

General Short-cut Equation for Equilibrium Stage Processes

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An equation for the prediction of the separation of a multicomponent mixture in equilibrium stage processes is presented. The equation is applicable to extraction processes with up to three feeds (an intermediate feed, a feed at the extract end, and the solvent feed at the raffinate end) and both raffinate and extract reflux. In its simplest form (simple extraction, stripping, or absorption) the equation reduces to the familiar Kremser equation. The use of the equation to solve multicomponent distillation and extraction problems is illustrated by examples.

Shortcut methods for the approximate solution of multicomponent, multistage, separation problems continue to serve useful purposes even though computers are available to provide rigorous solutions. In some problems the available equilibrium data may not be sufficiently accurate to justify the longer rigorous methods. In design studies a large number of cases can be worked quickly by a short-cut method to pinpoint the optimum conditions and then the exact solution found by some longer rigorous method. Such a procedure saves valuable computer time, since the time required for the rigorous method usually exceeds that for the short-cut method by a factor of ten or more. If a computer is not available, the designer usually must rely on the short-cut method alone.

This paper presents an analytical short-cut equation which is applicable to all equilibrium stage processes with any number of stages and handling any number of components. It is derived for an extraction column with three feeds (an intermediate feed, a feed at the extract end, and the solvent feed at the raffinate end) plus both raffinate and extract reflux, and it takes into account the degree of separation obtained for each component in the extract solvent recovery device. The complex equation for this complicated process reduces to correspondingly simpler expressions for operations which have fewer feeds and refluxes. Alders (1) and Tiller (12) have presented some of the simpler forms for extraction. In the case of a simple absorption or stripping column, the equation reduces to the familiar Kremser (6) equation. The forms for distillation and the more complicated extraction processes have not appeared before in the literature.

NOMENCLATURE

This section also applies to the next article in this issue.

Standard chemical engineering symbols (11) are used for the streams and

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concentrations. Standard usage requires that extract and raffinate concentrations be denoted as x and y respectively. This convention permits the same equations to be written for extraction as for distillation, absorption, and stripping.

The convention of numbering the stages from the bottom for the vapor-liquid processes (distillation, absorption, and stripping) and from the extract end for extraction is used in this paper. For the sake of uniformity an extraction column or train will always be depicted with stage 1 (extract end) at the bottom regardless of the actual direction of flow. In those extraction problems, where the extract is actually the lighter phase, the phrase stages below the feed stage should be interpreted as stages between the feed stage and the extract end.

Figure 1 is a sketch of the most complicated process considered. Note the use of N to designate the total number of stages and the use of M to denote the number of stages below the feed stage. The intermediate feed stage is designated as the $M + 1$ stage.

STATEMENT OF PROBLEM

This section also applies to the next article in this issue.

The number of design variables which must be specified by the designer of a multicomponent, multistage, separation process can be calculated by the method of Kwauk (7). In the solution method described in this paper the following variables are always specified: pressure in each stage; heat leak in each stage; the rate, temperature, pressure, and composition of all feed streams (including the solvent); total number of stages; and number of stages below the feed. In the case of an extraction process with extract reflux, it is also always necessary to specify g for each component in such a manner as to ensure a single phase extract product and reflux.

In addition to the variables which are always specified, it will be necessary to specify certain of the following: reflux ratios, product rates, maximum

phase rates within the column or train, recovery of certain components, heat load in reboiler or condenser, and temperature at some point. Once the correct number of design variables has been specified, the complete set of product rates and compositions can be calculated.

This manner of defining the problem has the disadvantage of not permitting the direct calculation of the required solvent-to-feed ratio or the number of stages required to obtain a desired separation or product purity, but it does simplify the convergence problem when a computer is used. If a new column is being designed, several cases must be calculated with specified solvent-to-feed ratios, number of stages, and reflux ratios until the desired separation or purity is obtained. However most design problems involve the study of several cases to define the optimum conditions for economic appraisal, and therefore the reliance on the case-study approach is not a serious drawback. If desired, the short cut calculation procedure can be rearranged to give N and the solvent rate directly, but this is not covered in this paper. If the performance of an existing unit is being studied, the designer usually will have no control over the first five specifications and the above method of defining the problem is the only possible one. Also this approach eliminates the need of worrying about minimum solvent treats and minimum refluxes.

REPRESENTATION OF EQUILIBRIUM DATA

The equilibrium data is utilized in the form of the distribution coefficient $K = y/x$. In the short-cut equations the K 's always appear as part of the stripping factors S_n or S_m . Ternary liquid-liquid data can be represented by plotting the K 's vs. the concentration of any one of the three components. Quaternary liquid-liquid data can be represented satisfactorily by plotting the K 's vs. the major solvent component (lowest K values) with concentration parameters of the component which has the next lowest K values. The correlation of the K values vs. two phase concentrations is justified by the phase rule. A four component, two phase systems (a vapor phase cannot exist if the system pressure is

greater than the vapor pressure of the liquid phases) has a variance of four. Once the temperature and pressure are specified, the specification of two-phase concentrations is sufficient to define one unique state of the system and fix all the distribution coefficients. Work is underway to develop a general correlation scheme for nonideal solution systems having more than four components, and it is hoped that the results will warrant future publication.

SHORT-CUT EQUATION*

The material presented below indicates the general method of attack.

The calculus of finite differences can be used to relate the concentration of any given component to the stage number. The distribution coefficient and the phase rates must be assumed constant within the column section under consideration if a simple analytical solution of the difference equation is desired. If these assumptions are made, the following equations can be written for each component. For the upper (or raffinate) end

$$x_n = C_1(S_n)^n + C_2 \quad (1)$$

For the lower section (or extract) end

$$x_m = C_3(S_m)^m + C_4 \quad (2)$$

Eliminating the constants in Equations (1) and (2) by component balances around stages N and 1 respectively, and then combining the two equations with a component balance around the feed stage, one gets the following general expression for the process pictured in Figure 1:

$$f = \frac{(1-S_n^{N-M}) + q_s(S_n^{N-M}-S_n) + R(1-S_n) + hq_{R'}S_n^{N-M}(1-S_m^M)}{(1-S_n^{N-M}) + hS_n^{N-M}(1-S_m^M) + R(1-S_n) + h\left(\frac{1+R'}{1+gR'}\right)S_m^MS_n^{N-M}(1-S_m)} \quad (3)$$

Equation (3) predicts the fraction of any given component which will be recovered at the lower (or extract) end of the column. The fractional recoveries of the various components will vary as their respective stripping factors vary. At $S = 1.0$, Equation (3) and all of its simpler forms take on the indeterminate form $0/0$. The value of f at $S = 1.0$ is found by differentiating the numerator and denominator and evaluating the new fraction thus obtained.

For processes simpler than the one pictured in Figure 1 the applicable form of Equation (3) is correspondingly simpler. For a simple extraction

or absorption process having only one feed and no refluxes, ($R = R' = F' = q_{R'} = M =$ zero and $S_n = S_m$), Equation (3) reduces to the following form of the Kremser equation:

$$f = \frac{1 - q_s S - S^N (1 - q_s)}{1 - S^{N+1}} \quad (4)$$

For a component entering only in the feed $q_s = 0$, and

$$f = \frac{1 - S^N}{1 - S^{N+1}} \quad (4a)$$

If $q_s = 1.0$, the component enters only in the solvent, and

$$f = \frac{1 - S}{1 - S^{N+1}} \quad (4b)$$

The expression for an extraction process with one feed and raffinate reflux is also quite simple. For $q_s = 0$

$$f = \frac{1 - S^N + R(1 - S)}{1 - S^{N+1} + R(1 - S)} \quad (5a)$$

For $q_s = 1.0$

$$f = \frac{1 - S + R(1 - S)}{1 - S^{N+1} + R(1 - S)} \quad (5b)$$

Casual inspection of Equations (5a) and (5b) might indicate that the use of raffinate reflux affects the recovery of a given component. However as R changes, the stripping factor must also change, and the net result is no change in f . This can be easily demonstrated for a one-stage process. The represen-

proximate the correct recovery. Average K values are assumed for each component, and these are combined with a set of assumed or specified phase rates to provide the initially assumed stripping factors. The stripping factors are used with the appropriate form of Equation (3) to predict a recovery, f , for each component. The recoveries are used to calculate better approximations of product rates and compositions. A new set of K values are obtained from the new end compositions and are combined with the new stream rates to provide the stripping factors for the next trial.

Convergence is rapid, and two trials are usually sufficient if the initial assumptions are of the correct order of magnitude. If the initially assumed rates differ widely from the rates calculated in the first trial, it is necessary in some cases to repeat the first trial with the same set of K values and more reasonable rate assumptions. The extraction example presented later illustrates the effect of poor, initial product-rate assumptions on the speed of convergence. The initially assumed extract product rate was off by a factor of 20 (50 compared with 2.5), but three trials were sufficient to obtain satisfactory convergence.

In computer-logic terms the solution of Equation (3) involves two major loops. The first, or inside, loop calculates the set of stream rates and end compositions which correspond to the set of assumed K values. Complete convergence can be obtained in this loop if desired; that is there is only one set of rates and compositions for any given set of assumed K 's. The second, or outside, loop uses the compositions obtained in the first loop to calculate a better set of K values and then returns the calculations to the first loop. Convergence is not so well defined in the outside loop. Each correction in the K values results in a subsequent correction in the next trial in the same direction, and if too many trials are made, the solution may gradually drift away from the correct one.

Solution time is shortened in stripping, absorption, and extraction calculation by making only one trial for each set of K values. Complete convergence in the inside loop is never desired in distillation calculations if the product rates are specified. The K values are adjusted after every trial until the rate specifications are met.

APPLICATION TO DISTILLATION

The form of Equation (3) applicable to a distillation column with a total condenser is obtained by setting $q_s = q_{R'} = R' = g = 0$ and combining the second and fourth terms in the denominator to give

* The complete derivation is available as document 6352 with the American Documentation Institute, Photoduplication Service, Library of Congress, Washington 25, D. C., and may be obtained for \$1.20 for photoprints or for 35-mm. microfilm.

TABLE 1. COMPARISON WITH GRAPHICAL SOLUTION RESULTS
FOR TWO TERNARY EXTRACTION SYSTEMS

Comparison I

Process: One feed. No reflux. Eight stages.

System: Acetone (A)—Chloroform (C)—Water (S) at 25°C.

Range of K 's: $A = 1.8$ – 3.7 $C = 35$ – 143 $S = 0.009$ – 0.028

Method	B	D	x_A	x_C	x_S	y_A	y_C	y_S
Graphical	204.1	52.4	0.221	0.015	0.764	0.090	0.900	0.010
Analytical	199.3	57.2	0.211	0.009	0.780	0.139	0.841	0.019

Comparison II

Process: Two feeds. No reflux. 5.4 stages.

System: Acetone (A)—Water (C)—Monochlorobenzene (S) at 25°C.

Range of K 's: $A = 0.93$ – 0.99 $C = 154$ – 465 $S = 0.0016$ – 0.0027

Method	B	D	x_A	x_C	x_S	y_A	y_C	y_S
Graphical	141.2	89.6	0.140	0.006	0.854	0.010	0.989	0.001
Analytical	140.8	90.0	0.139	0.005	0.856	0.011	0.987	0.002

$$f = \frac{(1 - S_n^{N-M}) + R(1 - S_n)}{(1 - S_n^{N-M}) + R(1 - S_n) + hS_n^{N-M}(1 - S_m^{M+1})} \quad (6)$$

Note that Equation (6) is the equation written earlier for an extraction column with one feed and raffinate reflux but with an additional term in the denominator to take into account the second section of the column present in a complete distillation column.

Equation (6) is applicable to distillation columns having either a partial reboiler or heat input to the bottom plate. The lowest equilibrium stage must always be numbered one regardless of whether it is a partial reboiler or the bottom theoretical plate. Equation (6) is not strictly applicable to a column with a partial condenser since it ignores any possible difference between the overhead and reflux compositions. However the effect of a partial condenser can be closely approximated by increasing N by 1.0.

The distillation problem, particularly if it concerns an existing column, is conveniently defined by specifying distillate rate, D/F ; maximum allowable vapor rate, V/F , at some point in the column; and condenser load equal to the latent heat of condensation of V_N in addition to the first five specifications listed in Statement of Problem.

If ideal solutions are assumed, the K values are functions of temperature only at the specified tower pressures. Estimation of the K values therefore reduces to the estimation of the temperature profile. Good initial estimates of the top and bottom temperatures are obtained by splitting the feed in a reasonable manner and performing dew- and bubble-point calculations on the resulting overhead and bottom streams. The feed tray temperature can be estimated roughly by a bubble-point, dew-point, or flash calculation on the feed depending upon the thermal condition. However it is easier, and often as accurate, to read the initially assumed feed tray temperature from a

straight line drawn between the assumed top and bottom temperatures.

The liquid and vapor rates are assumed to be constant within any column section, and their values are fixed by the specification of the feed, maximum vapor rate, distillate rate, and the thermal condition of the feed. Since the phase rates remain constant from trial to trial, the stripping factors can be varied only by changing the K values. This is done by adjusting the temperature profile. The top and bottom temperatures cannot be adjusted arbitrarily since they must always correspond to the dew and bubble points of the overhead and bottom streams respectively. The iteration variables therefore reduce to one, the feed tray temperature. The calculator must adjust this temperature until the vapor- and distillate-rate specifications are met. This method of calculation is analogous to the technique of a column operator who adjusts the control tray temperature until the column is loaded or until the product specifications are met.

Consider as an example the following typical plant problem. A bubble-point feed which contains 5% C_s , 15% $i-C_s$, 25% $n-C_s$, 20% $i-C_s$, and 35% $n-C_s$ is to be fed to a column with ten theoretical stages, feed on the sixth stage from the bottom, a total condenser and a maximum top vapor capacity of about 1.75 times the amount of feed to be charged. The column pressure is 120 lb./sq. in. abs. Curves which relate the losses of $n-C_s$ and $i-C_s$ in the bottom and overhead product streams respectively to the overhead rate must be developed to serve as a basis for economic calculations to pinpoint the optimum overhead rate.

Only one point on the curves (at $D/F = 0.489$) will be considered. Specification of D/F and the limitation

of V_N/F to 1.75 fixed the phase rates per 100 moles of feed at $V = V' = 175$, $L = 126.1$, and $L' = 226.1$. Also $B = 51.1$ and $R = 2.581$. The initially assumed temperature profile based on bubble- and dew-point calculations as described previously was 165°F. at the top, 185°F. at the feed, and 236°F. at the bottom. Average K values for each section were evaluated at the arithmetic average temperature for that section. The 165–185–234°F. (top-feed-bottom) profile resulted in calculated V_N and D rates of 162.3 and 42.5 respectively compared with the specified values of 175 and 48.9. The assumed temperature profile for the second trial was 165–210–236°F., and V_N and D rates of 177 and 49.5 were calculated. Once the specifications have been approached this closely, the calculated end compositions can be used to provide better end temperatures, by means of dew- and bubble-point calculations. The final temperature profile (third trial) was 163.5–210–234°F., and this profile met the V_N and D specifications exactly. A rigorous computer solution

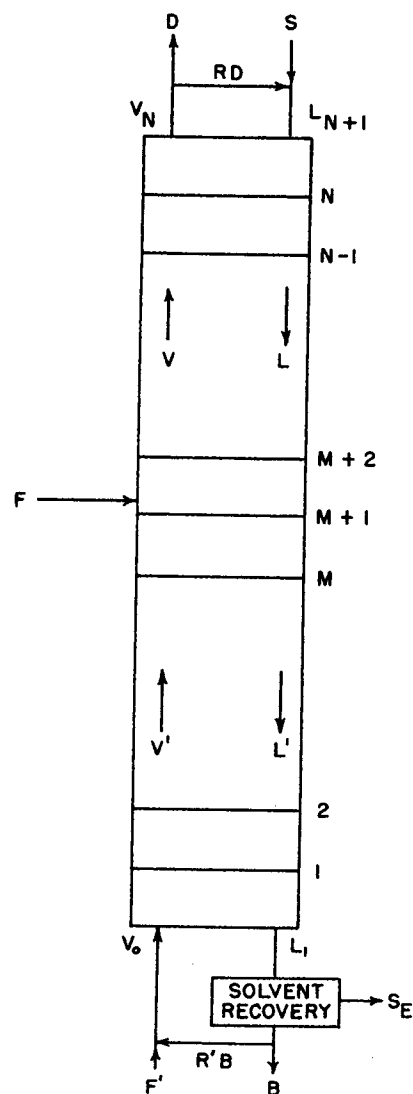


Fig. 1. Process variables and nomenclature.

of the same problem by the Thiele-Geddes method (8) provided temperatures of 166°, 202°, and 234°F. for the top, feed, and bottom stages. The rigorous solution predicted the recovery of nC_4 and iC_5 in the overhead as 92.5 and 17.9% respectively. Equation (6) predicted 90.5 and 17.9%.

The major advantage of Equation (6) over other short-cut distillation equations (3, 5) is that it provides a good estimate of the feed tray temperature as well as the end temperature. Knowledge of the intermediate temperature permits the estimation of average K values for each section rather

than for the entire column. Also the three point temperature profile is helpful if the short-cut results are to be used as the initial assumptions for a rigorous computer solution. Another advantage of Equation (6) is that it makes its predictions at a specified reflux ratio rather than at total or minimum reflux. Probably the most widely used short-cut distillation method at the present time involves a combination of the Fenske total reflux equation (3), the Underwood minimum reflux equations (14), and the Gilliland (4) correlation. This method does not provide an estimate of the feed tray tem-

perature and when applied to the above example predicts the nC_4 and iC_5 recoveries in the overhead product as 96.5 and 17.1% respectively.

The individual K values for the first trial were obtained by an arithmetic average of all the experimental values available. To illustrate that the speed of convergence is little affected by the initial rates assumed, the feed was assumed to split equally between the overhead and bottom products, D and B . In all but very high solvent treat cases the extract phase rate can be assumed to vary linearly from the solvent, $S = L_{N+1}$, to L_1 . The raffinate phase rates were then calculated by material balances. Average phase rates for each section were obtained by averaging the phase rates at the ends of the section. Stripping factors for each component were then calculated from the average rates and K values for use with Equation (7) to predict the various component recoveries.

The amounts of each component leaving each end of the column were calculated from the following definitions of f :

$$f = \frac{(1-S_n^s) + q_s (S_n^s - S_n)}{(1-S_n^s) + h S_n^s (1-S_m^s) + h \left(\frac{1+R'}{1+gR'} \right) S_m^s S_n^s (1-S_m)} \quad (7)$$

perature and when applied to the above example predicts the nC_4 and iC_5 recoveries in the overhead product as 96.5 and 17.1% respectively.

APPLICATION TO EXTRACTION

Immediately after its derivation the correctness of Equation (3) was checked by solving several ternary extraction problems and comparing the results with graphical solutions. To conserve space only two of the comparisons are shown in Table 1. The agreement is good and substantiates the validity of Equation (3).

In extraction calculations it is not usually convenient to specify the product rates. Therefore the rates become iteration variables and vary from trial to trial as the K values are changed. Also ideal solutions cannot be assumed, and the K values must be calculated as a function of the phase compositions. The relations between the K 's and the phase compositions are not linear, and an average K for a column section should be obtained by averaging several K values evaluated at various typical stage compositions along the section.

The application of Equation (3) to extraction calculations will be illustrated by the calculation of the recovery of acetone (20 wt. %) from chloroform (80 wt. %) with a mixed solvent composed of water (65 wt. %) and acetic acid (35 wt. %). Experimental tie-line data at 25°C. for this system have been published by Brancker, Hunter, and Nash (2), and additional tie lines have been calculated by Smith (10). A five-stage column or train was specified with the feed introduced in the middle stage. A 1:1 solvent-feed ratio was used, and in order to illustrate one of the more complicated forms of Equation (3) an extract reflux ratio of $R' = 10$ was specified. It was assumed that 98% of the

water and acetic acid in L_1 were recovered in S_E and that 1% of the acetone and chloroform in L_1 appeared in S_E .

The amounts of each component leaving each end of the column were calculated from the following definitions of f :

$$Bx_B + S_E x_{SE} = f(Fy_F + F'y_{F'} + Sx_S) \quad (8)$$

$$Dy_D = (1-f)(Fy_F + F'y_{F'} + Sx_S) \quad (9)$$

The amounts of each component in B and L_1 were then obtained from the following component balances:

$$Bx_B = \left(\frac{1-g}{1+gR'} \right) (Bx_B + S_E x_{SE}) \quad (10)$$

$$L_1 x_1 = \left(\frac{1+R'}{1-g} \right) Bx_B \quad (11)$$

The new end rates and compositions calculated from Equations (8) to (11) permitted the estimation of better values of K , S_n , and S_m for each component for the next trial. The trials were continued until the K values obtained from the results of one trial were es-

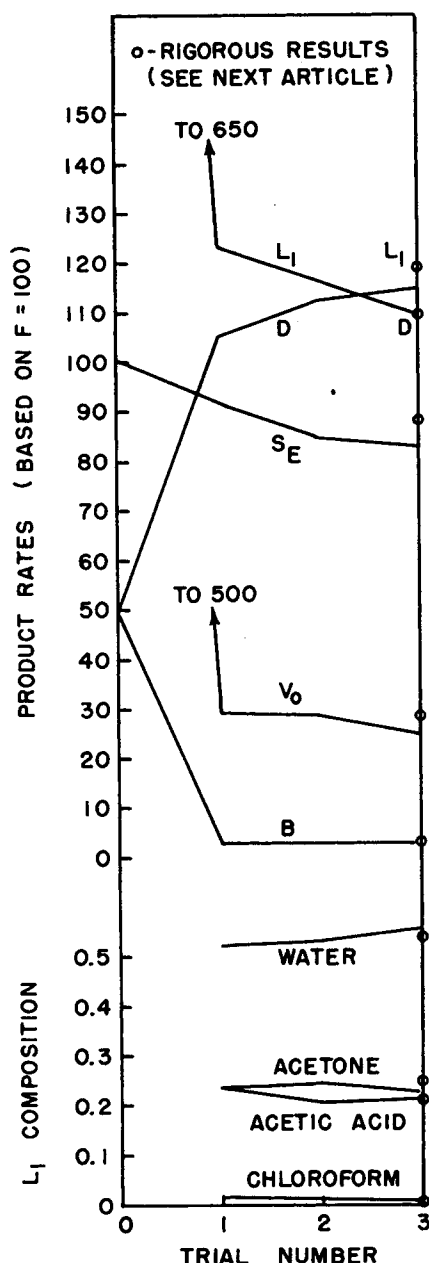


Fig. 2. Partial summary of results from short-cut solution of extraction example. Values at zero trial number are the initial assumptions. Circles at trial number three are results from rigorous method described in next article in this issue.

entially the same as those obtained from the previous trial. Some of the calculated results are shown in Figure 2. Note how erroneous the initially assumed rates were and how reasonable the results of the first trial were despite the poor assumptions. The K and K' values from trial 3 agreed well enough with those from trial 2 to end the calculations. In fact, the calculations could have been stopped after any trial without serious error. Each trial in the example required about 2 hr. calculation time.

The open circles plotted at trial 3 in Figure 2 represent the results of a more rigorous calculation method described in the next paper. Note that the results of trial 2 agree better with the rigorous method results than do those of trial 3. This illustrates the previously mentioned tendency of the short-cut method to drift away from the correct solution if too many trials are made.

The accuracy of the results shown in Figure 2 was checked by the graphical method of Powers (9). The product rates and compositions along with all the stage compositions obtained by the rigorous method were located on the projection of the two-phase surface on the water-chloroform-acetic acid face of the tetrahedron. The difference points were located, and the five stages stepped off. The rigorous method results agreed surprisingly well with the graphical construction considering the practical difficulties encountered in the accurate graphical representation of the phase-equilibrium data for a four-component system.

EXTRACTION PROBLEMS WITH HIGH SOLVENT-TO-FEED RATIOS

Two difficulties arise in problems with high solvent-to-feed ratios. First both the extract and raffinate rates undergo a large change through stage N if raffinate reflux is not used to pre-saturate the entering solvent. Care must be taken to ensure that the average L and V used for the raffinate end of the column are reasonable. It may be better to use V_{N-1} and L_N rather than D and S in the calculation of average rates in the raffinate end. A small error in estimating the large extract rates can cause a large percentage error in the smaller raffinate rates and a corresponding error in the stripping factors. Second a very small error in the predicted f for a solvent component (low K and large f) will result in a large percentage error in D . For example in one system studied the f predicted for component S was 0.9892 compared with the correct value of 0.9972. As a result the calculated concentration of the solvent in D was 0.30 compared with the correct value of

0.073. This difficulty can be simply corrected by checking the calculated solvent concentration in the raffinate against the solubility data and making the needed adjustment. This problem arises only with components which have low K values (solvent components), but it is, of course, possible to use the experimental solubility data to check the product concentration of any of the components.

NOTATION

- A = total amount of component a entering the column
 B = heavy or extract product rate, moles, weight, or volume per unit time
 C_1, C_2, C_3, C_4 = constant coefficients in solutions of difference equations
 D = light or raffinate product rate, moles, weight, or volume per unit time
 E = operator in difference equation
 f = fraction of a given component which is recovered in B (or $B + S_E$ in extraction)
 F = upper feed rate, moles, weight, or volume per unit time
 F' = lower feed rate if only one feed is used, $F' = 0$ and $M = 0$
 g = $S_E x_{SE}/L_1 x_1$ = fraction of a given component in L_1 which is removed with S_E
 h = $(L - KV)/(L' - K'V')$ if intermediate feed is similar to light or raffinate phase ($K_{M+1} = K$). Use

$$h = \frac{(L/K) - V}{(L'/K') - V'}$$

if intermediate feed is similar to heavy or extract phase ($K_{M+1} = K'$). If the column contains only one section, $h = 1.0$

- K = y/x = distribution coefficient. Primes denote lower or extract section of column
 L = heavy or extract phase rate, moles, weight, or volume per unit time
 m = subscript referring to any stage in lower or extract end of column (stages 1 to M)
 M = number of theoretical stages below the feed stage, $M + 1$
 n = subscript referring to any stage in upper or raffinate end of column (stages $M + 2$ to N)
 N = total number of theoretical stages including partial reboiler if any but excluding partial condenser if any
 q_s = fraction of a given component which enters in the fresh solvent stream S

- $q_{s'}$ = fraction of a given component which enters in the lower feed F'
 q_F = $1 - q_s - q_{s'}$ = fraction of a given component which enters in the upper feed F
 R = external reflux ratio at top or raffinate end of column, amount of reflux/amount of product or $(L_{N+1} - S)/D$
 R' = external reflux ratio at bottom or extract end of column, amount of reflux/amount of product or $(V_0 - F')/B$
 S = fresh solvent rate, moles, weight, or volume per unit time, also S is used for stripping factor when $S_n = S_m$
 S_E = solvent rich material recovered in the extract solvent recovery equipment, moles, weight, or volume per unit time
 S_n = KV/L = component stripping factor in upper or raffinate end of column, with K, V , and L representative averages for the section
 S_m = $K'V'/L'$ = component stripping factor in lower or extract end of column, with K', V' , and L' representative averages for the section
 V = light or raffinate phase rate, moles, weight, or volume per unit time
 x = concentration in heavy or extract phase, units consistent with units on rates
 y = concentration in light or raffinate phase, units consistent with units on rates

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