_n A_n P_n d\Theta_n$$

$$\text{or } \ln \frac{p''_n - y_{n-1} P_n}{p''_n - y_n P_n} = Kg''_n A_n P_n \Theta_n$$

$$\text{or } y_n = \frac{p''_n}{P_n} - \left( \frac{p''_n}{P_n} - y_{n-1} \right) e^{-Kg''_n A_n P_n \Theta_n} \quad (5)$$

The small change in  $P_n$  from the bottom to the top of the liquor on the plate has been neglected.  $p''_n$  for a given plate is a constant and has been so considered in the integration.

The ratio of  $p_n/P_n$  is the value of  $y$  in equilibrium with the liquor on the  $n$ th plate and will be designated by  $y_n^*$ . Equation 5 may therefore be written

$$y_n = y_n^* - (y_n^* - y_{n-1}) e^{-Kg''_n A_n P_n \Theta_n} \quad (6)$$

This equation will be slightly in error because artificial molecular weights and not real molecular weights are used. The area of contact for a mol of vapor on a plate may therefore vary with the change of composition of the vapor in passing through the plate. For the most cases of industrial importance, the artificial molecular weight will not vary greatly from the true molecular weight, and hence the error may be eliminated by considering  $A_n$  to be an average area of contact.

Equation 6 was derived for an imaginary isothermal column. On most plates of a rectifying column the amount of vapor condensed for heating purposes is small compared with the total amount of vapor. For these plates  $y_n dV_n/d\Theta_n$  is

negligible compared with  $V_n dy_n/d\Theta_n$  and Equation 6 may be used with confidence. If the feed is not preheated considerable heating may be done on the plate on which the feed is introduced and possibly on those immediately below it.

In general, the number of plates on which appreciable heating is done is small compared with the total number of plates. The values of  $y$  above the plates on which appreciable heating is done are probably not widely different from the value predicted by Equation 5, although this equation may not apply. For these reasons it is doubtful if any large error would be introduced by using Equation 5 for the whole column even though the temperature varies from plate to plate and the feed is not preheated.

If the amount of heating in the whole column is relatively small, it will be more convenient to consider the distillation as isothermal and take average quantities for the number of mols of liquor and vapor.

Equations similar to 6 can be obtained in a similar manner for the other two components. It is necessary, however, to use only the equations for the two more volatile components as the equation for the less volatile component is determined by a heat balance equation. The partial pressure of one of the components (probably the less volatile) will usually be close enough to its equilibrium pressure to determine the temperature on the different plates.

Nothing has been said so far as to how the quantity  $e^{-K_g^n A_n P_n \Theta_n}$  in Equation 4 changes from plate to plate.  $K_g$  increases<sup>6,7</sup> with increasing gas velocity except where  $k_g$  is negligible. The relation between  $K_g$  and the velocity cannot be estimated, however, unless the conductivities of the individual vapor and liquor films are known. The time of contact per plate decreases with increasing velocity. The change of  $e^{-K_g^n A_n P_n \Theta_n}$  for moderate change in velocity would often not be large because changes in  $K_g^n$  and  $\Theta_n$  tend to neutralize each other.  $A_n P_n$  would vary with the cube root of the pressure and would be proportional to the two-thirds power of the absolute temperature. As the temperature and pressure drops between the bottom and top of most rectifying columns are not large, and as their effects tend to neutralize each other, the change of  $A_n P_n$  throughout the column would not be great. If the artificial molecular weights used vary much from the true molecular weights,  $A_n$  would be a function of the composition of the vapor.

$K_g$  is a function of the temperature. When Henry's law holds, it may be written as  $K_g = \frac{1}{\frac{1}{k_g} + \frac{1}{Hk_c}}$ . According to

Haslam<sup>7</sup> and his co-workers,  $k_g$  increases and  $k_c$  decreases with decreasing temperature. ( $H$  is the Henry's law constant.) The rate of change of  $k_c$  with the temperature is considerably more rapid than that of  $k_g$ . The net effect of temperature change on  $K_g$  depends on the relative values of  $k_g$  and  $Hk_c$ . When the gas film resistance is relatively large, which is probably true in most rectifications, the effect of moderate temperature changes on  $K_g$  would not be expected to be large.

No decisive conclusions as to how  $e^{-K_g^n A_n P_n \Theta_n}$  would change from plate to plate can be drawn from the foregoing discussion. Such a question can best be decided by experiment. It has been shown, however, that for all changes there are effects tending to neutralize each other and consequently the value of the term may not vary greatly from plate to plate. In any case, for engineering purposes it would be best to use an average value of the term as determined by actual column tests for the substances being separated. When this simplification is adopted, Equation 6 becomes

$$y_n = y_n^* - M (y_n^* - y_{n-1}) \quad (7)$$

The analogous equation for the other more volatile component is

$$z_n = z_n^* - N (z_n^* - z_{n-1}) \quad (8)$$

In these equations  $M$  and  $N$  are the average values for the exponential terms. The ratio of the logarithms of  $M$  and  $N$  is equal to the ratio of  $K_g^n$  and  $K_g^{n'}$  and this ratio can probably be determined by small-scale experiment.

#### Application of Method to Rectifying Column Calculations

In applying Equations 7 and 8 to column calculations, the same method is used as in calculations based on the concept of the theoretical plate, except that these two equations are used instead of considering the liquor and vapor in equilibrium. In using these equations it is best to start at the bottom of the column and work up, as the calculations become considerably more involved in working down the column.

General equations will first be derived connecting the  $n-1$ ,  $n$ , and  $n+1$  plates. By making material and heat balances the following equations are obtained:

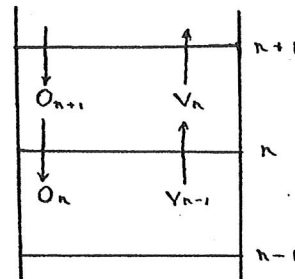
$$\left. \begin{aligned} b_{n+1} O_{n+1} + y_{n-1} V_{n-1} &= b_n O_n + y_n V_n \\ c_{n+1} O_{n+1} + z_{n-1} V_{n-1} &= c_n O_n + z_n V_n \end{aligned} \right\} \quad (9)$$

$$O_{n+1} + V_{n-1} = O_n + V_n \quad (10)$$

$$L(V_{n-1} - V_n) = m_{n+1}(t_n - t_{n+1}) O_{n+1} \quad (11)$$

Note—The sensible heat of the gases is neglected in this heat balance (Equation 11) as it is small in comparison with the other heat quantities.

These equations, together with Equations 7 and 8, are all that are needed to calculate a column. Starting with the bottom or first plate the composition and amount of vapor to and liquor from the plate are known. The composition of the vapor above the bottom plate can be determined from Equations 7 and 8. The temperature of the bottom plate is known and by making an estimate of the temperature of the plate above it, the amount of vapor leaving the bottom plate and the overflow from the plate above can be found by



Equations 10 and 11. The fact that one of the partial pressure components is usually near enough to its equilibrium pressure to serve as a measure of temperature serves as a check on this estimate. This assumes that the total pressure is known from plate to plate. Usually the total pressure drop may be considered equal from plate to plate. If the total pressure drop through the column is known, the total pressure above each plate may be estimated on the basis of this assumption. If the amount of vapor below and the amount of liquor from the second plate are known, the composition of the liquor may be calculated by Equations 9 and 10. The same procedure can then be followed for the next plate and so on up the column.

The calculations are relatively simple for two-component mixtures, but both the calculations and equilibrium data become considerably involved for three-component mixtures. For four-component mixtures they would probably become too involved to be used.

It is believed that even for binary mixtures this method of calculation based on the absorption equations offers a more reliable extrapolation of test and operation data than calculations based on the concept of the theoretical plate, because the mechanism of the process is better represented by these equations. It also has the advantage of being extended to mixtures of more than two components. The quantities  $1-M$  and  $1-N$  can be taken as measures of the efficiencies of rectification of the two volatile components. Both  $M$  and  $N$  lie between 0 and 1, being 0 for equilibrium and 1 when there

is no rectification. Hence  $1-M$  and  $1-N$  will lie between 0 and 1, approaching 1 for the maximum rectification.

If the material transfer must be represented by an equation of the type of (3), the equation which corresponds to (4), on omitting the primes, is

$$\frac{d(y_n V_n)}{d\theta_n} = \frac{V_n dy_n}{d\theta_n} + \frac{y_n dV_n}{d\theta_n} = K_{c_n} A_n V_n (C_{L_n} - C_{g_n}) \quad (12)$$

where  $K_{c_n}$  = an over-all conductivity for the component being considered

$C_{L_n}$  = concentration of that component in the liquid  
 $C_{g_n}$  = concentration of that component in equilibrium with the vapor

All other quantities have the same significance as in the previous sections.

The quantity  $C_{g_n}$  may be replaced by  $f(y_n)$ , which is some function of  $y_n$ . On making this substitution Equation 12 for an isothermal column becomes

$$\frac{dy_n}{C_{L_n} - f(y_n)} = K_{c_n} A_n d\theta_n \quad (13)$$

or on integrating

$$\int_{y_{n-1}}^{y_n} \frac{dy_n}{C_{L_n} - f(y_n)} = K_{c_n} A_n \theta_n \quad (14)$$

The functional relation between  $y_n$  and  $C_{g_n}$  may be obtained by fitting an empirical equation to the  $y-C$  curve. A straight-line relationship over the range of  $y$  for each plate of the form  $C_n = S_n y_n + R_n$  can be used as an approximation when  $S_n$  and  $R_n$  are constants for a given plate. These constants may change from plate to plate depending on the  $y-C$  curve. If this is done Equation 14 becomes

$$\int_{y_{n-1}}^{y_n} \frac{dy_n}{C_{L_n} - R_n - S_n y_n} = K_{c_n} A_n \theta_n$$

or

$$\frac{\frac{C_{L_n} - R_n}{S_n} - y_{n-1}}{\frac{C_{L_n} - R_n}{S_n} - y_n} = e^{-S_n K_{c_n} A_n \theta_n}$$

which since  $\frac{C_{L_n} - R_n}{S_n} = y_n^*$  is the value of  $y_n$  in equilibrium

with the liquor on the  $n$ th plate, becomes

$$y_n = y_n^* - (y_n^* - y_{n-1}) e^{-S_n K_{c_n} A_n \theta_n} \quad (15)$$

With the exception of the exponential term this equation is the same as Equation 6.

The same discussion as to the justification of using Equation 6 when the column is not isothermal also applies to Equation 15.

The greater the value of  $S_n \left[ = \left( \frac{dc}{dy} \right)_{c=c_n} \right]$  approximately—which means the smaller the rate of change of pressure with concentration—the nearer, other things being equal, is the approach to equilibrium.  $K_{c_n}$  should vary considerably with the temperature and hence would decrease in going up the column. According to Halsam, Hershey, and Keen<sup>7</sup> under the conditions at which they worked,  $k_c$  would not be a function of the gas velocity. This may not be true in a rectifying column.  $\theta_n$  would, however, vary inversely as the gas velocity; hence increasing the rate of working would, if  $K_c$  is constant, decrease the approach to equilibrium. The effect of pressure and temperature on  $A_n \theta_n$  would not be great.

Increasing the temperature should be an important factor in increasing the approach to equilibrium, and hence the efficiency of distillation (neglecting changes in relative volatility) when the liquid film is the controlling resistance. The temperature should not be an important factor (neglecting changes in relative volatility) when the gas film resistance is controlling.

In using Equation 15 an average value of  $K_{c_n} A_n \theta_n$  determined from column tests should be used. If this average value is designated by  $Q$ , Equation 15 becomes

$$y_n = y_n^* - (y_n^* - y_{n-1}) e^{-S_n Q} \quad (16)$$

Similar equations may be obtained for other components. Equations of this sort are used in connection with the calculations described above in the same way that Equations 7 and 8 are used.

The quantity  $1 - e^{-S_n Q}$  may be taken as a measure of the efficiency of distillation of that component. Its value must lie between 1 and 0.

## Mines Transferred to Hoover

President Coolidge on June 4 by Executive Order transferred the Bureau of Mines from the Department of the Interior to the Department of Commerce. This is another step in the general movement for coordination of the work of the various government agencies under one head. It will be recalled that a short time ago the Patent Office was transferred from the Department of the Interior to the Department of Commerce. Two offices of the Bureau of Mines which deal with coal, oil, and other mineral-land leasing will remain under the Interior Department, since they are not engaged in scientific research and hence cannot be transferred under the law. Secretary Hoover has announced that he will appoint a committee representative of organizations of mining engineers and the mining industry to study ways and means to increase the efficiency of the Bureau of Mines and the divisions of the Department of Commerce engaged in similar lines of investigation. Plans for reorganization will not be prepared until after this committee reports. Secretary Hoover regards the Bureau of Mines as a service bureau for the mining industry and its activities will be developed with this in mind. Secretary Work commented as follows upon the transfer:

The change is one of the reorganization plans approved by the Joint Congressional Committee. The President has a limited authority under the organic act creating the Department of Commerce to make such shifts. It does not apply to other departmental changes which must await Congressional authority. There are other functions which will be transferred from the Department of Commerce to the Department of the Interior when such authority is available.

The primary objects of such segregation are, of course, to secure economy in administration and more efficient relationship with the public.

The Department of Commerce contains a division for service in domestic

distribution and foreign trade in mineral products; the Bureau of Mines carries on work of economic character of much the same implication. The Department of Commerce provides statistics of mineral production every ten years, whereas the Department of the Interior provides statistics of mineral production every year and in many cases every month. The Department of Commerce recruits statistics on production of explosives every two years, while the Bureau of Mines procures statistics on their production at regular intervals.

The Department of Commerce carries on research into the strength of wire rope generally for all industries, whereas the Bureau of Mines investigates the strength of wire ropes for the mining industry. The Department of Commerce maintains economic research in use of raw materials for manufacture, a large portion of which are minerals, while the Bureau of Mines investigates the production of raw materials for manufacturing purposes.

Our investigations show that the laboratories of the Department of Commerce carry on scientific research in the use of gasoline and lubricating oils in gas engines, while the Bureau of Mines carries on research generally into gasoline and petroleum products. The research laboratories in the Department of Commerce carry on investigation into the quality of materials for manufacture of porcelain, whereas the Bureau of Mines laboratories carry on research into raw materials for porcelain manufacture. The Department of Commerce laboratories carry on investigation into the qualities of fuel whereas the Bureau of Mines laboratories test fuel as to its qualities.

While by constant adjustment, conferences and the appointment of cooperative committees a considerable amount of the actual duplication has been eliminated during the last four years, nevertheless such duplications cannot be eliminated and the confusion of citizens in dealing with different government departments for different purposes cannot be planned out unless single-headed authority is given for functions having the same general major purpose.