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A Rigorous Method for Calculating Minimum Reflux Rates in Distillation

A computer method has been developed to calculate the minimum reflux rate for a specified distillation separation. The method makes no unnecessary simplifying assumptions of the distillation process model. The limitation of the method is that it cannot solve problems in which the minimum reflux rate is determined by a point of tangent between the operating line and the equilibrium line on a McCabe-Thiele diagram (see Figure 1).

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SCOPE

The calculation of minimum reflux rates for specified distillation separations has long been one of the important problems in distillation calculations. The concept of minimum reflux is quite basic but useful in the design of distillation processes. Columns can be designed quickly and with confidence if the minimum reflux rate is known. Obviously, the designed reflux rate must always be greater than the minimum rate by a certain amount. This amount should be selected with considerations based on the certainty of the vapor-liquid equilibrium model as well as comparative costs of capital vs. utilities. If the equilibrium model in use is not accurate, or if costs of utilities are relatively low, one should choose a large ratio (1.3 or larger) of actual to minimum reflux. A known minimum reflux rate can therefore reduce significantly operating, start-up, and design costs of a distillation process.

Most of the well-known work published in the area of minimum reflux calculations appeared before the advent of high speed computers. Developed primarily for manual or low speed computer calculations, these methods used simplifying assumptions in order to save time. These assumptions often cause uncertain and inaccurate results. The more common of these assumptions are constant molal overflows (Underwood, 1948; McCabe and Thiele, 1925), constant relative volatilities (Underwood, 1948), binary distillation (Ponchon, 1921; Savarit, 1922; McCabe and Thiele, 1925), and known product compositions (Jenny, 1939).

Bachelor (1957) presented perhaps the best hand calculation method to date. His method did not contain any of the above assumptions; however, it used simplifying approximations in equilibrium and heat and material balances. Erbar and Maddox (1962) published a computer method using Bachelor's method with added refinements but did not include problems of superheated or subcooled feeds.

CONCLUSIONS AND SIGNIFICANCE

The method developed in this work is designed for use on a high speed computer and is rigorous in the sense that:

1. The vapor-liquid equilibrium relationship may be represented by any nonideal model.
2. Any component or mixture enthalpy model may be used.
3. All or any two components may be distributed between the distillate and the bottoms.
4. Plate material and energy balances are used where needed.
5. Overall column material and energy balances are satisfied at convergence.

The solution speed obviously depends on the complexity of physical property and equilibrium models used for the problem. But, in general, this method requires more computer time than is required by a rigorous distillation simulation program.

The most important and restrictive assumption used in the development of this method is that the pinch zone cannot be at a point where the operating line is tangent to the equilibrium surface (see Figure 1). Our experience indicated that the use of this method for such a problem resulted in large solution oscillations such that convergence was not possible. However, it has produced correct solutions to problems for which the minimum refluxes are negative.

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MODEL DESCRIPTION AND ALGORITHM

In the design of a simple distillation column, as the reflux is reduced, a specified separation can be maintained by increasing the number of stages in the column. The minimum reflux for the specified separation is reached when the stage requirement becomes infinite. At the minimum reflux, a column should have one or two pinch zones. A pinch zone is a section of the column with infinite number of stages having identical liquid compositions, vapor compositions, pressures, temperatures, and molal overflows.

Most columns at minimum reflux have two pinch zones, one above and one below the feed. These zones are usually near or adjacent to the feed. If both zones are adjacent to the feed, compositions and temperatures in the two zones will be the same, but the molal overflows will be different as a result of feed addition.

Columns with a single pinch zone are not as common. In these columns, the pinch zone is where the operating line is tangent to the equilibrium surface (see Figure 1). The method developed in this work and all other available analytical methods to date are designed to solve problems with two pinch zones. Unless stated otherwise, all following discussions pertain to problems with two pinch zones.

Two classes of separations have been defined in the distillation literature. Class one separations are those in which all components in the feed are distributed to the top and the bottom products. In this case, the two pinch zones are always adjacent to the feed stage. Binary systems are typical examples of this class.

Class two separations are those in which several components in the feed are not distributed. If one or more components do not appear in the top product, a section of stages will be present between the feed stage and the rectifying pinch zone to remove these nondistributed components. A similar section can also exist below the feed stage.

Class one problems can also be considered as a subset of class two problems, since any method that solves class two problems will undoubtedly solve class one problems as well. However, simplified methods designed specifically for class one problems cannot, in general, solve class two problems. In the algorithm presented below, two methods are used. A rigorous and iterative method for class one problems is applied first. The solution will be exact only for class one problems. For class two problems, it is used as an initial estimate for a second method.

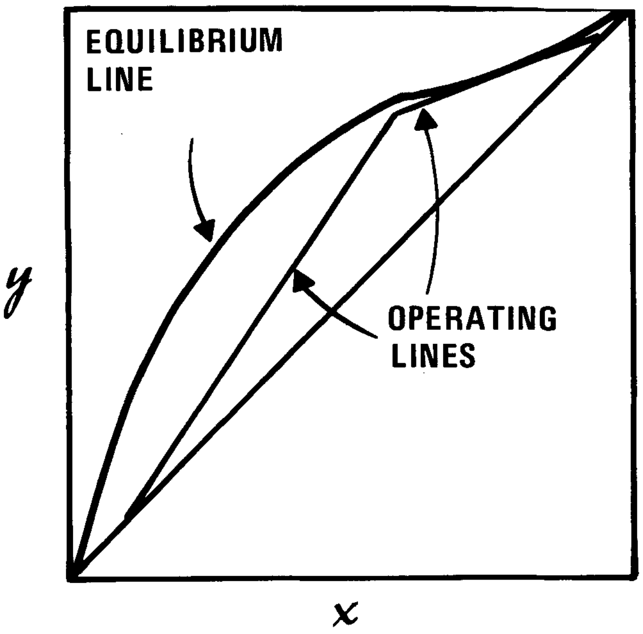

Fig. 1. Operating line tangent to equilibrium line.

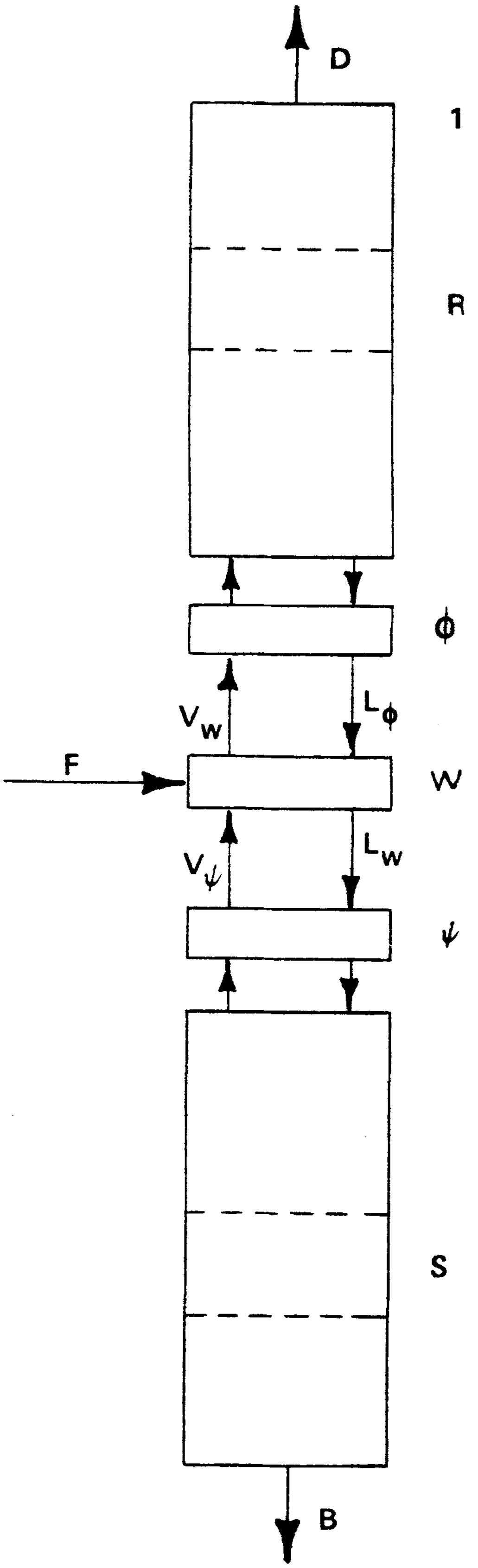

Fig. 2. Column configuration.

The second method is designed for class two problems. Before discussing the two methods used in the algorithm, we shall first present the concept of component recoveries and their use for overall material balance calculations.

Component Recoveries and Material Balances

Component material balances in a column can be expressed in terms of recoveries. For the i^{th} component, the rectifying recovery G_i and the stripping recovery U_i are defined as (see Figure 2)

$$G_i \equiv l_{\phi i}/v_{wi} \leq 1 \tag{1}$$

$$U_i \equiv v_{\psi i}/l_{wi} \leq 1 \tag{2}$$

The component flows are related to recoveries by

$$\begin{array}{llll} v_{wi} = \dfrac{f_i\overline{U}_i}{1-\overline{U}_iG_i} & (a) & l_{wi} = \left(\dfrac{L}{VK_i}\right)_w v_{wi} & (d) \\ l_{\phi i} = G_iv_{wi} & (b) & v_{\psi i} = U_il_{wi} & (e) \\ d_i = (1-G_i)v_{wi} & (c) & b_i = (1-U_i)l_{wi} & (f) \end{array} \tag{3}$$

where

$$\overline{U}_i \equiv \frac{v_{wi}}{v_{wi}+b_i} = \frac{1}{1+\dfrac{b_i}{v_{wi}}} = \frac{1}{1+(1-U_i)\left(\dfrac{L}{VK_i}\right)_w} \tag{4}$$

$$K_{wi} \equiv K_{wi}(x_w, y_w, T_w, p_w) \tag{5}$$

$$x_{wi} = \frac{l_{wi}}{\sum\limits_j l_{wj}} \quad y_{wi} = \frac{v_{wi}}{\sum\limits_j v_{wj}} \tag{6}$$

For a given set of U_i's and G_i's, the above equations bear close resemblance to those for a single equilibrium flash unit. Equations (3) and (4) are the component material balances. Equations (5) are the phase equilibrium composition ratios. As in an adiabatic flash calculation, the unknowns are T_w, and $(L/V)_w$ and the dependent equations are: the flash equation

$$\sum_i (K_{wi}-1)x_{wi} = 0 \tag{7}$$

and the enthalpy balance equation for stage w

$$Fh_f = V_wH_w + L_wh_w - V_\psi H_\psi - L_\phi h_\phi \tag{8}$$

where the enthalpies H_w, h_w, H_ψ, and h_ϕ must be evaluated at corresponding dew points or bubble points. The algorithm for solving the above equations will be discussed later. We shall first introduce the concept of relative recoveries and their use in satisfying separation specifications.

Relative Recoveries and Separation Specifications

One of the simplest ways to indicate a desirable distillation separation is by specifying key component splits. For example, one may specify

$$\left(\frac{d}{f}\right)_h \quad \text{and} \quad \left(\frac{b}{f}\right)_l$$

to define key component distributions and equivalently these following quantities:

$$d_h = \left(\frac{d}{f}\right)_h f_h, \quad b_h = f_h - d_h, \quad b_l = \left(\frac{b}{f}\right)_l f_l, \quad d_l = f_l - b_l \tag{9}$$

For an arbitrary set of G_i's and U_i's, the separation specification is usually not satisfied. One way to enforce a specification is to change column energy flows. However, a change in the energy flow affects all the component recoveries. If one assumes that all recoveries are changed by the same ratio, then

$$\hat{U}_i = \eta U_i \tag{10}$$

$$\hat{G}_i = \xi G_i \tag{11}$$

where $\hat{U}_i$ and $\hat{G}_i$ represent the modified recoveries that satisfy the separation specification. Variables η and ξ can then be adjusted to enforce separation specifications.

Equations (10) and (11) can also be written in terms of constant relative recoveries:

$$u_i = \frac{U_i}{U_h} = \frac{\hat{U}_i}{\hat{U}_h} \tag{12}$$

$$g_i = \frac{G_h}{G_i} = \frac{\hat{G}_h}{\hat{G}_i} \tag{13}$$

Substitute the above equations into Equations (3*c*), (3*d*), and (3*f*) to obtain overall balances of the heavy and light key components:

$$d_h = \frac{1-\hat{G}_h}{1-\hat{U}_h}\frac{b_h}{A_h} \quad A_h \equiv \left(\frac{L}{VK_h}\right)_w$$

$$d_l = \frac{1-\dfrac{\hat{G}_h}{g_l}}{1-u_l\hat{U}_h}\frac{b_l}{A_l} \quad A_l \equiv \left(\frac{L}{VK_l}\right)_w$$

For a given set of specifications (d_h, b_h, d_l, b_l) and for the assumed relative recoveries (g_l, u_l) and stripping factors (A_h, A_l), the above two equations can be used to obtain $\hat{G}_h$ and $\hat{U}_h$:

$$\hat{U}_h = \frac{1-\dfrac{A_l}{A_h}\dfrac{d_l}{d_h}\dfrac{b_h}{b_l}g_l + \dfrac{b_h}{A_hd_h}(g_l-1)}{1-u_lg_l\dfrac{A_l}{A_h}\dfrac{d_l}{d_h}\dfrac{b_h}{b_l}} \tag{14}$$

$$\hat{G}_h = 1 - A_h\frac{d_h}{b_h}(1-\hat{U}_h) \tag{15}$$

Other recoveries can then be calculated from Equations (12) and (13):

$$\hat{U}_i = \hat{U}_hu_i$$

$$\hat{G}_i = \hat{G}_h/g_i$$

$\hat{U}_i$ and $\hat{G}_i$ are used in Equations (3) and (4) to obtain overall material balances that match the separation specifications.

Algorithm for Solving Column Balances

For a given set of recoveries, U_i, G_i, the algorithm described in this section solves Equations (3) to (9) and (12) to (15) to obtain complete column energy and material balances that satisfy the separation specification. The algorithm outlined below follows the general approach of adiabatic flash calculations:

1. Estimate $(L/V)_w$.
2. Estimate T_w.

3. Estimate X_{wi}; $i = 1, \ldots, n$ and normalize.
4. Calculate K_{wi}; $i = 1, \ldots, n$ by Equation (5).
5. Calculate $\hat{U}_i$, $\hat{G}_i$; $i = 1, \ldots, n$ Equations (14), (15), (12), and (13).
6. Use $\hat{U}_i$, $\hat{G}_i$, for U_i, G_i, in Equations (3) and (4) and calculate v_{wi}, l_{wi}, $l_{\phi i}$, $v_{\psi i}$, d_i, b_i; $i = 1, \ldots, n$.
7. Return to 2 until Equation (7) is satisfied. Use any single variable equation solving method for estimating T_w. Use direct iterations for x_{wi}; $i = 1, \ldots n$.
8. Return to 1 until Equation (8) is satisfied. $H\psi$ and $h\phi$ must be evaluated at the dew point and the bubble point, respectively.

After convergence, the reboiler and condenser duties can be calculated by energy balances as

$$Q_c = V_w H_w - L_\phi h_\phi - D h_d \tag{16}$$

$$Q_b = B h_b + D h_d + Q_c - F h_f \tag{17}$$

where h_b must be evaluated at the bubble point of the bottoms and h_d at the given distillate condition. The reflux rate can also be calculated based on both Q_c and the distillate flow by using a stage calculation method to be discussed later.

This algorithm is strongly analogous to the one for the adiabatic flash. Techniques such as applying damping to composition changes in step 7 work equally well for both distillation column balance and adiabatic flash algorithms in stabilizing difficult problems. Also, matching of variable T_w with Equation (7) and $(L/V)_w$ with Equation (8) may have to be reversed for certain special problems. It is also essential that in the search of a solution, variables are kept within their defined ranges. For example, the modified recoveries $\hat{U}_i$ and $\hat{G}_i$ must not be greater than 1 nor less than 0. These bounds help stabilize the search and improve the efficiency of the algorithm.

Solving Problems with Distributed Components

As discussed previously for class one separations, all components in the feed are distributed to the top and the bottom products, and pinch zones are adjacent to the feed stage. Since stage ϕ is in the pinch zone, y_ϕ and y_w are identical.

The relative volatilities become identical to the relative recoveries

$$\alpha_{\phi i} = \frac{y_{wi}/x_{\phi i}}{y_{wh}/x_{\phi h}} = \frac{v_{wi} l_{\phi h}}{v_{wh} l_{\phi i}} = \frac{G_h}{G_i} = g_i \tag{18}$$

Similar conditions hold for stage ψ:

$$\alpha_{\psi i} = \frac{y_{\psi i}/x_{wi}}{y_{\psi h}/x_{wh}} = \frac{v_{\psi i} l_{wh}}{v_{\psi h} l_{wh}} = \frac{U_i}{U_h} = u_i \tag{19}$$

This property can be used readily for problems with distributed components. The algorithm for solving the column balance is repeated. Each time after convergence, the relative volatilities $\alpha_{\phi i}$'s and $\alpha_{\psi i}$'s are calculated and then used as g_i's and u_i's for the next iteration, until there are no significant changes in the values of g_i's and u_i's. This direct iteration method has proven very effective in solving class one problems. Four or five iterations have been adequate to achieve convergence and to obtain reflux ratios.

Since pinch conditions in Equations (18) and (19) are necessary for columns having pinch zones adjacent to the feed stage, a solution that satisfies these conditions as well as the overall material and energy balances, Equations (3) to (17), must be rigorous for a class one problem. For class two problems, Equations (18) and (19) cannot be satisfied because either G_i or U_i for the nondistributed components must always be one. Nevertheless, it is still possible to obtain with this method a solution in which the distributed components satisfy Equations (18) and (19), and the nondistributed components satisfy $U_i = 1$ and $G_i = 1$. Results are not final but often are so close that they are excellent estimates for the second method discussed below.

General Pinch Conditions

Equations (18) and (19) represent special pinch conditions for class one problems. A more general pinch equation can be derived in terms of component recoveries and component stripping factors (or rectifying factors). For the section of the column above the feed, the component balance can be expressed in the following recursive form

$$\frac{l_{\phi i}}{d_i} = \frac{l_{\phi i}}{v_{\phi i}} \frac{v_{\phi i}}{d_i} = A_{\phi i} \frac{l_{\phi-1}}{d_i} + 1 \tag{20}$$

where

$$A_{\phi i} = \frac{l_{\phi i}}{v_{\phi i}} = \frac{L_\phi x_{\phi i}}{V_\phi y_{\phi i}} = \left(\frac{L}{VK_i}\right)_\phi$$

Expanding the recursive equation and assuming that the pinch zone is immediately above the feed stage, we have

$$\frac{l_{\phi i}}{d_i} = A_{\phi i} + A_{\phi i}^2 + \cdots + A_{\phi i}^\infty + \cdots$$

$$= \frac{A_{\phi i}}{1 - A_{\phi i}} = \frac{A_{Ri}}{1 - A_{Ri}} \tag{21}$$

If the pinch zone is not adjacent to the feed stage, then, depending on the number of stages in between, we have

With one stage in between

$$\frac{l_{\phi i}}{d_i} = A_{\phi i}\left(\frac{A_{Ri}}{1 - A_{Ri}} + 1\right) = \frac{A_{\phi i}}{1 - A_{Ri}}$$

With two stages in between

$$\frac{l_{\phi i}}{d_i} = A_{\phi i}\left(1 + \frac{A_{\phi-1,i}}{1 - A_{Ri}}\right)$$

With $m + 1$ stages

$$\frac{l_{\phi i}}{d_i} = A_{\phi i}\left\{1 + A_{\phi-1,i}\left[1 + \cdots A_{\phi-m,i}\left(\frac{1}{1 - A_{Ri}}\right)\right]\right\} \tag{22}$$

A similar equation can be derived for the stripping section of the column:

$$\frac{v_i}{b_i} = S_{\psi i}\left\{1 + S_{\psi+1,i}\left[1 + \cdots S_{\psi+m,i}\left(\frac{1}{1 - S_{si}}\right)\right]\right\} \tag{23}$$

where

$$S_{\psi i} = \left(\frac{VK_i}{L}\right)$$

and S_{si} is the i^{th} component stripping factor at the stripping pinch zone. The component recoveries are related to the stripping factors (or rectifying factor) by the following equations:

$$G_i = \frac{\dfrac{l_{\phi i}}{d_i}}{\dfrac{l_{\phi i}}{d_i} + 1} \tag{24}$$

$$U_i = \frac{\frac{v_{\psi i}}{b_i}}{\frac{v_{\psi i}}{b_i} + 1} \qquad (25)$$

Equations (22) through (25) are valid for both class one and class two problems. The basic concept of this general pinch equations lies in the use of Equation (21) to express an infinite series. Other terms in Equations (22) and (23) are merely corrections for moving the infinitely long pinch zones away from the feed stage. For this reason, the quantities A_{Ri} and S_{si} must be very accurately determined especially for those components with recoveries close to 1.

Since component stripping (or rectifying) factors do not vary drastically from stage to stage, Bachelor (1957) assumed that

$$A_{Ri} = A_{\phi - m,i}$$

and

$$S_{si} = S_{\psi + m,i}$$

for an arbitrary m. Erbar and Maddox (1962) used plate-to-plate calculations from the terminals of the column to the pinch zones to obtain the A_{Ri}'s and S_{si}'s. Their method is very time consuming and subject to severe roundoff errors. A large number of plate material and energy balance calculations is required, and each is subject to a finite error equivalent to the convergence criteria. Neither Bachelor nor Erbar and Maddox applied the flash calculation as specified by Equation (7) for the feed stage.

Before we present a method to obtain the A_{Ri}'s and S_{si}'s, the overall algorithm is presented in the following section.

General Minimum Reflux Algorithm

This algorithm is general in the sense that it works for both class one and class two problems and that it is independent of the phase equilibrium and enthalpy models. If physical property models are rigorous, the converged solution also will be rigorous. Obviously, the solution can be only as accurate as the physical property models.

The algorithm is shown below. Each operation in the algorithm will be discussed in detail in the sections that follow.

1. Estimate component relative volatilities $\alpha_{\phi i}$'s and $\alpha_{\psi i}$'s.
2. Assume all components distributed (for four iterations). Use Equations (18) and (19).
3. Use the method for solving column balances based on the given recoveries and obtain reflux ratio.
4. Return to step 2 for four iterations using the latest calculated $\alpha_{\phi i}$'s and $\alpha_{\psi i}$'s.
5. Use calculated terminal conditions to find pinch zone conditions, A_{Ri}'s and S_{si}'s.
6. Use plate-to-plate calculations to find stripping and rectifying factors near the feed stage. Start with Equation (21) and include one more stage to the sequence [Equations (22) and (23)] after each iteration in steps 3 to 8.
7. Compute G_i's and U_i's with Equations (24) and (25).
8. Use a convergence acceleration method to estimate a new set of G_i's and U_i's and return to 3.

Steps 1 through 4 have been discussed in preceding sections. They provide solutions to class one problems as well as initial estimates for the general algorithm. Steps 3 through 8 are working parts of the general algorithm. The basic approach is as follows. Based on a set of estimated recoveries, the overall and the feed stage material and energy balances together with the reflux ratio can be calculated in step 3. The calculated feed stage conditions and terminal conditions are then used in steps 5 and 6 to generate terms for the pinch Equations (22) to (25). These pinch equations generate a set of calculated recoveries. If these calculated values are close to those estimated by certain given criteria, convergence has been achieved. If not, step 8 is used to generate a new set of estimated recoveries. Details for steps 5, 6, and 8 are discussed below.

Determining Pinch Zone Conditions

As mentioned earlier, the stripping and rectifying factors at the pinch zones must be accurately calculated for use in the pinch equations. It has also been mentioned that the method proposed by Erbar and Maddox suffers severe roundoff errors. One way to reduce roundoff errors in a multistage equilibrium calculation is to lump the stages into an integral section. Consider the section of a column between the reboiler and the stripping pinch zone. The component material balances can be written as follows:

$$L_s x_{si} = b_i + V_s y_{si}$$

or

$$x_{si} = \left(1 - \frac{V_s}{L_s}\right) x_{b_i} + \frac{V_s}{L_s} y_{si}; \quad i = 1, \cdots, n \qquad (26)$$

At the pinch zone, the compositions do not change. Therefore, the equilibrium relationship may be written as

$$y_{si} = K_{si} x_{si}; \quad i = 1, \cdots, n \qquad (27)$$

The enthalpy balance is

$$L_s h_s = V_s H_s + B h_b - Q_b \qquad (28)$$

where Q_b is the amount of heat added per unit time. The flash equation is the same:

$$\sum_i (K_{si} - 1) x_{si} = 0 \qquad (29)$$

The enthalpy balance, Equation (28), determines the slope of the operating line, Equation (26). The solution is determined by the intercept between the operating line and the equilibrium line, Equation (27). Equation (29) is necessary to determine T_s.

The above sets of equations again closely resemble adiabatic flash equations. However, from a numerical solution point of view, Equations (26) to (29) are less stable. In the flash calculation, the unknowns in the material balance equations (the operating line), y's and x's, are on the same side of the equal sign. Therefore, the operating line of a flash calculation has a negative slope. Since the equilibrium lines usually have a positive slope, at interception the two lines are nearly perpendicular. For the pinch zone calculation, Equations (26) and (27) often are nearly parallel at interception.

Rewriting Equation (26) as

$$x_{si} = \frac{1 - \frac{V_s}{L_s}}{1 - K_{si}\frac{V_s}{L_s}} x_{bi}$$

one finds that as x_{bi} approaches zero, $K_{si}\ (V_s)/(L_s)$ approaches one for a finite x_{si}. In other words, if the operating line intercepts the origin $y = x = 0$, the slope of the equilibrium line K_{si} and that of the operating line L_s/V_s become identical, and x_{si} becomes indeterminate.

A simple solution is to write the equilibrium Equation (20) as

$$y_{si} = m_{si}x_{si} + c_{si}; \quad i = 1, \cdots, n \tag{30}$$

where m_{si} is the new slope of the equilibrium line. Equation (30) can be designed to fit the slope of either isobaric or isothermal systems and viewed as local tangent planes to the equilibrium surfaces. Since K_{si} is usually expressed in terms of x_{si} and other variables as in Equation (5), m_{si} must also be related to K_{si} and x_{si}. We chose to relate m_{si} and K_{si} as follows. In the immediate neighborhood of x_{si}, one may assume constant relative volatilities, or

$$y_{si} = \frac{K_{si}x_{si}}{\Sigma\, K_{sj}x_{sj}} \tag{31}$$

The slope m_{si} can be determined as

$$m_{si} = \frac{dy_{si}}{dx_{si}} = \sum_j \frac{\partial y_{si}}{\partial x_{sj}} \frac{\partial x_{sj}}{\partial x_{si}} \tag{32}$$

The slope should be determined along the line between points $x_{sio} = 1$ and $x_{si} = x_{si}$, or

$$\frac{\partial x_j}{\partial x_i} = \frac{x_{sj} - x_{sjo}}{x_{si} - x_{sio}} = \frac{x_{sj}}{x_{si} - 1}$$

Substituting Equation (31) to Equation (32), we get

$$m_{si} = \frac{K_{si}}{\sum\limits_j K_{sj}x_{sj}} - \frac{K_{si}{}^2x_{si}}{\left(\sum\limits_j K_{sj}x_{sj}\right)^2} + \sum_{\substack{j\\ j\neq i}} \frac{K_{si}x_{si}K_{sj}}{\left(\sum\limits_l K_{sl}x_{sl}\right)^2} \frac{x_{sj}}{(1 - x_{si})} = \frac{K_{si}}{\sum\limits_j K_{sj}x_{sj}} - \frac{K_{si}x_{si}}{\left(\sum\limits_j K_{sj}x_{sj}\right)^2} \frac{1}{(1 - x_{si})}\left(K_{si} - \sum_j K_jx_{sj}\right) \tag{33}$$

and

$$c_{si} = \frac{K_{si}x_{si}}{\left(\sum\limits_j K_{sj}x_{sj}\right)^2} \frac{x_{si}}{(1 - x_{si})}\left(K_{si} - \sum_j K_jx_{sj}\right) \tag{34}$$

The familiar adiabatic flash algorithm is restated as follows:

1. Estimate x_{si}'s, y_{si}'s.
2. Estimate V_s/L_s and T_s.
3. Calculate K_{si} [Equation (5)], m_{si}, c_{si}.
4. Calculate

$$x_{si} = \frac{\left(1 - \frac{V_s}{L_s}\right)x_{bi} + \frac{V_s}{L_s}c_{si}}{1 - \frac{V_s}{L_s}m_{si}}$$

and y_{si} [Equations (30)].

5. Return to step 3 with new estimated T_s and normalized x_{si}'s and y_{si}'s if Equation (29) is not satisfied.
6. Calculate enthalpies.
7. Return to step 4 with new estimated V_s/L_s if Equation (28) is not satisfied.

After convergence, all component stripping factors including those of the nondistributed ones at the pinch zone, S_{si}'s, are calculated. Note that each S_{si} must not be greater than one.

The method described in this section can be similarly applied to the rectifying pinch calculation to obtain A_{Ri}'s.

Removal of Nondistributed Components

Nondistributed components in the feed must be removed by stages between the feed stage and the respective pinch zone. The removal calculation is needed to find the nondistributed component recoveries and to correct the distributed component recoveries through Equations (22) and (23). These later quantities are needed for the reflux ratio calculation.

In general, only a few stages are needed before the reflux ratio closely approaches its final value. Reasons are as follows. For components with small recoveries, Equations (22) and (23) converge in a few stages; for components with large recoveries, the concentrations reduce quickly from stage to stage and become insignificant to alter reflux rates. We found empirically that in most cases four or five stages on each side of the feed stage are adequate. In the algorithm, for nondistributed problems, a stage is added to either above or below the feed (or both) after each iteration until a preset maximum number has been reached. The use of a maximum number of stages for the removal of nondistributed components is merely a way to save computer time. Pinch zones are assumed to be adjacent to the last stage in the feed section. If for any reason the computation could not proceed to the last stage, the pinch zone is assumed to be adjacent to the last successfully calculated stage. The procedure to calculate stage material and energy balances is quite straightforward but time consuming. The following procedure is used to calculate vapor to and liquid from the stage ψ.

1. Calculate the dew point liquid composition $x_{\psi i}$ for $vv_{\psi i}$'s.
2. Estimate L.
3. Calculate $v_{\psi-1,i}$'s from material balances.

Table 1. Material Balance for the Example

Component No.	Feed (kgmole/hr)	Dist (kgmole/hr)	Botm (kgmole/hr)	ln α	α
1	12	12	0	1.5	4.4817
2	448	442	6	1	2.7183
3	36	13	23	0	1
4	15	0	15	−0.5	0.6065
5	23	0	23	−1.5	0.2231
6	39.1	0	39.1	−2.5	0.0821
7	272.2	0	272.2	−3.5	0.0302
8	31	0	31	−4.5	0.0111
Total	876.3	467	409.3		
Temp, °C	138.9	2.3	95.6		
Press, mmHg	775.5	775.5	775.5		
Vapor fraction	1	0	0		

4. Calculate the dew point of $v_{\psi-1,i}$'s and then $H_{\psi-1}$.

5. Return to 2 if stage enthalpy is not balanced.

Note that the above procedure should also be used to compute the reflux rate for a given distillate flow and a condenser duty. The procedure to calculate stage material and energy balances above the feed stage is basically the same and hence is not repeated here.

Since $v_{\psi i}$'s and l_{wi}'s are calculated by using overall component recoveries, it may be impossible sometimes to reach conditions at the bottom of the column from those at stage ψ. The reflux may be slightly less than the minimum, and conditions at two points of the column will diverge from each other in stage-to-stage material balance calculations. As iterations are made on successive stages down the column from stage ψ, a stage is reached where simultaneous heat and material balances become impossible.

If and when such conditions occur, tray-by-tray balances can no longer proceed; the algorithm will then truncate the series Equations (22) and (23) at the last successfully balanced stage and assume that the pinch zone follows immediately. There is perhaps intuitive justification to truncate the series at the stage next to the last, since the last stage may have already been unreasonable. Unfortunately, these different methods of handling temporary infeasible conditions sometimes result in slightly different solutions. In the example problem presented in a later section, the results are 1.0071 and 1.0075 kg mole/hr respectively, for using the last or the next to the last stage. This is not a significant difference from a process design point of view.

Convergence Acceleration

For a given set of G_i's and U_i's, this algorithm first computes overall material and energy balances. From these balances, it proceeds to compute component stripping and absorption factors based on tray-by-tray material and energy balances from the feed stage and on sectional material and energy balances between column terminals and pinch zones. These stripping and absorption factors are then used to compute new G_i's and U_i's. Hopefully, these new G_i's and U_i's calculated are closer to the solution than those initially estimated. If the estimated are the same as the calculated or close to each other by less than a set limit, a solution has been achieved. If not, the calculated values would be used as the new estimated values for the next iteration in a direct iteration procedure. In our experience. the direct iteration procedure was slow and sometimes unstable. In this algorithm, a bounded Wegstein convergence acceleration method is used to improve solution speed and stability.

Since U_i's and G_i's are used in functions involving terms of

$$\frac{1}{1-U_i} \quad \text{and} \quad \frac{1}{1-G_i}$$

the effects of roundoff errors are very large for U_i's and G_i's close to unity. Therefore, instead of using the common technique of switching to the direct iteration method whenever the estimated values for two successive iterations are very close to each other, we use the following criterion. If

$$\left| \frac{U_{ie1} - U_{ie2}}{(1 - U_{ie2})^2} \right| < 1.E - 5$$

direct iteration would be used for the i^{th} component.

In most cases, limiting the slope to a maximum of zero was found to be the best for the Wegstein method in this application.

Example

An example problem from a paper by Bachelor (1957) is shown. It involves the following eight components: *i*-butane, *n*-butane, *i*-pentane, *n*-pentane, *n*-hexane, *n*-heptane, *n*-octane, and *n*-nonane. To simplify the problem and to eliminate unnecessary complications, the component physical properties are changed as follows.

1. Vapor pressures are made proportional to those of the base component to form a system of constant relative volatilities. The relative volatilities are further simplified to those shown in Table 1. Vapor pressures of the base component are expressed by an Antoine equation:

$$\log P^o{}_{ic5} = 6.78967 - \frac{1\,020.012}{t^oc + 233.097}$$

2. To eliminate the composition effect on the enthalpy, all component enthalpies are made equal to those of the base component.

The material balance which also describes the separation specification is shown in Table 1.

Most engineers understand the limitations associated with the constant relative volatility assumption. The majority of them, however, do not have the same understanding with the constant molal overflow assumption. Through this method, serious consequences have been found when constant overflow was assumed. The following results show how the Underwood method can underestimate the minimum reflux ratio by 46%, because of the constant overflow assumption.

With vapor feed at its dew point, the Underwood method calculates a minimum reflux ratio of 1.0078. Using temperature independent enthalpies to simulate constant molal overflows in the column, this algorithm calculates a minimum reflux ratio of 1.007. The agreement is excellent between the two methods for an ideal system for which the Underwood method is designed. As temperature effects on the enthalpies are restored, the constant molal overflow assumption is no longer valid. In this case, this method calculates a minimum reflux ratio of 1.884. Apparently, additional reflux is needed to quench a hot feed, even though the reflux is quite cold. A hot feed temperature is required to keep the high boilers in the vapor phase. This large error in using a short-cut method is therefore primarily the result of a wide boiling feed. The error will be opposite in direction for bubble point feeds, because the low temperature feed and the low temperature reflux will generate large internal refluxes.

In order to avoid these errors, it is recommended that a rigourous method be used for all minimum reflux calculations except where it is known that relative volatilities and molal overflows are constant.

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R. V. Sanderson assisted in the attempt of reproducing the work of Erbar and Maddox (1962).

NOTATION

A = component rectifying factor
B = bottoms flow
b = bottom component flow
c = constant in a locally linearized equilibrium equation
D = distillate flow
d = distillate component flow
F = feed flow
f = feed component flow
G = component recovery above the feed

g = relative recovery above the feed
H = vapor molal enthalpy
h = liquid molal enthalpy
K = vapor-liquid equilibrium composition ratio
L = liquid flow
l = liquid component flow
m = slope in a locally linearized equilibrium equation
p = pressure
Q = heat duty
S = component stripping factor
T = temperature
U = component recovery below the feed
u = relative recovery below the feed
V = vapor flow
v = vapor component flow
x = liquid mole fraction
y = vapor mole fraction
α = relative volatility

Subscripts

b = reboiler
c = condenser
e = estimated
h = heavy key
i = component index
l = light key
R = rectifying pinch
S = stripping pinch
w = feed stage
ϕ = stage above feed
ψ = stage below feed

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Convective Heat Transfer in Gas-Solid Suspension Flow Through Packed Beds

A correlation for the convective heat transfer between a bed of metallic oxides and a flowing gas-solid suspension was obtained experimentally. Four industrially important commercial catalysts with different physical, thermal, and transport properties were used as the bed materials. A wide range of air flow rates and solids to gas loading ratios were used. The correlating parameters, to estimate the convective Nusselt number, were found to be the Reynolds number, the Archimedes number, the solids loading ratio, and the shape factor of the packing materials. Impressive increases in heat transfer rates were observed with increased solids loading.

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In highly exo or endothermic gas phase solid catalyzed reaction systems, it is necessary to achieve enhanced convective heat transfer rates to obtain temperature control. One way of doing this is to entrain solid fines in the gas flowing through the bed. While a considerable amount of work has been done on gas-solid suspension flow through pipes and conduits (summarized by Depew and Kramer, 1973), very little information is available on the convective heat transfer in gas-solid suspension flow through packed beds.

This paper presents an experimental study to obtain a correlation for the convective heat transfer between the bed and the flowing gas-solid suspension. An experimental technique using microwave heating similar to that used by Balakrishnan and Pei (1974) in their studies on convective heat transfer from packed beds to gas flow alone was employed. The use of microwave power to heat a bed of metallic oxides results in the entire bed attaining a constant uniform temperature almost instantaneously. This eliminates thermal gradients within the bed and thereby the conduction mode.

Four industrially important commercial catalysts with different physical, thermal, and transport properties were used as the bed materials. Glass fines in air was used as the gas-solid suspension. Glass was chosen because it hardly absorbs any microwave power. A wide range of air flow rates and solids to gas loading ratios were used.

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