Title
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Publication Date
1953-08-10
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BERKELEY, CALIFORNIA
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Dynamics of Stagewise Extraction Columns

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August 10, 1953

(Excerpt from UCRL-400, July 29, 1949)

Berkeley, California
ABSTRACT

A calculation method has been developed in order to correlate continuous-flow extraction results in mixer-settlers with rate data for batch experiments. This correlation utilizes dynamic extraction coefficients (E's) that are calculated from the rate data and the equilibrium extraction coefficients. The dynamic coefficients may then be used in stage-by-stage calculations, or their average values used in equations derived herein from the overall performance of center-fed columns.
The general extraction problem involved here may be defined as follows: Aqueous feed solution is introduced into the center of the column A. (Figure 2) The feed solution mixes with the wash solution and then passes to the left, coming into contact with an organic extract solution to which most of the solute to be extracted is transferred along with smaller quantities of impurity with a smaller equilibrium extraction coefficient. This organic phase then passes to the right of the feed plate, where the conditions are adjusted to give smaller equilibrium extraction coefficients. In this section the impurity is re-extracted into the aqueous phase, along with some of the desired solute. This solute then refluxes, or passes down the column to the extracting section, where it is again extracted into the organic phase. As the solute cannot leave the column in the aqueous stream, due to the high extraction coefficient maintained in the extracting section, it eventually finds its way out in the organic phase. The column shows continually a large accumulation of solute that is undergoing reflux. The organic phase leaving column A may serve as the feed for column B.

The calculation method involves a sharp distinction between equilibrium extraction coefficients and dynamic extraction coefficients. The equilibrium extraction coefficient, given the symbol $E_v$, is equal to the ratio of the concentration of the element under consideration in the organic phase to its concentration in the aqueous phase at equilibrium; while the dynamic extraction coefficient, given the symbol $E_d$, is the ratio of the concentration of the element in the
FIG. 2
NOMENCLATURE FOR CONTINUOUS FLOW COLUMNS

COLUMN A (AQUEOUS-PHASE FEED)

COLUMN B (ORGANIC-PHASE FEED)

MU-6006
organic phase to its concentration in the aqueous phase under any actual condition, i.e., it is a quantity which may change with location or time.

The extractability $\beta$ may be defined as the ratio of the $E$'s for any two elements. It is therefore similar to the $\alpha$-values used for distillation processes, except that the latter refer generally to equilibrium distributions.

A major development during the course of the present research program has been the derivation of algebraic relations to be used in calculating the dynamic extraction coefficients from rate data. These results predict steady-state column values of $E$ which may be surprisingly different from the values of $E$ determined for the same concentrations.

**Derivation of Dynamic Extraction Coefficients**

It is apparent that in the column at steady-state conditions the $E$ of any one stage does not change with time, even though solutions of concentration different from those in the stage are constantly being fed into the state, and that interphase transfer of solute is constantly taking place. The only way that $E$ can remain constant is for the rate of change of $E$ due to transfer between the phases plus the rate of change in $E$ due to flow rate alone be equal to zero, each rate of change of $E$ being taken with respect to time; in other words, that

$$\left(\frac{\partial E}{\partial t}\right)_{\text{flow rate}} = 0 + \left(\frac{\partial E}{\partial t}\right)_{\text{mass transfer}} = 0 = 0$$  \hspace{1cm} (7)

Since $E = Y/X$, where $X =$ moles of solute per liter of aqueous phase and $Y =$ moles of solute per liter of organic phase,

$$\left(\frac{\partial E}{\partial t}\right)_{f_{o}r_{o}=0} = \frac{1}{X} \left(\frac{\partial Y}{\partial t}\right)_{f_{o}r_{o}=0} - \frac{E}{X} \left(\frac{\partial X}{\partial t}\right)_{f_{o}r_{o}=0}$$ \hspace{1cm} (9)
and since

\[ X + \rho Y = \text{const.} \]  \hspace{1cm} (10)

where \( \rho \) = the volume of organic phase divided by the volume of aqueous phase.

\[
\left( \frac{\partial X}{\partial t} \right)_{r_*=0} + \rho \left( \frac{\partial Y}{\partial t} \right)_{r_*=0} = 0
\]  \hspace{1cm} (11)

therefore

\[
\left( \frac{\partial E}{\partial t} \right)_{r_*=0} = \frac{1 + \rho E}{X} \left( \frac{\partial Y}{\partial t} \right)_{r_*=0}
\]  \hspace{1cm} (12)

We next consider A ml/min of an aqueous solution entering and leaving a mixer containing \( V_a \) ml of aqueous phase, and at the same time B ml/min of an organic solution entering and leaving the same mixer containing \( V_b \) ml of the organic phase. Let this be the \( k \)th mixer with \( X_k \) moles/liter of solute in the aqueous phase of the mixer; if this phase is homogenous, substantially perfect mixing is obtained, and the average composition of this entire phase will be the same as that of the portion leaving the mixer. Let the entering aqueous phase containing \( X_{k+1} \) moles/liter of solute and the entering organic phase contain \( Y_{k-1} \) moles/liter of solute; while the organic phase in the stage and in the exiting organic stream contain \( Y_k \) moles/liter of solute. This mixer will be part of a "column" like those shown in Figure 2.

Under steady-state conditions the amount of solute in the stage does not change, therefore the amount entering the stage must equal the amount leaving the stage. That is
\[ AX_{k+1} + BY_{k-1} = AX_k + BY_k \]  
(13)

or

\[ A(X_{k+1} - X_k) = B(Y_k - Y_{k-1}) \]  
(14)

Since

\[ E_k = \frac{Y_k}{X_k} \]  
(8)

\[
\left( \frac{\partial E_k}{\partial t} \right)_{m.s.t.s=0} = \frac{1}{X_k} \left( \frac{\partial Y_k}{\partial t} \right)_{m.s.t.s=0} - \frac{E_k}{X_k} \left( \frac{\partial X_k}{\partial t} \right)_{m.s.t.s=0}
\]  
(15)

But

\[
\left( \frac{\partial Y_k}{\partial t} \right)_{m.s.t.s=0} = \frac{B}{V_b} \left[ Y_{k-1} - Y_k \right]
\]  
(16)

and

\[
\left( \frac{\partial X_k}{\partial t} \right)_{m.s.t.s=0} = \frac{A}{V_a} \left[ X_{k+1} - X_k \right]
\]  
(17)

\[
= \frac{B}{V_a} \left[ Y_k - Y_{k-1} \right]
\]

\[
= - \frac{\rho B}{V_b} \left[ Y_{k-1} - Y_k \right]
\]  
(18)

Therefore, substituting equation (16) and equation (18) in equation (15) given

\[
\left( \frac{\partial E_k}{\partial t} \right)_{m.s.t.s=0} = \frac{B}{V_b} (1 + \rho E_k) \frac{Y_{k-1} - Y_k}{X_k}
\]  
(19)

\[
= \frac{B}{V_b} (1 + \rho E_k) (C_k - E_k)
\]  
(20)

where

\[ C_k = \frac{Y_{k-1}}{X_k} \]  
(21)

that is, \( C_k \) is the ratio of concentrations in the two phases passing the boundary between stage \( k-1 \) and stage \( k \).
Substituting equation (12) and equation (20) in equation (7) gives

\[ \frac{1 + \rho E_k}{X_k} \left( \frac{\partial Y_k}{\partial t} \right)_{f^*r^*=0} + \frac{B_k}{V_b} \left( 1 + \rho E_k \right) (C_k - E_k) = 0 \]  

or

\[ \frac{1}{X_k} \left( \frac{\partial Y_k}{\partial t} \right)_{f^*r^*=0} = \frac{B_k}{V_b} (E_k - C_k) \]  

This expression is applicable for distribution of any component between immiscible solvents in any one extracting stage, and it is not necessary that the exact mechanism for extraction be known. In order to apply it, it is only necessary that a single mixer, similar to the one to be used in the continuous apparatus, be built, and batch extractions performed in order to follow the transfer between the two phases of the element under consideration as a function of time and of total concentration level.

Equation (19) may also be written in the form

\[ \left( \frac{\partial E_k}{\partial t} \right)_{m^*t^*=0} = \frac{A}{V_b} \left( 1 + \rho E_k \right) \left( \frac{X_{k+1}}{X_k} \right) - 1 \]  

but

\[ \frac{X_{k+1}}{X_k} = \frac{E_k}{C_{k+1}} \]  

therefore

\[ \left( \frac{\partial E_k}{\partial t} \right)_{m^*t^*=0} = \frac{A}{V_b C_{k+1}} \left( 1 + \rho E_k \right) (E_k - C_{k+1}) \]  

and

\[ \frac{1}{X_k} \left( \frac{\partial Y_k}{\partial t} \right)_{f^*r^*=0} = \frac{A}{V_b C_{k+1}} (E_k - C_{k+1}) \]  

The essential steps of stage-by-stage calculation will now be discussed in
more detail. The variables to be defined are indicated by reference to Figure 2. Ordinarily \( A_p \) and \( X_p \), the flow rate and composition of the feed stream, are predetermined. Trial values for the number of stages \((n)\), the location of the feed stage \((f)\), the flow rates for extract and wash phases, and, where possible, also the extraction coefficients for the two sections of the column, must be selected arbitrarily. (Certain principles governing the selection of these variables will become evident during the ensuing discussion.)

An exact calculation may be made by a trial and error procedure. Provided \( Y_0 = 0 \), \( C_1 = 0 \), and, provided \( X_{n+1} = 0 \), we obtain \( C_{n+1} = \infty \). A value of \( Y_n \) is assumed which represents the desired extent of recovery of the particular element in the extract. \( \left( \frac{\partial Y_n}{\partial t} \right)_{f,r=0} \) is next read from the experimental plots, and is substituted into equation (26) in order to solve for \( E_n \) and hence for \( X_n \). A simple material balance by equation (13) leads to \( Y_{n-1} \) and hence to \( C_n \) which is used similarly for calculating the next stage. If the net loss of this component from the end of the extracting section does not agree with the recovery assumed in the extract phase leaving the washing section, a new recovery must be assumed and the calculation repeated. Alternatively, such a calculation could be started at the extraction end of the column, using equation (23).

Such stage-by-stage calculations may be simplified by means of a graphical solution for equations (23) or (26). The \( Y \)-\( t \) data are converted to a plot of \( \frac{1}{X} \left( \frac{\partial Y}{\partial t} \right)_{f,r=0} \) versus \( E \) (or to a family of plots, if \( \partial Y/\partial t \) varies with total concentration level). This plot is superimposed upon a plot of \( Z \) \((C-E)\) versus \( E \) for various values of \( Z \) and \( C \), where \( Z = B/V \) if equation (23) is in use, and \( Z = A/VC \) if equation (26) is applied. A plot of \( Z \) \((C-E)\) is shown for an extracting section in Figure 3, and for a washing section in Figure 4, on which specimen rate curves have been drawn. The curve corresponding to the specified \( Z \) and \( C \) is located, and its intersection with the rate curve is used to determine \( E \) for this stage. A material balance is then applied, as before, to calculate \( C \) for the next stage.

Check calculations are obtained as the value of \( C \) does not change appreciably
FIG. 3
GRAPHICAL SOLUTION FOR EXTRACTING SECTION OF COLUMN A
FIG. 4
GRAPHICAL SOLUTION FOR WASHING
SECTION OF COLUMN A
except for the outermost stages. We have, by the definition of $E$,

$$Y_{k-1} = X_{k-1} E_{k-1}$$  \hspace{1cm} (8)

In the extracting section of column $A$, by material balance, $X_{k\phi e} = R Y_{k-1} e + x_l = R X_{k-1} e E_{k-1} e + x_l$, where the dynamic volumetric ratio $R = B/k_e$ in any section of interest. Then

$$C_{k\phi e} = \frac{Y_{k-1} e}{X_{k\phi e}} = \frac{X_{k-1} e E_{k-1} e}{R Y_{k-1} e E_{k-1} e + x_l}$$  \hspace{1cm} (27)

As $x_l$ normally is exceedingly small, and $X_{k\phi e}$ increases rapidly as one progresses up the column, we obtain an approximate relation that is valid throughout the extraction section except for the outermost stages:

$$C_{k\phi e} = \frac{1}{R_{k\phi e}} = \frac{A_e}{B_e}$$  \hspace{1cm} (28)

The washing section of column $A$ displays a similar material balance:

$$B_w Y_{k-1} w = A_w X_{k\phi w} = A_f X_f = A_e x_1$$  \hspace{1cm} (29)

Therefore

$$X_{k\phi w} = \frac{B_w X_{k-1} w E_{k-1} w + A_e x_1 - A_f X_f}{A_w}$$  \hspace{1cm} (30)

and

$$C_{k\phi w} = \frac{Y_{k-1} w}{X_{k\phi w}} = \frac{A_w Y_{k-1} w E_{k-1} w}{B_w X_{k-1} w E_{k-1} w + A_e x_1 - A_f X_f}$$  \hspace{1cm} (31)

If, in the vicinity of the feed plate, the reflux rate for the element of interest is large compared to the feed rate for that element, that is, if

$$B_w X_f E_f > A_f X_f$$  \hspace{1cm} (32)

and given that

$$A_f X_f \gg A_e x_1$$  \hspace{1cm} (33)

we obtain:

$$C_{k\phi w} \gg \frac{1}{R_w} = \frac{A_w}{B_w}$$  \hspace{1cm} (34)
RAPID CALCULATION OF CENTER-FED COLUMNS

The advantages of center-feeding a column to be used in the separation of two components often have been overlooked. In general it has found widespread use only in continuous fractional distillation; however, Varteressian and Fenske\textsuperscript{1,2} have shown in general how this method may be applied to systems other than distillation.

In any center-feeding column, a reflux stream is fed counter-current to the exit product stream. This imposes the requirement that the exit product stream be in equilibrium with a phase which has a higher concentration of the desired component than the feed. At the same time the reflux stream has the effect of scrubbing out unwanted components which may have been extracted in the extracting section. The overall result of reflux therefore is to achieve an extent of separation which would not otherwise be possible.

By extending the calculation methods of Kremser\textsuperscript{3} for absorption and Underwood\textsuperscript{4} for extraction and by joining two separate solvent extraction columns into a single column, a calculation method for a center-fed counter-current extraction column falls naturally into place. It is necessary only to assume that the ER in any one section of the column remains constant. Equations similar to those derived below, but using equilibrium coefficients instead of dynamic coefficients, have been given by Miles.\textsuperscript{5} Such an assumption will usually predict the required number of stages with less than 10 percent error, as compared to the number given in stage-by-stage calculations.

Any number of side streams of either phase may be fed into or taken from the column, although in the following equations only one
side stream has been introduced into the column and none taken from it.

Let the product fed to column A (Figure 2) be equal to unity 
\(A_F X_F = 1\), while neither of the other entering streams contains any 
of the desired product \((B_0 Y_0 = 0\) and \(A_W X_{n+1} = 0\) then

\[
A_X + B_Y = A_F X_F = 1
\]  \(\text{(a)}\)

Starting with the first stage of either column A or column B, a stage-
by-stage material balance gives

\[
\left\{ \left[ \left( \frac{(A_X E_1 + A_X X_1) E_2 R_2 + A_X X_1}{E_2 R_2^{...}} \right) E_1 R_1 \right] - B_n Y_n \right\} E_{f+1} R_{f+1} - B_n Y_n = 0
\]  \(\text{(b)}\)

where \(R = B/A\), the ratio of volumetric flows.

In column A, in which the center feed is an aqueous phase, let

\[
E_1 = E_2 = \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots = E_f = E_e,
\]

\[
R_1 = R_2 = \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots = R_f = R_e,
\]

\[
E_{f+1} = E_{f+2} = \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots = E_n = E_w,
\]

\[
R_{f+1} = R_{f+2} = \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots = R_n = R_w,
\]

\(A_1 = A_e\), and \(B_n = B_w\).

Substitution of equation \((c)\) in \((b)\) and removal of the brackets gives

\[
A_e X_1 \sum_{k=1}^{f} (E_e R_e)^k = B Y_n \sum_{k=0}^{n-f} (E_w R_w)^{-k}
\]  \(\text{(d)}\)

where \(f\) is the number of stages in the extracting section (including
the feed plate), and \((n-f)\) is the number of stages in the washing
section. Therefore the fraction \(r\) recovered in each exit stream is
given by
In column B, in which the center feed is an organic phase, let

\[ E_1 = E_2 = \cdots = E_{f-1} = E_{wl} \]
\[ R_1 = R_2 = \cdots = R_{f-1} = R_{w3} \]
\[ E_f = E_{f+1} = \cdots = E_n = E_e^p \]
\[ R_f = R_{f+1} = \cdots = R_n = R_e^p \]
\[ A_w X_1 + B_e Y_n = B_F Y_F = 1_p \]
\[ A_1 = A_{wp} \text{ and } B_n = B_{e^p} \]

Substitution of equation (e) in equation (b) and removal of the brackets gives

\[ A X_1 \sum_{k=0}^{f-1} (E_w R_w)^k = B_e Y_n \sum_{k=1}^{n-f+1} (E_e R_e)^{-k} \quad (f) \]

Therefore

\[ A X_1 = r_{B^p a} = \sum_{k=0}^{n-f+1} (E_e R_e)^{-k} \sum_{k=0}^{f-1} (E_w R_w)^k + \sum_{k=0}^{n-f+1} (E_e R_e)^{-k} \quad (54) \]
and
\[ E_{e_n} = \frac{\sum_{k=0}^{f-1} (E_{w} R_{W})^k}{\sum_{k=0}^{n-f+1} (E_{w} R_{W})^k + \sum_{k=1}^{f} (E_{w} R_{W})^{-k}} \]  \(55\)

In the simultaneous application of equations (52) - (55) to two or more components, the expressions may be simplified if one of the exit streams contains less than one percent of the total amount of one element and more than 99 percent of another element, both of which are fed to the column at the same point. To show this, consider a solute S and an impurity Q which has a smaller extraction coefficient. These are fed in the aqueous stream to column A, and the amount of each in the leaving organic stream is examined. It is seen that for greater than 99 percent recovery of S with less than 1 percent Q, \(r_{b}^{S}\) must be approximately unity while \(r_{b}^{Q}\) must be very small. This means that
\[ \sum_{k=1}^{f} (E_{e} R_{e})^k \gg \sum_{k=0}^{n-f} (E_{w} R_{W})^{-k} \]  \(56\)

and
\[ \sum_{k=1}^{f} (E_{e} R_{e})^k \ll \sum_{k=0}^{n-f} (E_{w} R_{W})^{-k} \]  \(57\)

Therefore dividing both numerator and denominator of equation (53) by \(\sum_{k=1}^{f} (E_{e} R_{e})^k\) gives
\[ r_{b}^{S} = \frac{1}{1 + \frac{n-f}{f} \left( \frac{S}{S + R_{W}} \right) \frac{E_{e} R_{e}}{E_{w} R_{W}}} \]  \(58\)

\[ = 1 - \frac{(E_{w} R_{W})^{n-f+1} - 1}{(E_{w} R_{W})^{n-f} (E_{w} R_{W}) - 1} \cdot \frac{(E_{w} R_{W})^{-1}}{(E_{e} R_{e}) (E_{e} R_{e})^{-1}} \]  \(59\)
Neglecting \( \sum_{k=1}^{f} \left( E_e^Q R_e^k \right) \) in equation (53) gives

\[
\begin{align*}
\sum_{k=0}^{n-f} \left( E_w^Q R_w^{-k} \right) \\
\sum_{k=0}^{n-f} \left( E_w^Q R_w^{-k} \right) \\
= \left( E_e^Q R_e \right)^{f-1} \left( E_w^Q R_w \right)^{n-f-1} \left( E_e^Q R_e \right) \left( E_w^Q R_w \right) - 1
\end{align*}
\]

It is desirable to make \( r_b^S \) as close to unity as possible and \( r_b^Q \) as small as possible for a given number of stages in the extracting and washing sections. It can be seen that \( r_b^S \) becomes increasingly closer to unity as \( E_e^S R_e \) and \( E_w^S R_w \) become larger and larger. But that although its difference from unity becomes small very rapidly as \( E_e^S R_e \) becomes increasingly larger than unity, the difference changes little as \( E_w^S R_w \) becomes greater than unity, the factor containing \( E_w^S R_w \) changing only from \( n+1 \) to unity as \( E_w^S R_w \) changes from one to infinity. On the other hand, \( r_b^Q \) becomes smaller and smaller as \( E_w^Q R_w \) and \( E_e^Q R_e \) become smaller and smaller, growing small very rapidly as \( E_e^Q R_e \) becomes less than unity, while changing relatively slowly as \( E_w^Q R_w \) becomes smaller than unity. Therefore, it becomes important that \( E_e^Q R_e \) be as large as possible and that \( E_w^Q R_w \) be as small as possible, while \( E_w^S R_w \) is unity or slightly greater than unity and that \( E_e^Q R_e \) is unity or slightly less than unity.

Since

\[
E_w^Q = \beta_w^{Q/S} E_w^S
\]
\[ E^Q_e = \beta^{Q/S}_e E^S_e \]

and since \( \beta^{Q/S}_e \) and \( \beta^{Q/S}_a \) are much less than unity, it is possible to make \( E^S_e \) large and at the same time have \( E^Q_e \) one or smaller since

\[ E^Q_e = \beta^{Q/S}_e E^S_e R_e. \]

Similarly, \( E^Q_w \) may be made small while \( E^S_w \) is one or slightly larger since

\[ E^Q_w = \beta^{Q/S}_w E^S_w R_w. \]

Thus, due to the strong dependence of \( r_b^S \) on \( E^S_e R_e \) and of \( r_b^Q \) on \( E^Q_w R_w \), it is found empirically that for the total number of stages to be a minimum for a given recovery and separation that

\[ E^S_w R_w = 1 \]

and that

\[ E^Q_e R_e = 1 \]

If these values are substituted in equations (58) and (60) then equation (58) becomes

\[ r_b^S = 1 - (n-f+1) \cdot \frac{(E^S_e R_e) - 1}{(E^S_e R_e) \left[(E^S_e R_e)^{f-1} - 1\right]} \]

and equation (60) becomes

\[ r_b^Q = f (E^Q_w R_w)^{n-f} \cdot \frac{(E^Q_w R_w) - 1}{(E^Q_w R_w)^{n-f+1} - 1} \]

\[ \approx f (E^Q_w R_w)^{n-f} \left[1 - (E^Q_w R_w)^{n-f+1}\right] \]
By examining equations (62) and (63) and considering the great differences in the values of $E_{e} R_{e}$ and $E_{w} R_{w}$, one can see how very powerful the method is in the separation of two elements.

However, in actual separations it may not always be possible to make $E_{e} R_{e}$ and $E_{w} R_{w}$ as widely different as one would like, because of interfering mechanical or chemical difficulties. Even though making $E_{w}^{s} R_{w}$ and $E_{e}^{Q} R_{e}$ both equal to unity would theoretically make the number of stages a minimum, this might make $E_{e}^{s} R_{e}$ so large and $E_{w}^{Q} R_{w}$ so small that entrainment would limit the extraction per stage. Therefore, care must be exercised in determining the various extraction coefficients to be used, keeping in mind the desired recoveries and decontaminations.

The work described in this paper was performed under the auspices of the Atomic Energy Commission.
NOMENCLATURE

A = flow rate of aqueous phase, ml/min.
B = flow rate of organic phase, ml/min.
C = ratio of concentration of product in the incoming organic phase to its concentration in the leaving aqueous phase, \( Y_{n-1}/X_n \).
E = dynamic extraction coefficient, \( Y/X \).
R = ratio of organic flow rate to aqueous flow rate, \( B/A \).
V = volume, ml.
X = concentration of product in the aqueous phase, moles/liter.
Y = concentration of product in the organic phase, moles/liter.
Z = symbol representing \( B/V_b \) or \( A/V_a \), min \(^{-1} \)

Greek Symbols

\( \beta \) = relative extractability, ratio of any two E's, \( E/E \).
\( \mathcal{E} \) = equilibrium extraction coefficient, \( (Y/X) \) at equilibrium.
\( \rho \) = ratio of organic volume to aqueous volume, \( V_b/V_a \).

Subscripts

1, 2, etc. refer to the stage numbers, starting with the left-hand or lower end.

A refers to the unit whose feed stream is aqueous.
a refers to the aqueous phase.
B refers to the unit whose feed stream is organic.
b refers to the organic phase.
e refers to the extracting section of the unit.
F refers to the feed stream.
f refers to the feed stage.
k or m refers to any general stage.
n refers to the last stage (the right-hand or upper end).
w refers to the washing section of the unit.
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