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Transient- and Steady-State Concentration Profiles in a DIAMEX-like Countercurrent Process for An(III) + Ln(III) Separation

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ABSTRACT

A new version of the DIAMEX (DIAMide EXtraction) process is reported to separate trivalent Am,Cm (An) and lanthanides (Ln) from a PUREX (Plutonium Uranium Refining by EXtraction by tributyl phosphate) process raffinate at a high aqueous/extractant flow-rate ratio while taking care of prevent third-phase formation. The process has been modeled by a computer program and tested on a 16-stage mixer/settler battery to perform extraction, scrubbing, and stripping to obtain an Am (representing the mixture of Am + Cm) decontamination factor (feed/raffinate) higher than

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10^3 and a Mo + Zr fission product content in the product lower than 1 wt% of that of Am. Transient and steady-state concentration profiles of the solutes have been calculated and experimentally determined, and good agreement was generally observed.

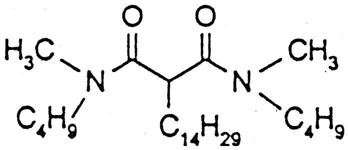
Key Words. Liquid–liquid extraction; Partitioning; Transuranium elements; DIAMEX process

INTRODUCTION

Minor actinides [MA = AnIII(Am + Cm) + Np] separation from spent nuclear fuel reprocessing wastes (HLLW = high level liquid wastes) for their transmutation or for separate conditioning and disposal, is an attractive option for reducing the environmental long-term pollution risk (1).

High MA separation factors from HLLW (e.g., $>10^3$), are required and the separated fraction has to be sufficiently free of other elements if nuclear transmutation is envisaged. Owing to the high concentrations of nitric acid and of trivalent lanthanides (Ln) in the HLLW, the selective separation of MA has been investigated by two-step processes, each based on liquid–liquid extraction to first separate An + Ln from the bulk of the fission product mixture and then An from Ln. Many organophosphorous compounds have been studied for use in each step of the partitioning processes (2–6), e.g., bifunctional extractants of the carbamoyl-methyl-phosphoryl type [e.g., CMPO = octyl(phenyl)-(*N*,*N*-diisobutyl carbamoylmethyl)phosphine oxide] and alkyl-phosphoric acids (HDEHP = diethylhexyl ortho-phosphoric acid, and DIDPA = diisodecyl ortho-phosphoric acid). Within the framework of the 4th European Nuclear Fission Safety Program, the NEWPART research contract (1996–1999) has been set up to define methods of AnIII(Am + Cm) separation from a PUREX process raffinate (HLLW) without modifying the HLLW composition or the Am and Cm stable oxidation state (i.e., +III). Moreover, a CHON-type extractant (i.e., an extractant composed only of C, H, O, and N) has been recommended in view of the possibility of decomposing it into gases that can be freely released into the atmosphere at the end of its use. The DIAMEX process (7–10), which is based on the use of a malonamide extractant, has been selected to perform the first step of the process, and several extractants are under study to define the second step for the separation of An from Ln.

The purpose of the study reported in the present paper was the development of a new version of the DIAMEX process. A comparison of the transient and steady-state solute concentration profiles was evaluated by a computer model with the experimental results obtained by using a mixer/settler (M/S) battery. ENEA and PoliMi developed the new version of the DIAMEX process based on the reference extractant dimethyldibutyltetradecylmalonamide (DMDBT-DMA, named C14 in this paper). Its structure is



A 1 mol/L solution (instead of 0.5 mol/L as for the first DIAMEX flow sheet) of C14 in hydrogenated tetrapropene (TPH) as the diluent was used in order to 1) minimize the occurrence of third-phase formation (11, 12), which allows the nitric acid concentration in the feed to be increased (making the feed more similar to real HLLW); and 2) reducing the organic/aqueous phase flow-rate ratio in two process sections (extraction/scrubbing) due to the higher extractant concentration. To reduce Mo and Zr fission product co-extraction with An + Ln, oxalic acid was added both to the feed (in order to complex them, thus reducing their extraction) and to the scrubbing solutions to improve their backextraction. Experimentally measured distribution ratios of all the feed constituents were used as input data for the computer model. The operating conditions were defined for a 16-stage M/S battery to reach the following objectives:

- Am and Cm (simulated by Eu) decontamination factors (feed/raffinate) higher than 10^3
- Mo + Zr extracted amount in the product lower than 1 wt% of that of Am
- Low Eu (and then low Cm) accumulation in the scrubbing stages
- No oxalate precipitation in any battery stage

EXPERIMENTAL

Chemicals

Diamide C14, supplied by Panchim (Lisses, 91, France), was purified before use to remove the impurities responsible for emulsion formation when it is shaken with aqueous solutions. Hydraulic tests with nonpurified C14 samples showed long aqueous/organic settling times (days or even weeks) independent of the nitric acid concentration of the aqueous phase.

The C14 purification was performed according to the CEA procedure (9) by passing a 33% wt/vol solution of technical C14 in pentane through an alumina B column. Impurities trapped in the alumina B column were analyzed and found to be mainly tetradecanol [myristyl alcohol = $CH_3(CH_2)_{13}OH$]. Pentane was removed from the percolate by rotovapor distillation, and the C14 thus obtained was used to prepare the extractant solution of 1 mol/L C14 in

TPH. The titration of purified C14 was performed with 0.1 mol/L $HClO_4$ in glacial acetic acid by using a solution of 0.1 mL of purified C14 in 50 mL of acetic acid. All other chemicals were of analytical grade.

The composition of the simulated HLLW used as feed in our tests is reported in Table 1 together with that suggested by CEA-Marcoule (named UOx2), corresponding to a PUREX raffinate coming from the reprocessing of spent nuclear fuel with a burn-up of 45 GWd/t H.M. U and Pu were not considered within the framework of the NEWPART research contract.

This feed has been used to determine the settling times and the solute distribution ratios at different HNO_3 concentrations with and without 0.15 mol/L oxalic acid ($H_2C_2O_4$). The reference HNO_3 concentration in HLLW is 4 mol/L. The feed has been traced with ^{241}Am, ^{152}Eu, and ^{137}Cs to measure the distribution ratios of these elements and to follow their concentration profiles in the M/S battery stages in the transient and in the steady states.

Operating Procedures and Analytical Methods

At the ENEA Saluggia Laboratories, settling times and feed constituent distribution ratios have been measured after 5 minutes of shaking of the feed samples with the extractant (A/O = 1) previously equilibrated twice with

TABLE 1
Composition of the Raffinate from the Reprocessing of an UOx2 (45 GWd/t H.M.) Nuclear Fuel and of the New DIAMEX Process. Test Feed Composition[a]

Element	UOx2 HLLW composition (g/L)	Test feed composition (g/L)
Am	0.117	Trace
Cm	0.018	—
La	0.315	0.339
Cs	0.938	0.941
Eu	0.039	0.042
Sm	0.207	0.213
Mo	0.876	0.936
Ce	0.614	0.638
Zr	0.922	0.749
Nd	1.051	1.117
Pr	0.289	0.287
Gd	0.029	0.032
Rh	0.120	0.211
Ru	0.594	0.688
Pm	0.014	—

[a] The simulated HLLW used as feed in all tests corresponds to the second column.

HNO_3 at the same concentrations of the feed samples. The first extractant equilibration with HNO_3 was performed at a phase ratio of $A/O = 3$, and the second with HNO_3 of the same concentration at $A/O = 1$. Settling times (in seconds) refer to complete phase separation. Total H^+ concentration in the aqueous phase was determinated by potentiometric titration.

Tests have been carried out at the FZ Jülich Laboratories using a standard lab-scale M/S battery, made of Plexiglass, with 16 stages, each of them with a mixer volume of 12.4 mL and a settler volume of 18.8 mL. All the process operations (i.e., extraction, scrubbings, and stripping) were simultaneously performed by this battery in the countercurrent mode.

Inductively coupled plasma/atomic emission spectroscopy (ICP/AES) or inductively coupled plasma/mass spectrometry (ICP/MS) was used to measure the stable isotope content in the aqueous and organic phases (after quantitative backextraction or after microwave destruction).

Standard radiometric assay and counting procedures (gamma spectrometry, LSC, and alpha counting) were used to measure the content of ^{137}Cs, ^{152}Eu, and ^{241}Am in solutions spiked with them. Duplicate assays of each phase were routinely performed, and the material balance of each element was found to be satisfied within ±10%.

RESULTS AND DISCUSSION

Settling Times

Settling times have been measured at 23°C for emulsions made of 1 mol/L C14 in TPH with different aqueous phases: 1) pure nitric acid solutions at different concentrations; 2) HLLW simulating solution (Table 1) at different nitric acid concentrations; 3) HLLW simulating solution (Table 1) mixed with 0.15 mol/L $H_2C_2O_4$ at different nitric acid concentrations.

The results are reported in Table 2. It can be observed that the settling times become shorter at higher nitric acid concentrations, but they increase when the aqueous phase has the HLLW composition of Table 1. The addition of oxalic acid (0.15 mol/L), whose main objective is to reduce Mo and Zr extraction, has the effect of shortening the settling time to values comparable with those obtained with pure nitric acid solutions.

Distribution Ratios

Batch extractions at 23°C were performed to measure the distribution ratios of all the constituents of the HLLW simulating solution at different nitric acid concentrations up to about 4 mol/L (which is the HNO_3 concentration of the reference UOx2 HLLW). Each measurement was repeated in the presence of 0.15 mol/L of oxalic acid.

TABLE 2
Settling Times for Emulsions Made of the Solvent 1 mol/L C14 in TPH and Different Aqueous Phases[a]

Pure HNO_3		Feed (Table 1) + HNO_3		Feed (Table 1) + HNO_3 + 0.15 mol/L $H_2C_2O_4$	
mol/L HNO_3	time	mol/L HNO_3	time	mol/L HNO_3	time
0.05	3 min 40 s				
0.08	3 min 40 s	0.1	9 min		
0.16	3 min 40 s				
0.25	3 min 40 s	0.2	8 min		
0.50	3 min 40 s				
1.0	2 min 50 s				
1.5	2 min 40 s				
2.0	2 min 25 s	1.91	3 min 50 s	1.92	1 min 25 s
2.5	1 min 50 s				
3.0	30 s	2.85	1 min 40 s	2.84	50 s
4.0	30 s	3.70	1 min 40 s	3.72	50 s

[a] The height of each phase in the vial used to measure the settling times was 2 cm.

Nitric Acid Distribution Ratios

They were measured in the HNO_3 concentration range $0.08 \leq x \leq 4.2$ mol/L (at equilibrium in the aqueous phase) and their values are well represented by the following polynomial function of x:

$$D_{HNO_3} = 0.007x^3 - 0.0515x^2 + 0.1493x + 0.0542 \quad \text{(correlation coefficient} = 0.9687) \tag{1}$$

Oxalic Acid Distribution Ratios

They were measured by using the reference waste solution (Table 1) mixed with 0.15 mol/L of oxalic acid and in the HNO_3 concentration range $0.5 \leq x \leq 3.72$ mol/L (at equilibrium in the aqueous phase). Their values are well represented by the following polynomial function of x:

$$D_{H_2C_2O_4} = 0.0032x^2 - 0.0325x + 0.1064 \quad \text{(correlation coefficient} = 0.999) \tag{2}$$

Element Distribution Ratios

Batch extraction tests were performed at 23°C using the HLLW simulating solution with and without 0.15 mol/L of oxalic acid and in the HNO_3 concentration range $1.92 \leq x \leq 3.71$ mol/L (at equilibrium in the aqueous phase).

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TABLE 3
Feed Constituent Distribution Ratios for Different HNO_3 Concentrations without and with 0.15 mol/L $H_2C_2O_4$[a]

HNO_3 (mol/L)	Mo	Ru	Zr	Gd	Rh	Sm	La	Pr	Nd	Am	Eu	Cs
1.92	5.06	0.15	69.8	1.05	0.04	2.11	3.06	3.41	2.81	3.14	1.29	0.0014
*	0.18	0.29	0.08	1.22	0.155	2.14	3.27	3.3	2.82	3.18	1.31	0.0015
2.84	10.84	0.23	213.8	3.4	0.04	6.54	8.44	9.8	8.31	7.87	3.69	0.0013
*	0.29	0.43	0.11	3.72	0.13	6.42	8.49	9.7	8.35	7.78	3.71	0.0014
3.71	15.9	0.26	342	7.33	0.04	11.96	12.6	15.8	13.68	13.14	7.74	0.0015
*	0.56	0.44	0.18	7.45	0.10	11.72	13.1	15.7	13.6	13.11	7.67	0.0014

[a] ^{137}Cs, ^{152}Eu, and ^{241}Am contents in the aqueous and in the organic phase were radiometrically measured and the material balance of each element was found to be satisfied within ± 10%. The aqueous and organic contents of all other elements were determined by ICP/AES and by ICP/MS with an uncertainty range up to ± 20%, and the detection limit was of about 2×10^{-5} mmol/L.

* Same HNO_3 concentration but with 0.15 mol/L $H_2C_2O_4$ added.

The results are reported in Table 3, and they show that the element distribution ratios increase by increasing the nitric acid concentration with only the exceptions of Rh and Cs. Oxalic acid complexes Mo and Zr in the aqueous phase and thus reduces their distribution ratios. Cs is very weakly extracted at the considered nitric acid concentrations independently of the oxalic acid content, which on the contrary has the effect of increasing the Rh and Ru distribution ratios by a factor of 3 to 4 and about 2, respectively.

Samples of loaded extractant were scrubbed with equal volumes of 2.5 mol/L of nitric acid solution mixed with 0.15 mol/L of oxalic acid. A second scrub with 1 mol/L HNO_3 solution followed in order to reduce both nitric and oxalic acid content in the organic phase. Distribution ratios were obtained in agreement with the values of the extraction tests performed under the same conditions; no metal oxalate precipitation was observed.

Samples of extractant, loaded and scrubbed as mentioned before, were shaken with successive equal volumes of 0.2 and 0.1 mol/L HNO_3 solutions to measure the distribution ratios of the stripping operation. From the results reported in Table 4, it can be deduced that some accumulation problem in the extractant could arise for Ru and Rh.

Flow Sheet Choice and Test for An + Ln Separation by a 16-Stage Mixer/Settler Battery

Distribution ratios experimentally measured have been used as input data of a computer program (5, 13) to optimize the operating conditions of a 16-stage

TABLE 4
Metal Ion Distribution Ratios Obtained by Stripping a Loaded and Scrubbed Solvent[a]

Stripping solution	Mo	Ru	Zr	Gd	Rh[b]	Sm	La	Pr	Nd	Am	Eu	Cs
1st 0.2 mol/L HNO_3	0.14	13.8	0.26	0.06	v.h.	0.09	0.04	0.10	0.11	0.09	0.09	1.5*
2nd 0.1 mol/L HNO_3	0.09	9.5	0.23	0.04	v.h.	0.08	0.04	0.10	0.10	0.07	0.06	1.3*

[a] Temperature = 23°C; extraction feed of Table 1 at 3 mol/L HNO_3 + 0.15 mol/L $H_2C_2O_4$; first scrub with 2.5 mol/L HNO_3 + 0.15 mol/L $H_2C_2O_4$; second scrub with 1 mol/L HNO_3.

[b] v.h. = very high.

* Uncertain data owing to the low concentration of Cs remaining in the scrubbed solvent; see also note in Table 3.

battery with the view of reaching the objectives reported in the Introduction. The program simulates the dynamic behavior of countercurrent liquid/liquid extraction processes. It can be employed to develop the transient state (i.e., to evaluate the transient-state solute concentration profiles) or to determine the steady state (i.e., to evaluate the steady-state solute concentrations in both phases at each stage of the M/S battery). The second option (steady-state evaluations) was first adopted to perform simulations based on different sets of operating conditions (i.e., oxalic and nitric acid concentrations in the feed, composition of scrubbing and stripping solutions, phase ratio and stage number of each section of the process, etc.) and that set of operating conditions was chosen for the process reference flow-sheet which better leads to the process objectives. Figure 1 shows the process selected for the tests on the 16-stage battery. The battery transient state was then developed by the computer program according to this flow sheet and by adopting phase flow-rates satisfying the phase settling times reported in Table 2. The assumption of counter current "piston flow" of the phases was made in the computer program, and its inputs were: 1) stage efficiency 0.8; 2) number of aqueous and organic settler zones 5; 3) initial battery configuration made with half the volume of each stage filled with extractant not preequilibrated with HNO_3 and half the volume filled with HNO_3 aqueous solution at 4 mol/L in the extraction section, 3.5 mol/L in the first scrubbing section, 1 mol/L in the second scrubbing section, and 0.1 mol/L in the stripping section; and 4) the organic concentration transient state coming out of the second scrubbing section was used as the feed of the stripping section.

Two runs were performed, the first one at the following flow rates:

- Extractant flow rate = 25 mL/h
- Feed flow rate = 50 mL/h

- First scrub flow rate = 12.5 mL/h
- Second scrub flow rate = 12.5 mL/h
- Stripping flow rate = 50 mL/h

A second test was performed at doubled flow-rates after having verified the good hydraulic behavior of the battery.

Good agreement has been found between the results of the two tests. In what follows, the results of the 2nd test will be reported.

Values have been found in the battery stages for the HNO_3 and $H_2C_2O_4$ steady-state distribution ratios which are a few percentage points different from those obtained by the computer program.

Transient state concentrations of some elements in the product and in the raffinate are reported in Figs. 2 and 3. They lead to the following remarks:

1. A delay of about 1.5 hours with respect to what was expected has been observed for the product concentrations at the beginning of the transients. This was caused by the time required to stabilize the level of the settler interphase at a position which was found to be different from that assumed as the input of the computer program.
2. Good agreement has been found with the calculated concentration transient states of Am and lanthanides, whereas Mo asymptotic concentration values were found to be lower by a factor of about 2 with respect to the calculated ones.

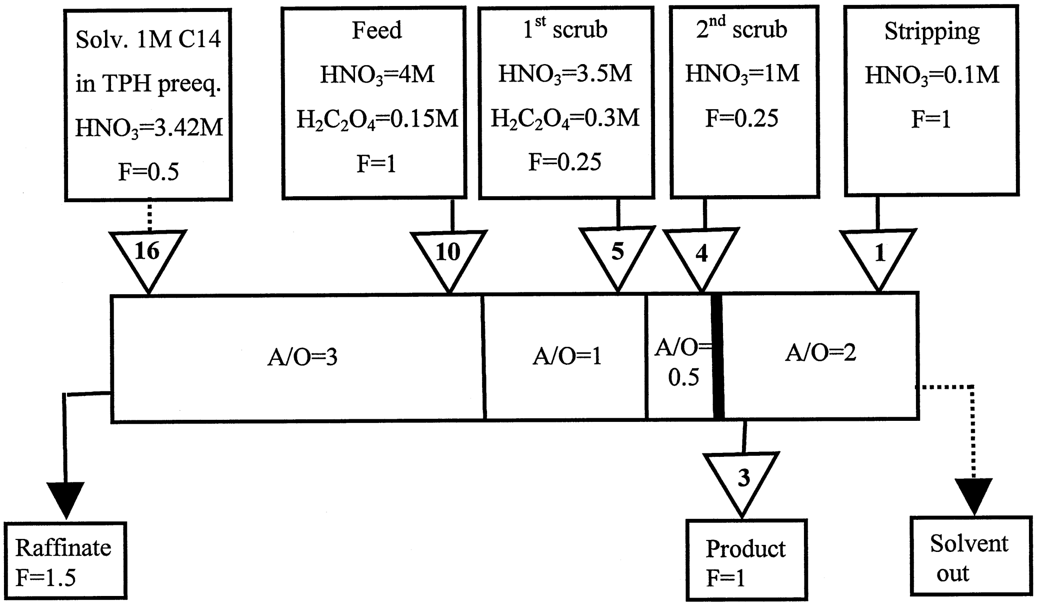

FIG. 1 New DIAMEX flow sheet for the 16-stage mixer/settler battery tests (solvent: C14 1 mol/L in TPH).

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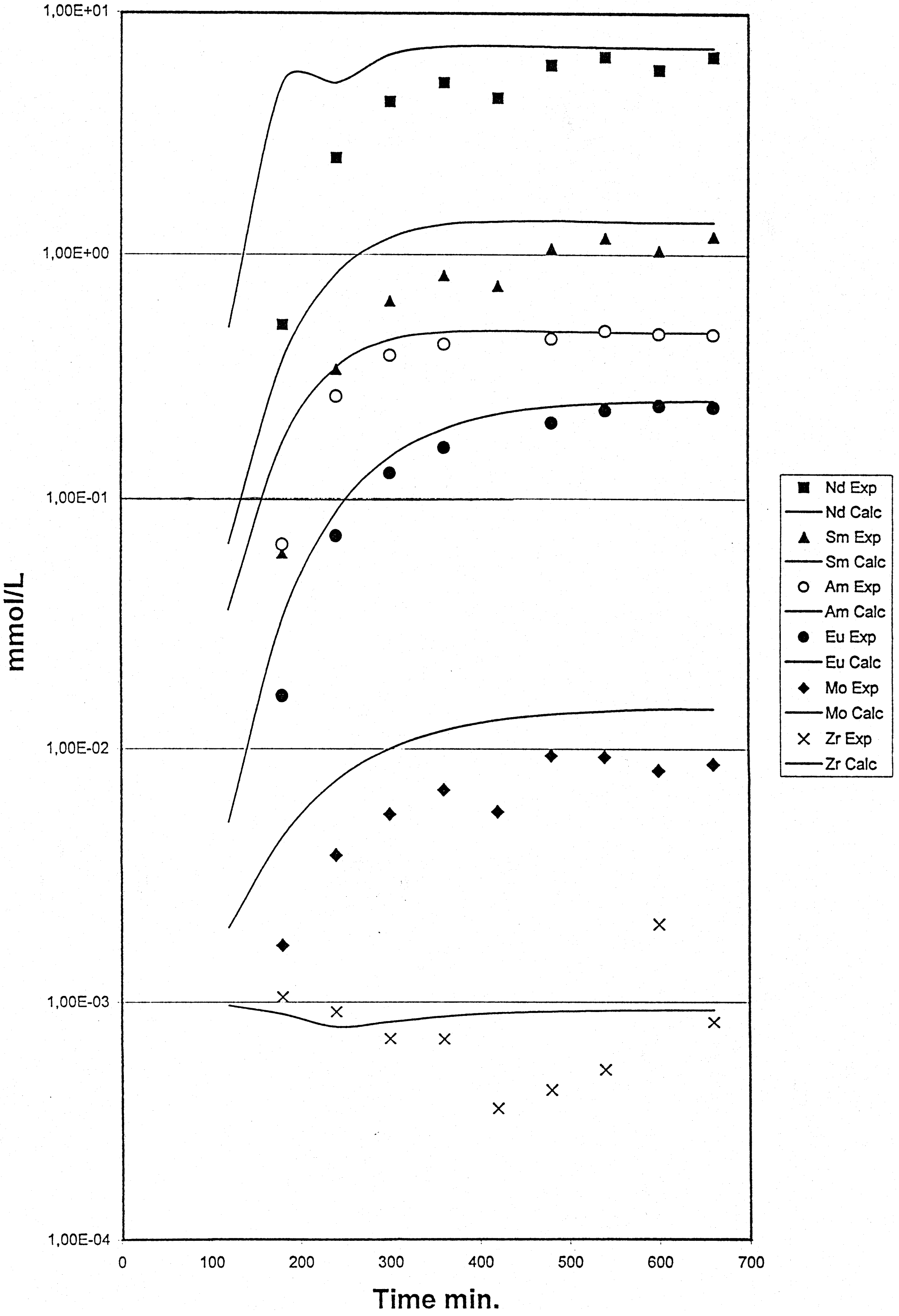

FIG. 2 Transient-state concentration profiles of the product obtained for the second test for Nd(III), Sm(III), Am(III), Eu(III), Mo(VI), and Zr(IV).

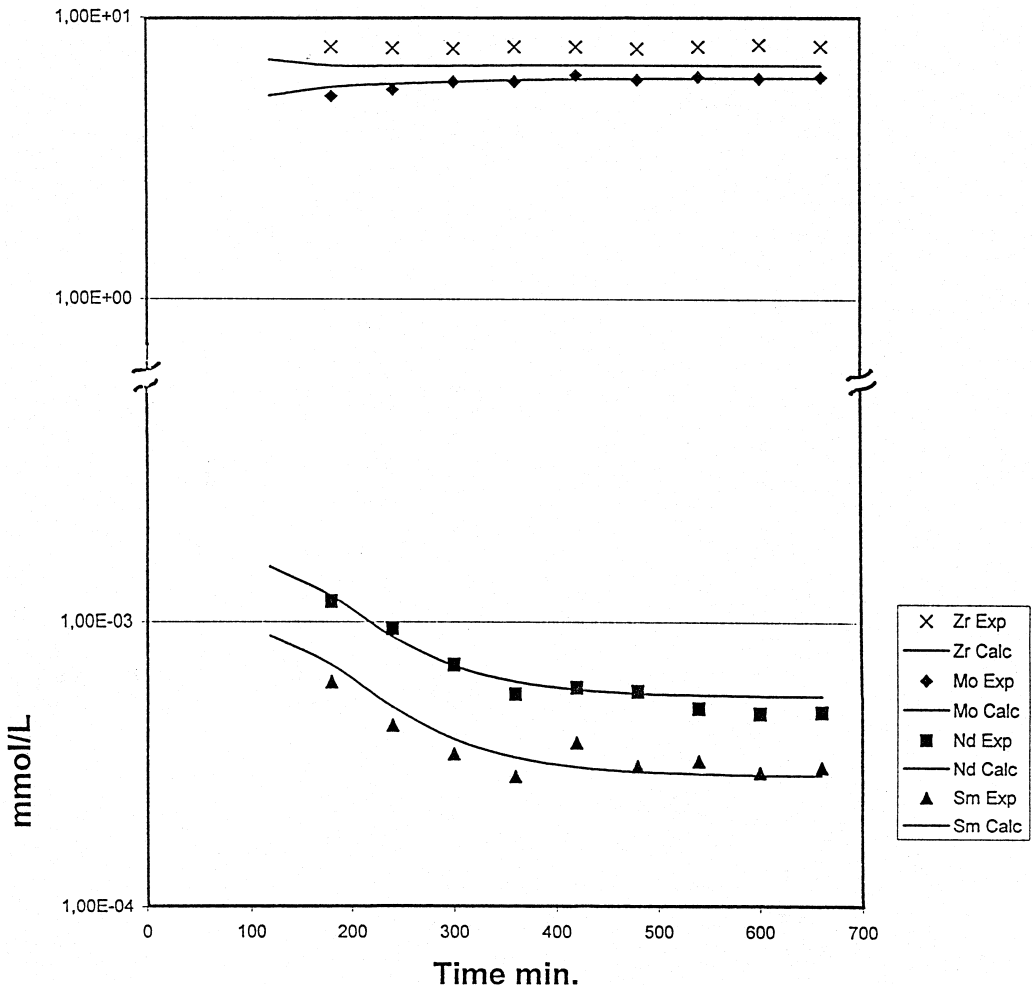

FIG. 3 Transient-state concentration profiles of the raffinate obtained for the second test for Nd(III), Sm(III), Mo(VI), and Zr(IV).

3. Am can be considered to one of the "fastest" elements to be extracted because it approaches the steady-state concentrations in a shorter time whereas Eu and Sm are the "slowest" elements.

The steady-state concentration profiles of some elements are reported in Fig. 4 for the organic and aqueous phases. These results were obtained from samples taken from both phases after the run was stopped; that is, 11 hours after the start. There is general good agreement between calculated and experimental concentration profiles for extraction, scrubbing, and stripping sections for all metallic species. Nevertheless, slight differences between calculated and experimental results are observed for some elements, mainly in the stripping section, which may be due to a misestimate of the stage efficiency.

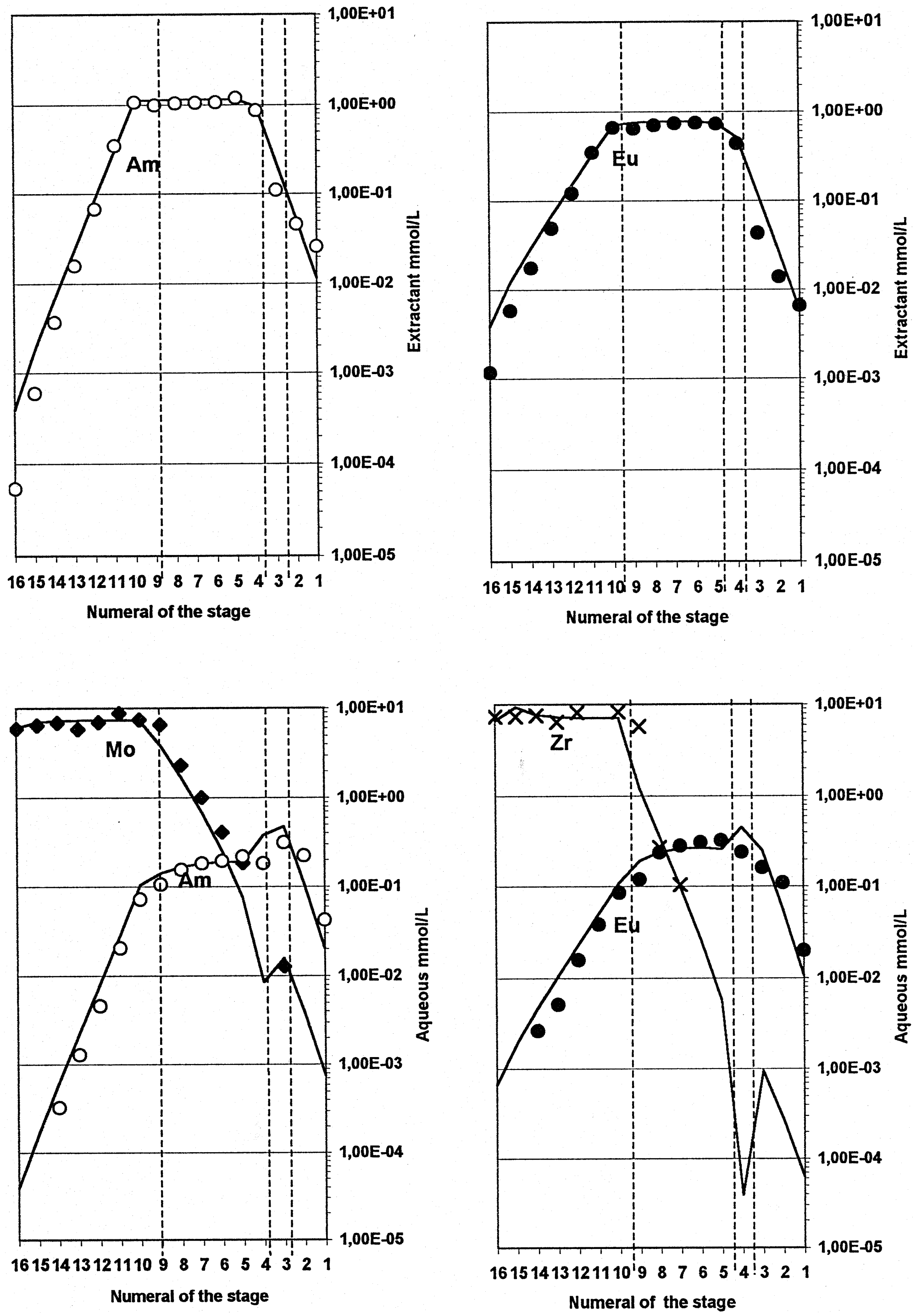

FIG. 4 Steady-state concentration profiles of Am(III) and Eu(III) in the solvent (upper graphs) and of Am(III), Eu(III), Mo(VI), and Zr(IV) in the aqueous solutions (lower graphs) in the 16-stage mixersettler battery.

TABLE 5

Product and Raffinate Concentrations (mmol/L) Evaluated and Measured (by ICP/MS) after 11 hours for the 2nd Test of the New DIAMEX Flow Sheet[a]

	Product		Raffinate	
Constituent	Evaluated concentration	Measured concentration	Evaluated concentration	Measured concentration
Zr	9.27E-4 (9.53E-4)	8.22E-4	6.71 (6.74)	7.88
Sm	1.36 (1.35)	1.19	3.01E-4 (2.36E-4)	3.21E-4
Nd	7.20 (7.18)	6.65	5.64E-4 (4.54E-4)	4.98E-4
Mo	1.44E-2 (1.57E-2)	8.72E-3	6.05 (6.07)	6.09
Ru	1.33E-3 (1.53E-3)	4.71E-2	3.91 (3.93)	3.03
Cs	1.76E-7 (1.63E-7)	<2.35E-5	4.69 (4.71)	4.73
Eu	2.52E-1 (2.52E-1)	2.37E-1[b]	8.49E-4 (6.63E-4)	1.87E-4
Gd	1.80E-1 (1.81E-1)	1.16 E-1	7.06E-4 (5.57E-4)	1.26E-3
La	2.25 (2.25)	2.27	2.13E-4 (1.75E-4)	1.94E-4
Rh	2.65E-5 (2.65E-5)	7.29E-5	7.74E-1 (7.77E-1)	6.58E-1
Pr	2.03 (2.02)	1.91	6.25E-5 (5.02E-5)	6.92E-5
Am	4.77E-1 (4.77E-1)	4.67E-1[b]	4.85E-5 (3.87E-5)	—
HNO_3	239 (246)	202[c]	3.31E3 (3.6E3)	3.41E3[c]
$H_2C_2O_4$	0.92 (0.92)		149 (149)	

[a] Data in parentheses correspond to values evaluated at steady-state; the flow rates were: solvent = 50 mL/h, feed = 100 mL/h; 1st scrub 25 mL/h; 2nd scrub = 25 mL/h; stripping solution = 100 mL/h. See also note in Table 3.

[b] Has been obtained by gamma spectrometry.

[c] Refers to total H^+ concentration.

Table 5 shows the product and raffinate compositions evaluated and measured after 11 hours under the 2nd test operating conditions.

As mentioned in the Operating Procedures and Analytical Methods section, concentrations were measured by gamma spectrometry for Am and Eu and by ICP/MS for the other elements. ICP/MS has a detection limit of about 2×10^{-5} mmol/L and an uncertainty range up to ±20%. Differences between calculated and measured values are therefore due to both the analytical methods and the difference of the element concentration from its steady-state value. This difference seems too large for Mo, and it is probably due to distribution ratios achieved in the battery run which are different (and more favorable) from those adopted as input of the computer program.

CONCLUSIONS

The process described in the paper by computer program calculations and test results proved able to obtain a separation factor of Am + lanthanides from a PUREX process raffinate higher than 10^3 with a total number of 16 stages at a feed/extractant flow-rate ratio of 2. A low Mo + Zr concentration in the

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product (about 1 wt% of that of Am) is achieved by appropriate oxalic scrubbing to prevent any oxalate precipitation. Compared with the first version of the DIAMEX process (which uses 0.5 mol/L C14 in TPH), the flow sheet proposed in this paper strongly reduces the risk of third-phase occurrence and minimizes the volume of involved solvent. Work is in progress to define a second step of the process for the separation of An from lanthanides.

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