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Structural Aspects of Hydrometallurgical Solvent Extraction

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ABSTRACT.

Hydrometallurgical liquid extraction systems have previously been modeled using sum-of-the-rates (SR) forms, a noted exception is the procedure outlined by Chapman for a single cascade with two hypothetical solutes. In this paper Chapman's simultaneous convergence (SC) procedure is extended to use in a real multicascade, multisolute system with competing mass action equilibrium expressions. Methods for inclusion of nonstandard specifications directly into the convergence procedure are examined. A finite region of convergence from initial estimates of nonstandard specifications is developed.

It was found that for the base extraction case (iteration only on concentration variables for both phases) that convergence was rapid, no special scaling or initial iteration procedures were required. The inclusion of the specifications into the problem was found to: 1) reduce the region of convergence; and 2) require damped newton iterates for convergence.

INTRODUCTION

Solvent extraction has been widely used as a separation process in the chemical, petrochemical, food, and pharmaceutical industries. Computer modeling of the behavior of extraction cascades for these industrial applications has relied on analogies with the vapor-liquid equilibrium separations of absorption and distillation combined with the additional complexity of needing to describe the composition dependent liquid-liquid equilibria. Using this approach dual solvent systems and multiple cascade organic flowsheets with and without reflux are easily modeled (eg. Bouvard [1]). Models for single solute transfer (eg. McSwain and Durbin [2]) and multisolute transfer (Ricker et al. [3]) for single cascades are well known. The advantage of using an SC (simultaneous convergence) scheme over an SR (sum-of-the-rates) scheme has been documented for organic systems [1,3,4].

In contrast, computer modeling of hydrometallurgical systems has not been as extensively examined. The systems are usually complex, with highly non-linear phase equilibrium expressions combined with reaction kinetics and mass transfer effects (eg. Kojima and Miyauchi [5]). Computational schemes for specific systems have usually torn the full equation set, and reduced it to an SR form; or have included the reaction equilibria and the phase equilibria but only by defining a combined rate [8]. Incorporation of multiple competing complex equilibrium expressions for the reactions in a multiple cascade system is the focus of this research.

Use of a simultaneous convergence (SC) scheme for a hypothetical hydrometallurgical system in a single cascade has been discussed by Chapman [9]. He solved a single copper exchange reaction and a sequence of two hypothetical exchange reactions in a single cascade of equilibrium stages. Simultaneous convergence required no special initialization procedures utilized by others [10,6]. He performed parametric specification studies by externally manipulating the specifications and iterating on the concentrations alone. Although backmixing effects are significant in most extraction processes [3,11] he did not include them in his model. This research extends his work by including multiple cascades, backmixing effects in the model, examining the behavior of a real system that has stronger non-linearities, and examining procedures for including specifications directly into the iteration scheme.

Process Model For Hydrometallurgical Solvent Extraction

The basic process model includes a stage with backmixing for each phase even though evidence exists that: 1) significant backmixing is present only in the

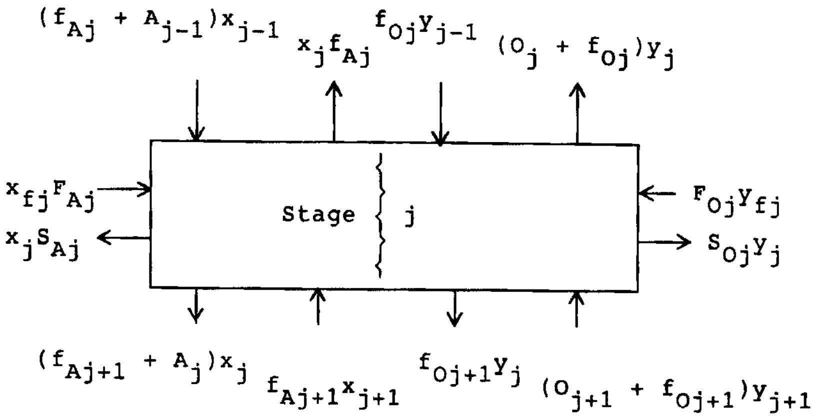

Fig. 1. A typical stage representation.

continuous phase [11] and that 2) backmixing is a function of position in the cascade [12]. A representation of any solute on a typical stage is shown in figure 1.

The material balance for each species can then be written as follows:

$$\begin{aligned}&(f_{Aj} + A_{j-1})x_{j-1} + f_{Aj+1}x_{j+1} + f_{Oj}y_{j-1}\\&+(O_{j+1} + f_{Oj+1})y_{j+1} - (f_{Aj+1} + A_j + S_{Aj})x_j - f_{Aj}x_j\\&- f_{Oj+1}y_j - (O_j + f_{Oj} + S_{Oj})y_j + x_F F_{Aj} + y_{Fj}F_{Oj} = 0 = M_j\end{aligned}$$

Equilibrium is assumed between the concentrations on each stage for each species that distributes with a mass action equilibrium expression of the form

$$M^{n+} + nA^- + aS \rightleftharpoons MA_n \cdot aS$$

with the corresponding equilibrium relationship

$$y_M - K\, x_M\, x_A^{\,n}\, y_S^{\,a} = 0 \qquad E_j$$

The extractant concentration (S) is established by iteration utilizing an inventory balance in the organic phase assuming that the extractant is completely immiscible in the aqueous phase. The aqueous anion, A^-, is likewise assumed to only be present in the aqueous phase.

An entire cascade system can thus be described by the following equations.

For species that distribute:

$$M = (f_{Aj} + A_{j-1})x_{ij-1} + f_{Aj+1}x_{ij+1} + f_{Oj}y_{ij} + (O_{j+1} + f_{Oj})y_{ij+1} - (f_{Aj+1} + A_j + S_{Aj})x_{ij} - f_{Aj}x_{ij} - (O_j + f_{Oj} + S_{Oj})y_{ij} + x_{iF}F_{Aj} + y_{iF}F_{Oj} = 0$$

$$E_{ij} = y_{ij} - k_i x_{ij}(x_A)^n (y_S)^a = 0$$

For an extractant:

$$Ex = y_S + ny_{HS} + my_{MS} - y_{Sfeed} = 0$$

For the aqueous anion, A^-:

$$C = x_A - nx_H - mx_M - 1x_{In} = 0$$

For any inextractable aqueous cation:

$$In = x_{In} - x_{Infeed} = 0$$

This formulation only includes those concentrations which are measurable thus avoiding the necessity of converging on interface concentrations.

An Example Problem

The problem chosen for detailed analysis is found as an example in Nuclear Chemical Engineering by Benedict, Pigford and Levi [13]. The problem is the separation of zirconium from hafnium discussed by Hure and Saint James [14]. A zirconium fraction recovery of .98 and a decontamination factor of zirconium from hafnium of 200 are desired in the extract stream. Interlinked extraction and scrubbing cascades are used to accomplish this task. The solution proposed by Benedict, Pigford and Levi tears all the material balances in each cascade individually. The material balance condition around the feed stage is not met in their analysis.

The feed for the column consists of 3.5M $NaNO_3$, 3.0M HNO_3, .123M $Zr(NO_3)_4$, and .00246M $Hf(NO_3)_4$. The scrubbing section has an aqueous feed of 3.0M HNO_3 and 3.5M $NaNO_3$. The extraction section receives an organic feed containing 2.25M TBP and 1.6M HNO_3. The volume of organic feed is 100 liters and the aqueous feeds are both 48 liters. The problem is illustrated in figure 2.

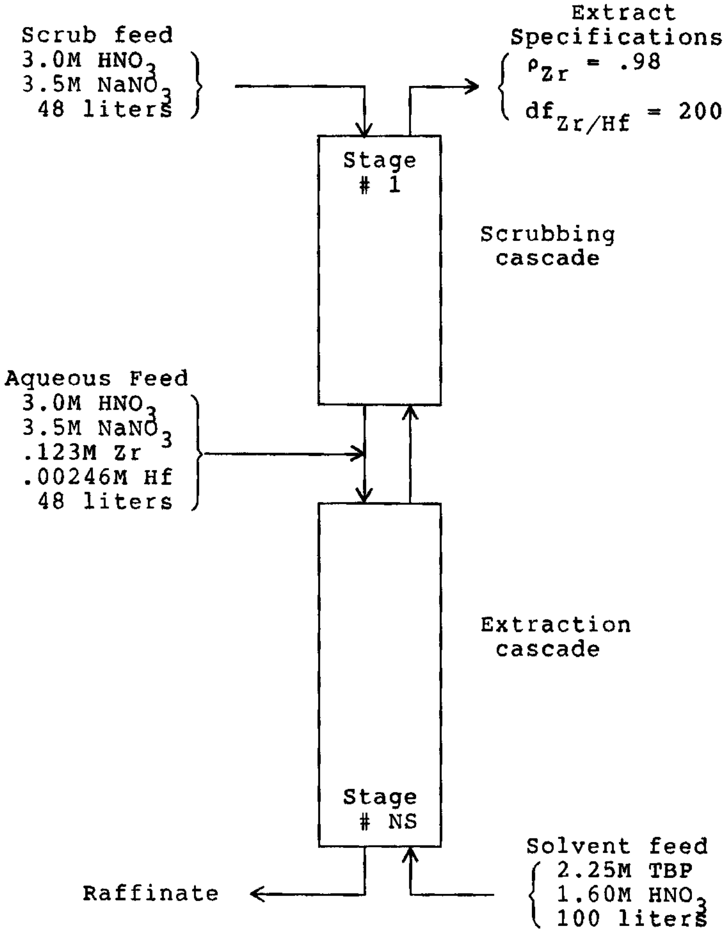

Fig. 2. Problem specifications.

For this example it is assumed that: Each stage is an equilibrium stage; The aqueous and the organic flows are immiscible, and only a central aqueous feed is added. The effect of specifying equilibrium stages without backmixing is to strengthen (stiffen) the non-linear aspects of the problem. Thus the stage model is reduced to that shown in figure 3.

There are nine variables for each stage representing the problem.

x-H,	Concentration of HNO_3 in aqueous phase.
y-H,	Concentration of $HNO_3 \cdot TBP$ in organic phase.
x-Zr,	Concentration of Zr^{+4} in aqueous phase.
y-Zr,	Concentration of $Zr(NO_3)_4 \cdot 2(TBP)$ in organic phase.
x-Hf,	Concentration of Hf^{+4} in aqueous phase.
y-Hf,	Concentration of $Hf(NO_3)_4 \cdot 2(TBP)$ in organic phase.
x-NO3,	Concentration of NO_3^- in aqueous phase.
y-TBP,	Concentration of free TBP in organic phase.
x-Na,	Concentration of Na^+ in aqueous phase.

The nine equations relating these variables on each stage:

(1) Three mass balances for H^+, Zr^{+4}, and Hf^{+4}:

$$A_{j-1}x_{i,j-1} + O_{j+1}y_{i,j+1} + A_f x_{if} - A_j x_{ij} - O_j y_{ij} = 0$$

where i = H, Zr, Hf, j = no. stage, and f represents an aqueous feed.

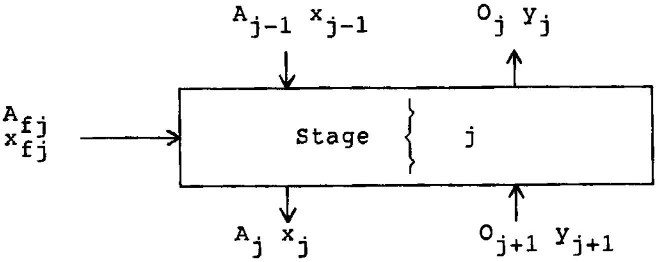

Fig. 3. Stage representation.

(2) Three equilibrium equations relating x-H to y-H, x-Zr to y-Zr, and x-Hf to y-Hf are:

$$y_H - k_H x_H x_{NO3} y_{TBP} \quad = 0 = \quad E_H$$
$$y_{Zr} - k_{Zr} x_{Zr} x_{NO3}^4 \, y_{TBP}^2 \quad = 0 = \quad E_{Zr}$$
$$y_{Hf} - k_{Hf} x_{Hf} x_{NO3}^4 \, y_{TBP}^2 \quad = 0 = \quad E_{Hf}$$

This form has the desired property that as any concentration approaches zero these equations will still hold. The disadvantage is the strong non-linearity with respect to NO_3^- and TBP.

(3) A nitrate chemistry balance in the aqueous phase is:

$$x_{NO3} - x_H - x_{Na} - 4(x_{Zr} + x_{Hf}) = 0 \quad = \quad C_i$$

This balance represents the nitrate chemistry in the aqueous phase. In this case it also represents a charge balance. However, in general this is not true. The addition of other anionic species that are either neutral or that can complex with the cationic aqueous species would require a similar competing balance.

(4) An extractant inventory balance:

$$y_{TBP} + y_H + 2(y_{Zr} + y_{Hf}) - yf_{TBP} = 0 \quad = \quad Ex_i$$

This assumes that the extractant is insoluble in the aqueous phase.

(5) An inextractable species material balance:

$$x_{Na} - x_{Nac} = 0 \quad = \quad In_i$$

Where $x_{Nac} = (x_{Na1} A_1 + x_{Naf} A_f)/A_j$ is calculated at each stage.

Based on previous formulations for other separation processes (primarily distillation) these equations would be ordered with the aqueous phase concentrations coupled to the material balances and the organic phase compositions coupled to the equilibrium expressions on each stage [4].

$$\begin{bmatrix} M_i \\ C_i \\ In \\ E_i \\ Ex \end{bmatrix} \begin{bmatrix} x_i \\ x_{NO3} \\ x_{Na} \\ y_i \\ y_{TBP} \end{bmatrix} = \begin{bmatrix} 0 \\ 0 \\ 0 \\ 0 \\ 0 \end{bmatrix}$$

However in this case it is advantageous to order the equations differently. The resulting equations are ordered as follows:

$$\begin{bmatrix} M_H \\ E_H \\ M_{Zr} \\ E_{Zr} \\ M_{Hf} \\ E_{Hf} \\ C \\ Ex \\ In \end{bmatrix}_j \begin{bmatrix} x_H \\ y_H \\ x_{Zr} \\ y_{Zr} \\ x_{Hf} \\ y_{Hf} \\ x_{NO3} \\ y_{TBP} \\ x_{Na} \end{bmatrix}_j = \begin{bmatrix} 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \end{bmatrix}$$

This arrangement preserves the block tridiagonal form of the Jacobian because the concentrations are only dependent on stage j, stage j-1, and stage j+1. Utilizing the method of Newman[15] A_j represents the submatrix for the stage j-1, B_j the submatrix for stage j, and D_j the submatrix for stage j+1. The form of these matrices are shown in figures 4a,4b, and 4c.

These equations were solved simultaneously utilizing the procedure suggested by Newman [15,4], no special scaling or initialization procedures were required. This result parallels that found by others [1,3].

Results For the Unspecified System

The following results were obtained converging the above system of block tridiagonal equations. After rapid convergence, a physically meaningful profile developed for each component showing the distribution of all nine species for each stage. After the profile was obtained the recovery and the decontamination factors were calculated. These are shown in Table 1. Specifying a different number of stages in the scrubbing section and the extraction section several different recovery factors and decontamination factors were obtained. The proposed solution of 10 stages with feed stage 5 is shown here to be a valid solution for ρ greater than .98 and a df greater than 200.

Analogous to the solution in Benedict, Pigford and Levi[13], figure 4.20, the profiles for the concentrations in the organic phase are shown as a function of stage location in figures 5-8.

$$A_j = \frac{\delta F_j}{\delta x_{j-1}} = \begin{array}{c|ccccccccc|}
 & x_H & y_H & x_{Zr} & y_{Zr} & x_{Hf} & y_{Hf} & x_{NO3} & y_{TBP} & x_{Na} \\
\hline
M_H & + & & & & & & & & \\
E_H & & & & & & & & & \\
M_{Zr} & & & + & & & & & & \\
E_{Zr} & & & & & & & & & \\
M_{Hf} & & & & & + & & & & \\
E_{Hf} & & & & & & & & & \\
C & & & & & & & & & \\
Ex & & & & & & & & & \\
In & & & & & & & & & \\
\hline
\end{array}$$

Fig. 4a. The form of the submatrix A.

$$B_j = \frac{\delta F_j}{\delta x_j} = \begin{array}{c|ccccccccc|}
 & x_H & y_H & x_{Zr} & y_{Zr} & x_{Hf} & y_{Hf} & x_{NO3} & y_{TBP} & x_{Na} \\
\hline
M_H & + & + & & & & & & & \\
E_H & + & + & & & & & + & + & \\
M_{Zr} & & & + & + & & & & & \\
E_{Zr} & & & + & + & & & + & + & \\
M_{Hf} & & & & & + & + & & & \\
E_{Hf} & & & & & + & + & + & + & \\
C & + & & + & & + & & + & & + \\
Ex & & + & & + & & + & & + & \\
In & & & & & & & & & + \\
\hline
\end{array}$$

Fig. 4b. The form of the submatrix B.

$$D_j = \frac{\delta F_j}{\delta x_{j+1}} = \begin{array}{c|ccccccccc|} & x_H & y_H & x_{Zr} & y_{Zr} & x_{Hf} & y_{Hf} & x_{NO3} & y_{TBP} & x_{Na} \\ M_H & & + & & & & & & & \\ E_H & & & & & & & & & \\ M_{Zr} & & & & + & & & & & \\ E_{Zr} & & & & & & & & & \\ M_{Hf} & & & & & & + & & & \\ E_{Hf} & & & & & & & & & \\ C & & & & & & & & & \\ Ex & & & & & & & & & \\ In & & & & & & & & & \end{array}$$

Fig. 4c. The form of the submatrix D.

Table 1
Recovery fractions and decontamination factors for several stages.

Total Stages	Feed Stage	Recovery	Decontamination
3	2	.8225	13.40
4	2	.9145	14.69
5	2	.9581	15.36
6	2	.9793	15.70
7	2	.9898	15.90
8	2	.9950	15.99
9	2	.9975	16.03
4	3	.8140	36.43
5	3	.9097	40.42
6	3	.9555	42.56
7	3	.9780	43.79
8	3	.9891	44.22
9	3	.9947	44.53
5	4	.8118	95.82
7	4	.9548	115.60
8	4	.9776	117.80
9	4	.9889	119.40
10	4	.9945	119.30
6	5	.8111	253.90
7	5	.9080	286.60
8	5	.9546	298.10
9	5	.9775	291.30
10	5	.9889	303.90
20	10	.9997	.118e+8

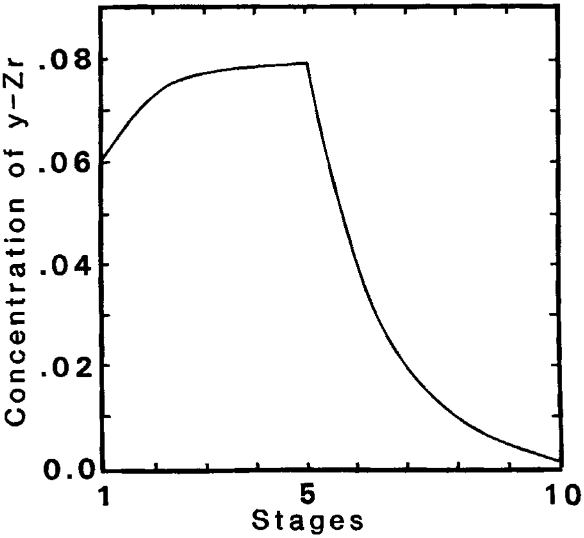

Fig. 5. Concentration of Zr in organic phase vs. stage location.

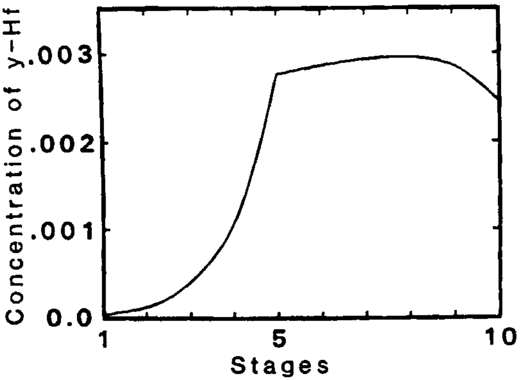

Fig. 6. Concentration of Hf in organic phase vs. stage location.

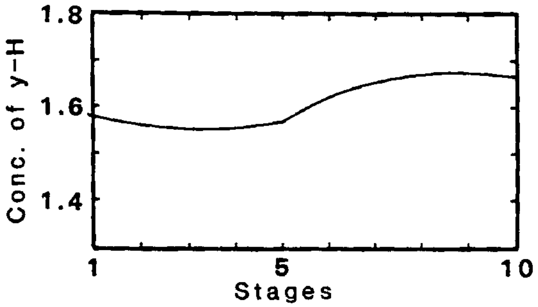

Fig. 7. Concentration of H in organic phase vs. stage location.

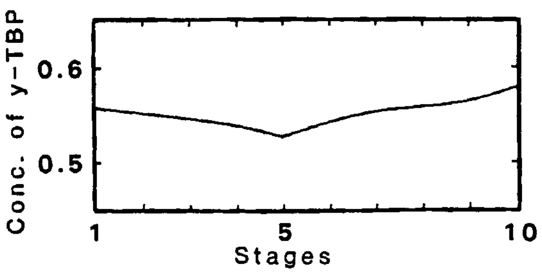

Fig. 8. Concentration of free TBP vs. stage location.

Procedure For Including Problem Specifications

An examination was performed to determine if an efficient method for including the specifications directly into the iteration scheme could be accomplished. Paralleling the results obtained for organic systems [1], the specifications were directly included in place of the material balances for the required components as follows:

For each stage the nine component equations above were used. For the first stage (the first scrubbing stage) equations using the recovery factor ρ and the decontamination factor df replaced the material balance equations for Zr and Hf, i.e.

$$A_f \cdot x_{Zrf} \cdot \rho - y_{Zr1} \cdot O_1 = 0 = M_{Zr1}$$

$$x_{Zrf} \cdot y_{Hf1} \cdot fd - x_{Hff} \cdot y_{Zr1} = 0 = M_{Hf1}$$

Convergence was difficult to achieve without negative concentrations appearing in the profile. To prevent large fluctuations and negative values damped iterations were used. For each negative value obtained the distance from the previous value and zero was measured and the half of that distance divided by the total distance was used as the damping factor for all values. If no value was negative a full iteration was used.

Specifying ρ = .98 and df = 200 converged profiles developed for any number of stages. A profile would develop for 20 stages as well as for 3 stages. The major reason for this ease of convergence was the absence of compliance with the material balance for Zr and Hf in stage one. Calculating this balance discrepancy showed, as expected, that the further one was from the proposed solution of 10 stages (feed stage 5) the larger the discrepancy. These calculations are shown in table 2.

The specification for decontamination was eliminated and the material balance for Hf reinstated. Now only the equation for ρ was used in stage one. Converged profiles developed again. The profiles for Zr were nearly identical to the ones obtained with both ρ and df specified. The amount of Hf present at stage one was probably too small to have an influence on the concentration of Zr.

That profiles developed for all stages implied that the equations used did not sufficiently specify the problem. Either the specifications for ρ and df were met or the material balances for Zr and Hf were met but all restrictions were never met simultaneously.

The next consideration was the addition of the specifications to the material balance for the first stage. The material balance for Zr was added to the

Table 2
Balance discrepancies for Zr and Hf.

Stages	Discrepancy for Zr	Discrepancy for Hf
20	.1162e-2	-.1475e-4
10	.5257e-3	-.5222e-5
5	-.4182e-2	.5664e-4
3	-.9717e-2	.2035e-3

specification equation for ρ to form M_{Zr1}. Likewise the material balance for Hf was added to the specification equation for df to form M_{Hf1}.

Converged profiles developed again for all stages specified but neither the material balances nor the specifications were met. A trade-off between the two spliced equations was taking place to reach a converged profile as shown in table 3.

To make the specification equations more dominant the part of M_{Zr1} specifying ρ and the part of M_{Hf1} specifying df were squared. At the same time the residuals from each equation part became a part of the criteria for convergence meaning it had to be less than 1.e-8. After less than ten iterations a repeating profile developed. The implication of these results was that the specification equations could not replace the species material balance equations.

Following the procedure suggested by Newman [16] an additional equation is added to each stage for each specification to be included. To include the specifications for ρ and df two new variables would have to be found. The aqueous and the organic flows were chosen. The equation for the aqueous flow through the column was:

$$A_{j-1} - A_j + A_f = 0 = A_j$$

The equation for the organic stream was similar:

$$O_{j+1} - O_j = 0 = O_j$$

At stage one the specification equation for ρ and df were used in place of the material balances for the flows. The equation specifying ρ replaced the material

Table 3
Values for ρ, df and mass balance discrepancies.

Stages	ρ	df	Discrepancy for Zr	Discrepancy for Hf
20	.9898	179.1	.5807e-3	.1664e-4
10	.9844	191.9	.2629e-3	.5978e-5
5	.9447	378.2	.2083e-2	.6474e-4
3	.8995	106.2e5	.4752e-2	.2306e-3

balance for the aqueous flow. The equation for df replaced the organic mass balance but the organic stream leaving stage one would change to satisfy the df equation. Replacing the mass balance for the aqueous flow caused no problem since the stream leaving stage one was incorporated in the balance for stage two.

Incorporation of the ρ specification alone coupled with the aqueous flows converged rapidly to a solution. The organic flow would stay at the original guessed value and the aqueous flow would adjust to meet the recovery specification.

To balance the mass balance for the organic flow through the column the ideal place for the df specification is at the last stage. The overall mass balance for the hafnium was used to express the concentration at the end of the cascade as follows:

The overall hafnium mass balance is

$$A_f\ x_{Hff}\ -\ y_{Hf1}\ O_1 = A_{NS}x_{HfNS}$$

The $y_{Hf1}O_1$ term is eliminated using the definitions for ρ and df.

Here the recovery fraction

$$\rho = \frac{O_1 y_{Zr1}}{A_f x_{Zrf}}$$

Is solved for y_{Zr1} and combined with the definition for df

$$df = \frac{\rho_{Zr}}{\rho_{Hf}} = \frac{y_{Zr1}x_{Hff}}{y_{Hf1}x_{Zrf}}$$

to obtain and expression for $O_1 y_{Hf1}$

$$O_1 y_{Hf1} = \frac{\rho\ A_f x_{Hff}}{df}$$

With the hafnium balance included, the equation used for df at the last stage could now be expressed as:

$$(df - \rho)\ A_f\ x_{Hff} - df\ A_{NS}\ x_{HfNS} = 0 \quad = \quad O_{NS}$$

Using the above equation at the last stage and the equation for ρ at the first stage a profile developed which satisfied all the criteria specified. An aqueous scrub flow to aqueous feed flow ratio (A/A_f) equal to .5688 and an aqueous scrub flow to organic flow ratio (A/O) equal to .3906 was obtained using ten stages. The solution proposed in Benedict, Pigford and Levi[13] gave ratios for ten stages of A/A_f = 1.0 and A/O = .48.

If eight stages were used, an A/A_f ratio equal to 2.081 and an A/O ratio equal to .6244 was obtained. This development showed that the desired specifications could be met at least two different ways. If ten stages were used lower flowrates than those found in the original problem could accomplish the specified separation. On the other hand if higher flowrates were desired less stages could be used. In this example eight stages were the lowest number of stages that would give the desired result but more than ten stages were possible.

Convergence Procedures For the Specified System

When material balances and equilibrium balances were the only equations present, convergence to a meaningful profile was rapid using full Newton iterates. No particular attention had to be paid to the initial estimates. Using the feed concentrations and flat concentration profiles lead to rapid convergence but even unusual estimates would converge. Introducing the aqueous and the organic balances and the specifications for ρ and df complicated the convergence process. Damped Newton iterates equal to .3 were necessary for convergence. The process was also sensitive to initial estimates. As the solid line on figure 9 shows, only certain combinations of initial flow estimates for aqueous and organic flows would converge.

By changing the equation for the inextractable species to a material balance form

$$In(new) = x_{Naj-1}A_{j-1} - x_{Naj}A_j + x_{Naf}A_f = 0$$

the area for convergence expanded in some areas and contracted in other areas as shown by the dashed line in figure 9.

The limiting flow ratios for the extracting-scrubbing cascade in this example as calculated by Benedict, Pigford and Levi [13] were A^*/A_f = .1055 and A^*/O^* = .1146, where A* and O* are the limiting flows. Considering these limitations in conjunction with the region shown on figure 9 would not significantly reduce the region of initial estimates.

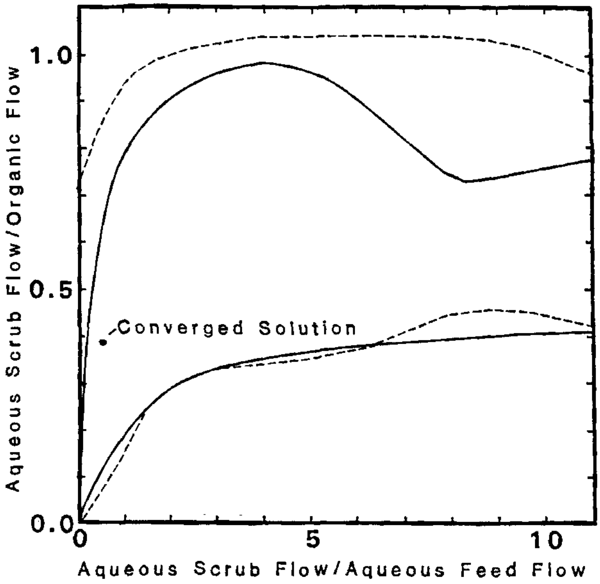

Fig. 9. Initial estimates for aqueous and organic flows.

CONCLUSION

The simulation of the hydrometallurgical separation of zirconium from hafnium was accomplished using a simultaneous convergence method. The non-linear equilibrium equations characteristic of hydrometallurgical operations were inserted directly into the equation set. This approach to solving non-linear equations has the advantage that all concentrations, both aqueous and organic, are solved simultaneously. No scaling or initial iteration procedures were found to be necessary for convergence. The equilibrium equations were also formulated in such a way that concentrations equal to zero would not upset the scheme. A physically meaningful profile for the system under consideration developed when the material balances and equilibrium relationships were satisfied for each stage.

The inclusion of non-standard specifications was accomplished by changing the aqueous and the organic flows. Including two new equations, not just adding them to existing equations, ensured the compliance of the mass balance for each species at each stage. In that case damped newton iterates and special initial estimates were necessary for convergence. A reduced region of initial estimates was found. By changing the flows and adjusting the number of stages, ways to achieve the separation specifications were found. Fewer stages with greater flows and more stages with smaller flows would also meet them.

Notation.

A	Aqueous flow
A_f	Aqueous feed
A^-	Aqueous anion concentration
A^*	Limiting aqueous flow
df	Decontamination factor
f	Backmixing flow
k	Equilibrium constant
M^{n+}	Metal ion concentration
O	Organic flow
O^*	Limiting organic flow
S	Extractant concentration
x_{ij}	Concentration of species in aqueous flow i is species, j is stage
x_f	Feed concentration in aqueous flow
y_{ij}	Concentration of species in organic flow
y_f	Feed concentration in organic flow
ρ	Recovery fraction

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