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Cascade Design for Uranium Enrichment Employing Chemical Exchange and Solvent Extraction

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ABSTRACT

This paper describes the optimization of a solvent extraction plant used for the enrichment of uranium by redox chemistry and chemical exchange. The basic unit of the plant is a standard mixer-settler where the organic and aqueous phases are in chemical equilibrium. Repeated contact of the phases transfers small amounts of ^{235}U into the organic phase and equally small amounts of ^{238}U into the aqueous phase. The mixer-settler units, all of the same size, are grouped into a countercurrent train. The plant or cascade as a whole consists of a number of trains. Each of these consists of mixer-settlers of different sizes, and the number of stages in the trains varies. The optimization procedure is a four-stage process designed to minimize the whole plant size.

INTRODUCTION

The following study is part of a recent investigation into the use of solvent extraction coupled with chemical exchange to enrich uranium for use in conventional nuclear reactors that generate electricity. It is a possible alternative to the classical processes used for uranium enrichment using gaseous processes involving UF_6 (1). This consideration of an alternative processes involving solvent extraction follows earlier work by French researchers (2). The process under consideration has not been brought to commercial acceptance. However, from the standpoint of separation processes it is an interesting one, and this paper will describe the design and optimization of a multistage plant in the form of a cascade for

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this enrichment. A simple model of this process has been published recently (3).

Figure 1 gives a basic outline of the type of plant being considered. The enrichment process is carried out by contacting an organic solution containing UO_2^{2+} as a chloride anionic complex with an aqueous phase which contains both UO_2^{2+} and U^{4+} in significant amounts dissolved in hydrochloric acid. These two phases are thermodynamically in equilibrium, but in the contact process a small amount of U-235 will pass into the organic phase with an equally small flux of U-238 into the aqueous stream. The reason this occurs is that the stability constant for the UO_2^{2+} chloride anionic complex of the U-235 isotope is slightly larger (4). The chemical requirements of the process demand that the compositions of the aqueous and organic streams of all the trains in the plant remain fixed. To accomplish the contacting process, the two uranium valence states are treated in a membrane cell to change their valence states for the reflux operations. Figure 1 shows that this can be carried out in a membrane cell so that the process theoretically needs electricity only.

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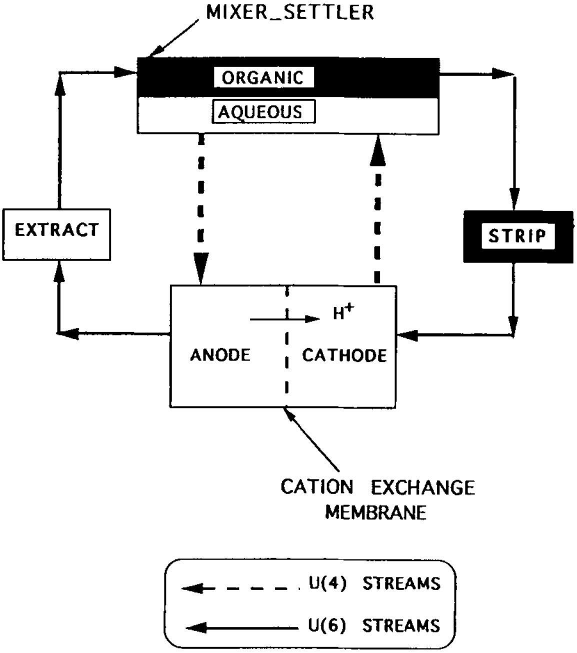

FIG. 1 The solvent extraction plant.

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Most of the previous work in this area has been carried out in ion-exchange columns and has been considered theoretically (5). In this particular method a three-stage process is involved. First, the column is filled with a solution containing an oxidizing agent. Then the uranium is passed through, and multiple contact of the aqueous and organic phases causes isotope separation to take place. Finally, a solution containing a reductant is added (6) to elute the uranium band. When the uranium comes off, the first half of the eluted band consists of depleted uranium. This is a comparatively costly process since exotic reductants such as titanous chloride ($TiCl_3$) are used. Nevertheless, the process is reported to have been brought to pilot-plant status in Japan (7).

CHEMICAL CONSIDERATIONS

In this study preliminary work was carried out to specify the chemical conditions for equilibrium (8). The organic phase consisted of a 30% by volume solution of trioctylamine (TOA) in a benzene diluent and the aqueous phase was 4 M HCl (9). The experiments showed that U^{4+} was virtually insoluble in the organic phase under these conditions. However, the hexavalent UO_2^{2+} was readily extracted into the organic phase as it formed an anionic complex with hydrochloric acid whereas U^{4+} did not associate with chloride radicals in the same way. The equilibrium isotherm could be correlated by a "Langmuir" type isotherm as follows,

$$U = \frac{C_1 u}{1 + C_2 u} \tag{1}$$

In this expression U denotes the concentration of UO_2^{2+} in the organic phase and u the concentration in the aqueous phase, both expressed in grams of uranium per liter. The measured values of the constants C_1 and C_2 were 16.91 and 0.161, respectively, in 4 M HCl. As the plants would be operated under a large reflux ratio, the total amounts of uranium, expressed in moles, in the organic and the aqueous streams would be equal.

The organic phase consists entirely of UO_2^{2+} with a concentration of U g/L and the aqueous phase has a mixed composition of u g/L of UO_2^{2+} and z g/L of U^{4+}. If Φ is the ratio of the volume of aqueous phase to organic contacted, then, since the uranium inventories are equal,

$$U = \Phi(u + z) \tag{2}$$

Substitution for U in the above equation and rearrangement gives

$$z = \frac{1}{\Phi}\left\{\frac{C_1 u}{1 + C_2 u} - \Phi u\right\} \tag{3}$$

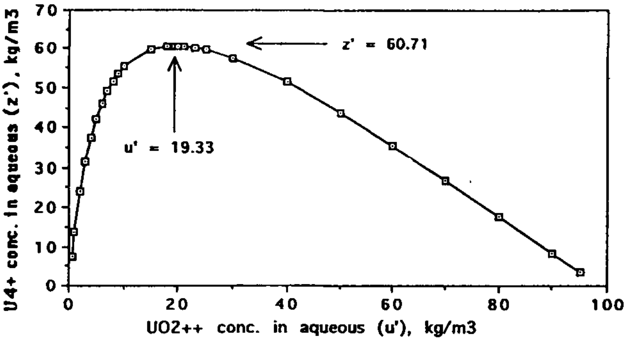

FIG. 2 Maximizing the U(IV) concentration.

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The variation of z as a function of u is shown in Fig. 2 for the case where $\Phi = 1$, and it can be seen that there is a maximum for the value for z whose value can be determined by differentiation of the last expression and setting dz/dx equal to zero. In this particular case the operating values are $u = 19.33$, $z = 60.71$, and $U = 79.49$. The fraction of UO^{2+} in the aqueous phase (θ) is 0.243.

One of the ways of optimizing the conditions in the contact train is to maximize z, the concentration of U^{4+} in the aqueous phase due to the mixed nature of this particular stream. Differentiation of Eq. (3) gives

$$\frac{dz}{du} = \frac{1}{\Phi}\left\{\frac{C_1}{(1 + C_2 u)^2} - \Phi\right\} = 0$$

The solution of this equation is

$$u = \frac{1}{C_2}\left\{\sqrt{\frac{C_1}{\Phi}} - 1\right\} \tag{4}$$

Once Φ is defined, the optimal value of u is found using Eq. (4). U can then be calculated using Eq. (1), and z finally estimated by means of Eq. (2). It should be stressed that the compositions of the various redox components of uranium and the value of Φ have to remain constant throughout the whole cascade to retain the chemical equilibrium.

SINGLE TRAIN CALCULATIONS

The calculation of the plant configuration is based on consideration of a single train. The mass balance basis for these calculations is based on

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the production of 1 m^3 of enriched organic solution with an isotopic composition of Y mol fraction U-235 and a total uranium concentration U g uranium per liter in the organic. The waste produced is T m^3 at an isotopic content of W mol fraction U-235. The total cascade consists of a number of mixer-settler trains connected in a countercurrent mode. Each train consists of N mixer-settlers of the same size. Such a train is illustrated in Fig. 3, and stage 1 with the lowest enrichment is on the left-hand side and stage N on the right. The isotopic concentration of the aqueous stream leaving the train (x_1) is specified in the cascade design. The uranium solutions are refluxed round the train, and the quantities of solution treated are much larger than the quantities of feed, product, or tailings. R m^3 of solution has to be treated to produce 1 m^3 of product.

In particular, Fig. 3 represents a train at a point above the feed and shows details of the organic and aqueous streams passing between stage n and stage $n + 1$. The mass balance is

$$RUy_n - Ru\Phi y_{n+1} - Rz\Phi x_{n+1} = YU \tag{5}$$

In Eq. (5), y_n is the isotopic concentration of UO_2^{2+} in stage n, x_n is the value for U^{4+}, and θ is the mole fraction of UO_2^{2+} in the aqueous phase. The first term in the expression is the mass flux for the organic phase, and the next two give the values for the aqueous phase. However, from Eq. (2), $u + z = U/\Phi$, and from the definition of θ, $\theta(u + z) = u$. This means that the values of u and z are given by

$$u = \frac{\theta U}{\Phi} \quad \text{and} \quad z = \frac{(1 - \theta)U}{\Phi} \tag{6}$$

Substituting the values from Eq. (6) into Eq. (5) gives

$$RUy_n - RU\theta y_{n+1} - RU(1 - \theta)x_{n+1} = YU \tag{7}$$

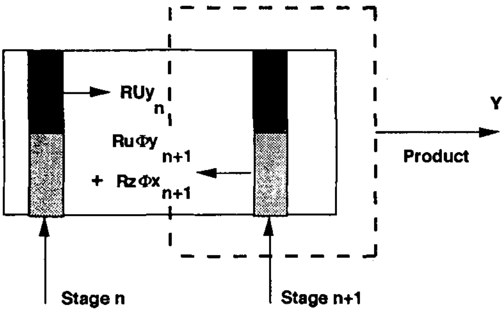

FIG. 3 Mixer-settler mass balance.

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since the equilibrium relationship between y_n and x_n is

$$y_n = (1 + \epsilon)x_n \tag{8}$$

Here ϵ is the isotope enrichment constant which is very small compared with unity and has the reported value of 0.0013. This last expression is only valid when x_n is small, say less than 0.1. Substitution of Eq. (8) into Eq. (7) and rearranging gives the following relationship between x_n and x_{n+1}:

$$Y/R = (1 + \epsilon)x_n - (1 + \theta\epsilon)x_{n+1}$$

or

$$\frac{Y}{R(1 + \theta\epsilon)} = rx_n - x_{n+1} \tag{9}$$

where

$$r = \frac{1 + \epsilon}{1 + \theta\epsilon} \approx 1 + (1 - \theta)\epsilon \tag{10}$$

since ϵ is small. In addition, $(1 + \theta\epsilon)$ is also close to unity, so this term can be omitted from Eq. (9). Applying this equation to stages n and $n - 1$ in the train,

$$Y/R = rx_{n-1} - x_n \tag{11}$$

Finally, by combining Eqs. (9) and (11):

$$x_{n+1} - (1 + r)x_n + rx_{n-1} = 0 \tag{12}$$

This is a finite difference equation whose general solution is

$$x_n = A + Br^n \tag{13}$$

A and B are constants that have to be evaluated. In particular, the last expression gives

$$x_1 = A + Br \qquad \text{and} \qquad x_2 = A + Br^2$$

Applying Eq. (9) to x_1 and x_2,

$$Y/R = rx_1 - x_2 = A(r - 1) = (1 - \theta)\epsilon A$$

This gives the important result that the value of A is given by

$$A = \frac{Y}{R\epsilon(1 - \theta)} \tag{14}$$

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Since x_1 is fixed, Eq. (13) can be used to obtain a value for B such that

$$B = \frac{x_1 - A}{r} \tag{15}$$

Having calculated the values of A and B, the composition of the organic stream leaving the train (y_N) is given by Eqs. (13) and (8) as $y_N = A + (x_1 - A)r^N$, and rearranging this will give

$$\frac{y_N - A}{x_1 - A} = r^N \quad \text{or} \quad N = \frac{\ln\left\{\dfrac{y_N - A}{x_1 - A}\right\}}{\ln(r)}$$

However, as r is close to unity, $\ln(r) \approx (1 - \theta)\epsilon$. This finally gives the expression for the number of stages in the train (N) as

$$N = \ln\left\{\frac{y_N - A}{x_1 - A}\right\}\Big/(1 - \theta)\epsilon \tag{16}$$

From Eq. (16) it can be seen that the value of N is independent of Φ. The specification of the train now has to be optimized using the concept of separative work. This requires the total amount of solution treated by the train as SW, given by the product of N and R. From Eq. (14), R is given by

$$R = \frac{Y}{A(1 - \theta)\epsilon}$$

so that

$$SW = \frac{Y}{(1 - \theta)^2\epsilon^2 A} \ln\left\{\frac{y_N - A}{x_1 - A}\right\} \tag{17}$$

From Eq. (17) it can be seen that SW is infinite when A is zero and when $A = x_1$. Between these two values there is a point at which SW is a minimum when $d(SW)/dA = 0$. Differentiation of Eq. (17) gives this optimum value which is obtained by solving

$$\frac{1}{x_1 - A} - \frac{1}{y_N - A} = \frac{1}{A} \ln\left\{\frac{y_N - A}{x_1 - A}\right\} \tag{18}$$

Finally inspection of Eq. (17) suggests that the optimum value of A increases with x_1 so that R decreases toward the product end.

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CONFIGURATION OF THE CASCADE

A number of solvent extraction trains are connected in a countercurrent mode in the plant as a whole. The way in which this is done is shown in Fig. 4. The plant contains both stripping and enrichment sections, and the tails isotopic concentration and flow rate are W and T, respectively. For any stage in the cascade the composition of the aqueous stream leaving the plant (x_1) is denoted by X_j and the value of the effluent organic (y_N) is equal to X_{j+1}. The separative work for stage j, $(SW)_j$, is given by a modified form of Eq. (18):

$$\frac{1}{X_j - A} - \frac{1}{X_{j+1} - A} = \frac{1}{A} \ln\left\{\frac{X_{j+1} - A}{X_j - A}\right\} \tag{19}$$

It can be seen that the fraction of UO_2^{2+} (θ) in the aqueous phase increases the separative work quite noticeably. The separative work, as has been shown by previous investigators, is proportional to $(1/\epsilon)^2$. As before, from Eq. (18) the optimal value of A_j is given by

$$\frac{1}{X_j - A_j} - \frac{1}{X_{j+1} - A_j} = \frac{1}{A_j} \ln\left\{\frac{X_{j+1} - A_j}{X_j - A_j}\right\} \tag{20}$$

where A is given by

$$A = \frac{Y}{(1 - \theta)\epsilon R}$$

To evaluate the total separative work of the enrichment section, the various values of X_j are selected and the sum $\sum_{j=1}^{E} SW_j$ is calculated. For the stripping section the procedure is the same. However, with reference to Fig. 4, it is clear that the values of A change and for this part of the

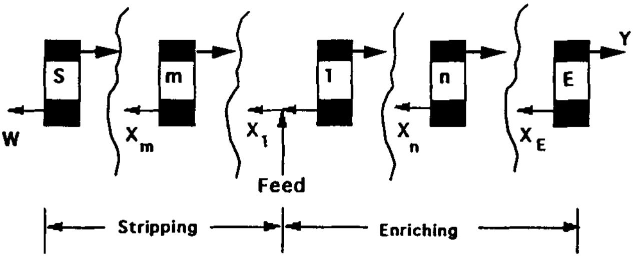

FIG. 4 The chemical exchange cascade.

plant Y changes to TW or the product of the tails flow and its isotopic composition, so

$$A = \frac{TW}{(1 - \theta)\epsilon R} \tag{21}$$

RESULTS

Determination of the optimal configuration of this kind of cascade turns out to be rather complex. It depends, to a large degree, on the number of separate mixer-settler trains that comprise the configuration of the enrichment or stripping section of the plant. The simplest example is the case where there are two trains in which the values of $X_F = X_1$, $Y = X_3$ are fixed and X_2 is allowed to vary. The results of a typical calculation are shown in Fig. 5 for the case where $X_F = 0.7\%$ and $Y = 3\%$, and this particular example is used in the rest of this paper as it represents a typical case for uranium enrichment for a PWR power reactor. In the diagram the $Y/(1 - \theta)^2\epsilon^2$ term of Eq. (17) is omitted as this calculation is only for comparative purposes. The graph shows a minimum for the separative work when X_2 is 1.32% and $SW = 455$. The other piece of information that can be gleaned from this graph is the value of SW when there is only one train in this section of the plant. In this particular case, $X_2 = Y = 3\%$, and the value of SW is 505. Therefore, adding a second train to the plant reduces the SW by around 10%.

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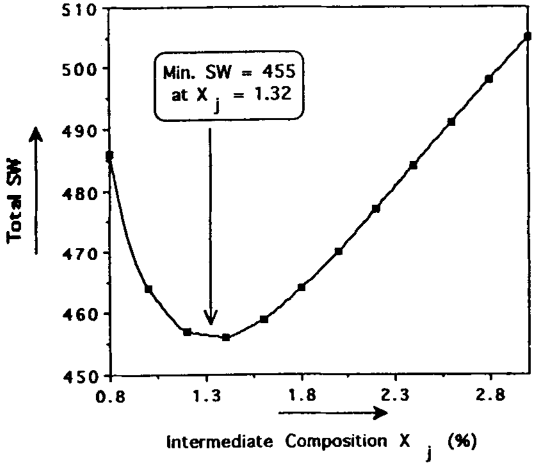

FIG. 5 Optimizing a two train cascade.

When there are more than two trains in the cascade, the calculation of the X_j values becomes more complex. The Appendix shows how this can be done. It was found that these optimization equations are badly conditioned for solution using a computer "package," so this development proved to be necessary. From the algebra presented, the results for the number of trains in the enrichment section is given in Fig. 6. Again the factor $Y/(1 - \theta)^2\epsilon^2$ has been excluded from this particular graph as this is only another comparison. This shows quite clearly that increasing the number of mixer-settler trains will reduce the separative work. However, the results only indicate a modest decrease in this important parameter as the number of trains is increased.

In addition, Fig. 7. gives more details of the cascade design sequence. In this particular figure the optimal calculations for the reflux ratio (R) and the X_j values are given. For the values of R, the factor $Y/(1 - \theta)\epsilon$ has been omitted for the purposes of comparison. The data in this figure are given for the cases where the number of mixer-settler trains is 2, 3, and 4. This figure illustrates the way in which an increase in the number of trains makes the process more efficient. It also shows how the nest of trains "meshes" together.

From the previous discussion it is clear that there should be an large number of mixer-settler trains in the plant for the separative work to be a minimum. The parameter A can thus be assumed to be a continuous function of X, and the subscripts for A can be omitted. For this case X_{j+1}

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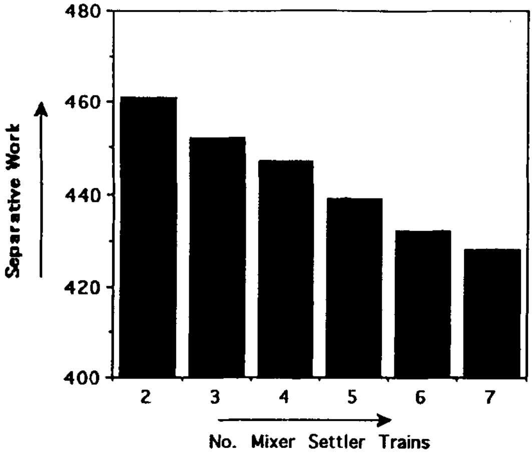

FIG. 6 The effect of the number of trains on the *SW*.

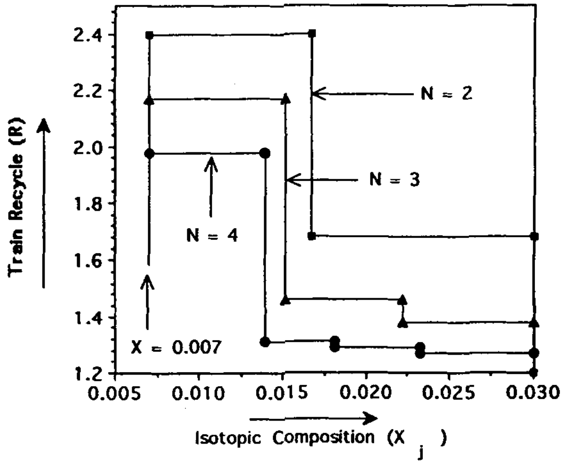

FIG. 7 Details of the "squared-off" cascades.

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$- X_j = \delta X$, where δX is a small quantity. From Eq. (19),

$$SW_j = \frac{Y}{(1-\theta)^2\epsilon^2 A} \ln\left\{\frac{\delta X + X - A}{X - A}\right\} \tag{22}$$

or

$$SW_j = \frac{Y}{(1-\theta)^2\epsilon^2 A} \ln\left\{\frac{\delta X}{X - A} + 1\right\}$$

Thus

$$SW_j \approx \frac{Y}{(1-\theta)^2\epsilon^2 A} \frac{\delta X}{X - A} \tag{23}$$

In the last expression $(1/A)(1/X - A)$ has to be minimized for the optimal result, which gives $A = X/2$. From Eq. (20) this shows that at the optimum,

$$R_{\text{OPT}} = \frac{2Y}{(1-\theta)\epsilon X} \tag{24}$$

It can be seen that R_{OPT} is inversely proportional to X. Equation (23) gives the following result for the separative work with an infinite number of stages:

$$SW_j \approx \frac{4Y}{(1-\theta)^2\epsilon^2} \frac{dX}{X^2}$$

Integration of this last expression between the limits $X = X_F$ and $X = Y$ will give the optimal minimum value of the separative work for the enrichment section (SWE_{OPT}) as

$$SWE_{\text{OPT}} \approx \frac{4Y}{(1 - \theta)^2\epsilon^2} \int_{X_F}^{Y} \frac{dX}{X^2} = \frac{4Y}{(1 - \theta)^2\epsilon^2} \left[\frac{1}{X_F} - \frac{1}{Y}\right] \tag{25}$$

Following the result given for the reflux in the tailing section of the plant, the optimum separative work of the tailings or stripping section (SWS_{OPT}) is given by

$$SWS_{\text{OPT}} = \frac{4TW}{(1 - \theta)^2\epsilon^2} \left[\frac{1}{W} - \frac{1}{X_F}\right] \tag{26}$$

where X_T is the isotopic composition of the tails from the plant. The total minimal seperative work for the plant as a whole is given by the sum of Eqs. (25) and (26).

CONCLUSIONS

This paper has shown that the optimization of a solvent extraction train for the enrichment of uranium by chemical exchange involves a number of steps and thus makes it interesting from the viewpoint of a separation process. There are four steps in this process. The first is the specification of the bulk concentrations of the uranium species UO_2^{2+} and U^{4+} in the aqueous and organic phases of the mixer-settler train. For this optimization procedure the concentration of the U^{4+} (z) in the aqueous phase has been maximized as a function of Φ which is the ratio of organic to aqueous phases treated in the plant as a whole.

The next phase of the optimization process is to ensure that the size of a mixer-settler train is as small as possible to produce the required separation. For this purpose the "size" of the train is expressed as the product of the ratio of the organic flow rate in the train and the number of stages. As an analogy to work on isotope enrichment using conventional gaseous methods for uranium enrichment, this optimization strategy is based on the principle of the minimization of the separative work (SW) of the train. In this case the separative work is the product of the parameters R and N.

As the plant consists of a number of separate trains, the next optimization exercise is the determination of the configuration of the plant. In particular, the concentration of the tails and product concentrations of the individual trains is specified as a classical "squared-off" cascade. Again the main emphasis of this process has been the minimization of the

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separative work in the section of the plant as a whole. This particular exercise shows that the larger the number of trains in the plant, the lower are the *SW* requirements. So the final process in the optimization procedure has been the calculation of the separative work for the case where the number of trains in the plant is infinite.

APPENDIX

If Eq. (20) is modified with the substitutions $\alpha = A_j/X_j$ and $r = X_{j+1}/X_j$, the following expression is obtained:

$$\frac{1}{1-\alpha} - \frac{1}{r-\alpha} = \frac{1}{\alpha}\ln\left\{\frac{r-\alpha}{1-\alpha}\right\} \tag{A1}$$

This means that the optimal value of α can be calculated for a given value of r. Several calculations can therefore be carried out with different values of r, ranging from 1 to 5, to determine the optimal value of α. The next step is to determine $1/\alpha \ln\{r - \alpha/1 - \alpha\}$ for the optimal conditions as the function β. Once these values have been obtain, β can be correlated by an empirical polynomial correlation. This will simplify the subsequent algebra. However, it may introduce some degree of error in the calculations. As β is zero when $r = 0$,

$$\beta = ar + br^2 + cr^3 + dr^4 \tag{A2}$$

and

$$d\beta/dr = \beta' = a + 2r + 3r^2 + 4dr^3 \tag{A3}$$

In Eqs. (A2) and (A3), a, b, c, and d are constants determined by the correlation process. These constants were found to be $a = 0.242$, $b = -0.135$, $c = 0.044$, and $d = -0.0059$.

This empirical correlation gives a convenient method of working out the best configuration for a multi-train plant. In particular, the optimal value of *SW* for the *j*th stage of the plant (*SWO*) is

$$SWO_j = \frac{K}{X_j\alpha}\ln\left\{\frac{r-\alpha}{1-\alpha}\right\} \tag{A4}$$

Here $K = Y/[\epsilon(1 - \theta]^2$, and this means that $SWO_j = (K/X_j)\beta(r_j)$, where $\beta(r_j)$ is the value of b for the particular stage where $r = r_j$. Therefore, for the section overall,

$$SWO = \sum_{j=1}^{N} \frac{K}{X_j}\beta(r_j)$$

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For the case where $j \geq 2$, only terms that contain X_j in this summation are

$$\frac{1}{X_{j-1}} \beta(r_{j-1}) + \frac{1}{X_j} \beta(r_j) = F \tag{A5}$$

At the optimum, $\partial F/\partial X_j = 0$, so differentiation of Eq. (A5) will give

$$\frac{\partial F}{\partial X_j} = \frac{1}{X_{j-1}} \beta'(r_{j-1}) \frac{dr_{j-1}}{dX_j} - \frac{1}{(X_j)^2} \beta(r_j) + \frac{1}{X_j} \beta'(r_j) \frac{dr_j}{dX_j}$$

$$= \frac{1}{X_{j-1}} \beta'(r_{j-1}) \frac{1}{X_{j-1}} - \frac{1}{(X_j)^2} \beta(r_j) + \frac{1}{X_j} \beta'(r_j) \left[- \frac{X_{j+1}}{(X_j)^2} \right]$$

Multiplying the last equation by $(X_j)^2$ gives a series of $N - 1$ equations for the optimal r_j values for $j \geq 2$, such that

$$(r_{j-1})^2 \beta'(r_{j-1}) - \beta(r_j) + (r_j)\beta'(r_j) = 0 \tag{A6}$$

The other equation needed to specify this solution is the product of the r_j values over the cascade as a whole, which will give

$$r_1 r_2 r_3 \cdots r_N = Y/X_F \tag{A7}$$

Solution of this set of equations will specify the cascade containing N mixer-settler trains in a section of the plant.

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NOMENCLATURE

A, B	constants in Eq. (11)
C_1, C_2	constants in the U vs u relation. Eq. (1)
N	number of stages in a mixer-settler train
r	$= (1 + \epsilon)/(1 + \theta\epsilon)$
R	ratio of flow in the organic flow in a train divided by the product flow
SW	separative work
T	Tails/product flow ratio
u	UO_2^{2+} concentration in the aqueous phase (g/L)
U	UO_2^{2+} concentration in the organic phase (g/L)
W	isotopic composition of the tails
x	isotopic composition in the aqueous phase
y	isotopic composition in the organic phase
z	U^{4+} concentration in the aqueous phase (g/L)

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Greek Symbols

ϵ isotope enrichment constant (= 0.0018)
θ ratio of UO_2^{2+} in the aqueous phase ($= u/u + z$)
Φ volume aqueous divided by organic treated in mixer-settler units

Subscripts

j train number
n stage number in a train

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