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Liquid/Liquid-Mass Transfer at Swarms of Droplets: Kinetics of Mass Transfer in the System Uranylnitrate, Nitric Acid, Tributylphosphate

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ABSTRACT

Mass transfer of uranium and nitric acid between a water phase and a kerosine phase, loaded with 30 vol % tributylphosphate (T) was measured in an apparatus designed to produce swarms of droplets of equal size. The aim of this investigation was to produce a suitable kinetic model and to derive the values of the individual transport coefficients.

It is shown that the reactions are interfacial and controlled by transport processes. The model of the system is supported by the agreement between calculation and experiment and by the values of the individual transport coefficients obtained. Deviations between the model and experiment at high uranium concentrations are caused by interfacial instabilities.

INTRODUCTION

Liquid/liquid extractions are used in the reprocessing of nuclear fuels (PUREX-process). In particular, this involves the exchange of uranium, plutonium and nitric acid between water and kerosine loaded with T (30 vol%). In this investigation the liquid/liquid reactions involved are stoichiometrically defined by

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$$UO_2^{2+} + 2T + 2NO_3^- \rightleftharpoons Ko$$

$$H^+ + NO_3^- + T \rightleftharpoons Kx$$

corresponding to reliable distribution models (1,2).

Where columns are utilized for the liquid/liquid operations, the problem has been that, until now, the axial concentration profiles could not be calculated for such coextraction systems. This has meant that such columns have had to be designed empirically. There are two reasons for this unsatisfactory situation: axial backmixing (3,4) and kinetics of mass transfer (5). Whereas backmixing in pulsed columns has been investigated for more than 15 years (6,7,8,9), the introduction of kinetic models for the calculation of profiles has been restricted to an "overall model" (9).

The basic object of such kinetic approaches is to clarify the site of the chemical reactions and the rate determining steps. In this respect there exists a conflict, typical for reactions in liquid/liquid phases: there is no agreement in the literature over the method or the arguments suitable for distinguishing between chemical reactions or diffusion processes being rate determining.

The discussions relevant to the system investigated in this work are distributed among many publications (10 - 27, 33, 42). The reason for the contradiction in the available literature is that the arguments used are ambiguous.

Thus, for example: "Overall" activation energies up to ca. 40 kJ/mol are compatible for fast chemical reactions but also for transport processes. The comparison of the rates of mass transfer in both directions across a particular phase boundary or a comparison between different types of phase contact cannot, unequivocally be assumed by solely a comparison of chemical processes; it can also involve transport processes which may be rate determining. Finally arguments over reaction orders are indications for chemical steps only for these cases, where the calculated orders have been proved to be incompatible with possible transport processes.

In contrast, measurements in suitable stirred cells are suitable for distinguishing, unequivocally, between flow independent chemical and flow dependent diffusion steps. However, this is only the case when the applied type of the stirred cell has been demonstrated to be efficient: provided a transport limited system is characterized by a linear relationship between flux and stirring speed, then fluxes in other systems, which are independent of convection, can demonstrate that the chemical steps are rate determining. Only with stirred cells, which have been calibrated with characterized systems of known kinetic behaviour, can conclusions for unknown systems be just. Even for the most frequently used Lewis-type cell (11) indications of the efficiency have not been described. Thus, previous results with such cells are ambiguous.

With our measurements in a stirred cell, for which the kinetic efficiency had been proved, it was possible to show that reactions occuring during the mass transfer of uranium and nitric acid are interfacial and are controlled by transport processes (24, 25). The aim of this investigation was to check this model with regard to its efficiency for the mass transfer into, or out of, swarms of droplets in order to ascertain a kinetic base for the design of columns.

MEASUREMENTS AND APPARATUS

Mass transfer of uranium and nitric acid between water and dodecane loaded with T was measured in both directions, in a range of concentrations corresponding Table 1.

The dispersed phase was always that of the solvent. The essential features of the apparatus are its production of droplets with equal sizes and the continuous recyc-

TABLE 1

Concentration Fields of the Performed Measurements.

Extraction

Transfer-component	U_a mol/l	H_a mol/l	Kerosine 30 vol % T_{tot}
H	-	2,4	Dodecane
H	-	0,606-3,6	Hexane
U,H	$2{,}4\cdot10^{-4}$-0,52	2,4	Dodecane

Reextraction

Transfer-component	Ko_a mol/l	Kx_a mol/l	Kerosine 30 vol % T_{tot}
H	-	0,09-0,909	Dodecane
H	-	0,207-0,934	Hexane
U,H	$5{,}7\cdot10^{-4}$-0,281	0,210-0,35	Dodecane

ling of the solvent phase via a mixing vessel (26) (Fig. 1). The droplets are formed by capillaries and coalesce instantaneously on wettable Teflon-beads. By recycling the solvent phase it is possible to derive mass fluxes for a continuous range of different concentrations with only one experiment. Since the volume of the solvent phase in the vessel is always large enough to neglect the volume of the solvent phase inside the plate, the rotameter and the pipes, it is possible using:

$$c_E = c_K + \frac{dc_K}{dt} \cdot \frac{V_K}{\dot{V}_d} \qquad (1)$$

to calculate the concentrations inside the droplets at the top of the plate with measurements of the change in

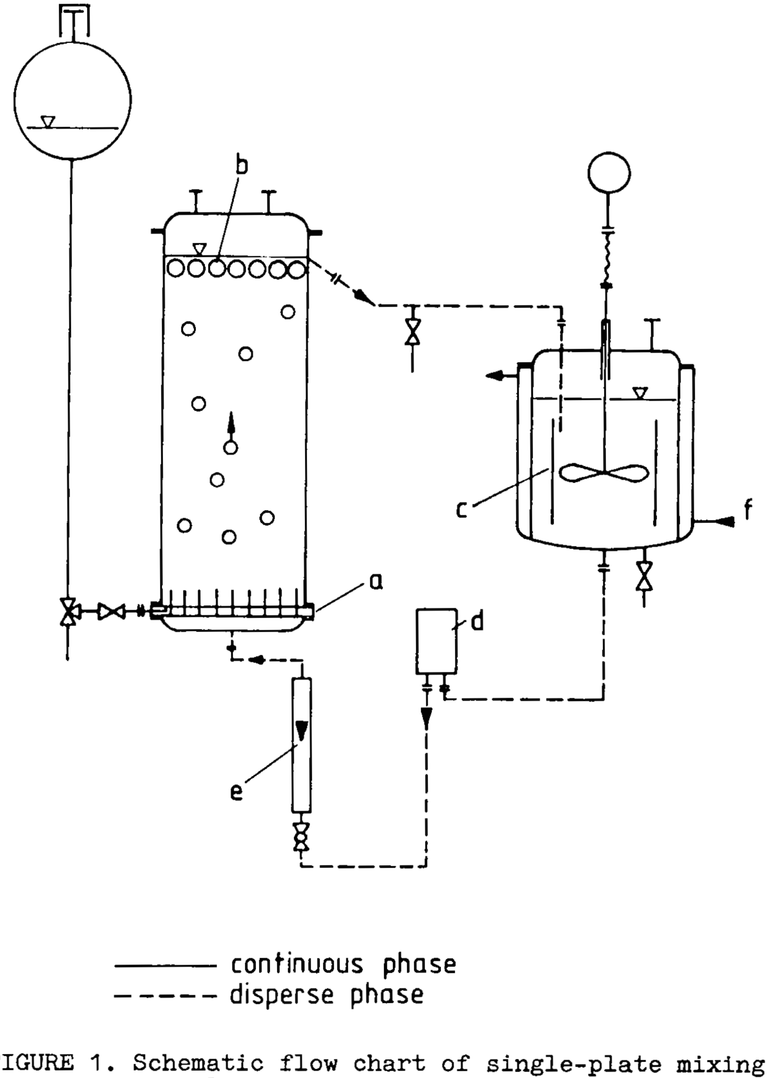

FIGURE 1. Schematic flow chart of single-plate mixing vessel layout.

concentration with time inside the vessel. The inlet concentration of the droplets corresponds to the concentration inside the well mixed vessel c_K. The agreement between the calculated and the measured concentrations c_E is satisfactory (27), which suggests that the arrangement single plate/mixing vessel with recycling of one or both phases should be of interest for liquid/liquid-mass transfer in general.

Two different plates were applied with heights of 9 and 20 cm. The throughput was varied between 0,42 - 12,0 cm^3/s always outside the range of the jets. Depending on throughput the range of the diameters of the droplets was between $2{,}1 \leqslant d_P \leqslant 2{,}5$ mm. The capillaries were arranged in a triangular fashion at the bottom plate equipped with 73 or 283 capillaries.

Temperatures were always kept constant at 20°C. The chemicals $UO_2(NO_3)_2 6H_2O$ and nitric acid were p.a. products of Merck Company. The applied highly purified water comes from a quartz bidestiller, Dodecane (for "synthesis", Merck Company) and Tributylphosphate (for "extraction analysis", Merck Company) were not further purified.

Nitric acid concentrations and uranium concentrations for $U \geqslant 5 \cdot 10^{-3}$ mol/l were analyzed with potentiometric titration (28). For $U < 5 \cdot 10^{-3}$ mol/l photometric analysis of the TAM-complex was applied (29).

MASS TRANSFER KINETICS

With measurements in a stirred cell one can infer that the reactions are interfacial with rates determined by the transport processes (24, 25) shown by the scheme in Figure 2. This model is supported by the following facts:

1) The relation between flux and stirring speed was always linear (25) comparable with the same behaviour in numerous reference systems in which transport processes are rate determining (30, 31).

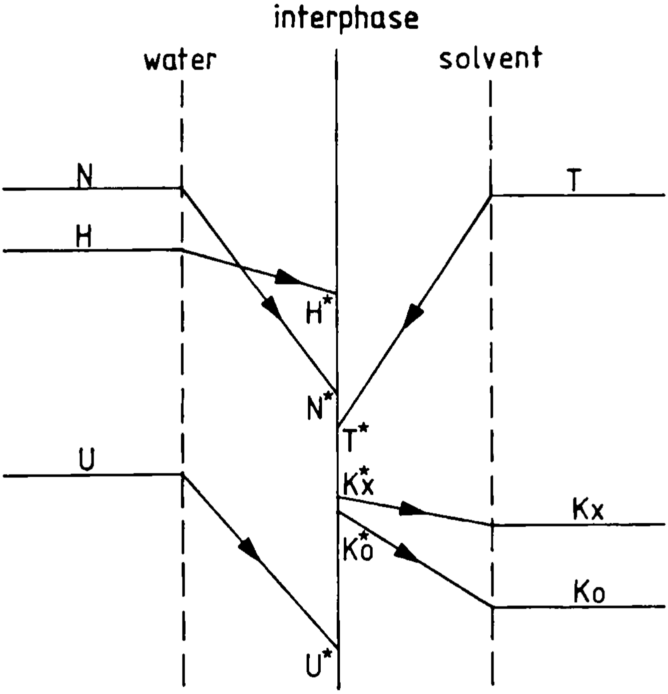

FIGURE 2. Schematic concentration profile for the components at the interphase.

2) The values of the individual coefficients, calculated on the basis of the model, are comparable with the corresponding coefficients for more simple systems in a stirred cell at equal Reynolds numbers (24).
3) With the individual transport coefficients and the proposed model it is possible to calculate $c_i = f(t)$ which agrees with the experimental results even when the nitric acid concentration overshoots the equilibrium value (24, 25).

We suppose that the mass transfer at droplets follows the same mechanism because in stirred cells there is no indication that the mass flux is independent of Re, the typical sign that chemical reactions are rate determining (32) whereas in chemically controlled systems one observes flow-independent rates in the stirred cell with the same specific flux at the droplets (32).

Equations.
Corresponding to the fast reactions at the interface, the interfacial concentrations c^* are connected by the equilibrium conditions

$$K_U = \frac{Ko^*}{U^* T^{*2} N^{*2}} \qquad (2)$$

$$K_H = \frac{Kx^*}{H^* N^* T^*} \qquad (3)$$

for both reactions. The individual fluxes are conveniently described by equations of the type

$$\dot{n}_i = \pm \beta_i \,(c_i - c_i^*) \qquad (4)$$

$(i = H, U, T, Ko, Kx)$. Because of the condition of electroneutrality the transport processes of the ions in the water phase are not independent (24, 26). We have arbitrarily selected the nitric flux as fixed and thus the corresponding transport equation need not be taken into consideration.

Corresponding to the stoichiometry of the two reactions the conditions for the individual fluxes are:
$\dot{n}_U = \dot{n}_{Ko}$, $\dot{n}_H = \dot{n}_{Kx}$, $\dot{n}_T = 2\dot{n}_U + \mathring{n}_H$.

Because the interfacial concentrations are unknown and unmeasurable quantities, the efficiency of the model can only be checked by numerical methods if the values of the individual transport coefficients are known.

INDIVIDUAL TRANSPORT COEFFICIENTS.

It has been shown (24, 33) that the individual coefficient for any component i is accessible provided the interfacial concentration c_i^* relative to the concentration of the same component in the bulk phase is negligible ("ß-condition"). In such cases the corresponding transport equation reduces to $\mathring{n} = \beta \cdot c$. Thus, with the known values of the bulk concentration and the measured flux the coefficient ß is obtainable.

The ß-condition is fulfilled for β_U in the examined range $2 \cdot 10^{-4} \leq U_a \leq 5 \cdot 10^{-1}$ mol/l für the extraction direction with 30 vol % T (25). For β_{Kx} and β_{Ko} the condition $Kx^* \ll Kx$ or $Ko^* \ll Ko$ demands reextraction in an unloaded water phase (25). This is predicted by the numerical calculations based on the kinetic model and performed with reasonable variations of individual transport coefficients. For such bulk concentrations, where $c^* \ll c$ is fulfilled, for any component in the whole range of reasonable variations of β_i, the ß-condition is considered to be realistic.

Evaluation

A schematic development of the concentration gradients during the contact time of droplets is shown in Figure 3. It can be seen that the realization of $c^* \ll c$ demands the achievement of initial rates which should be accessible from extensive measurements for different contact times and using a procedure similar to that used for the single droplet system. However, with the assumption that the individual transport coefficients are independent of concentration it is possible to calculate initial rates from measurements at only one plate; i.e., with only one contact time:

Numerical calculations with the equations 2 - 4 show that the interfacial concentrations are always linearly related to the bulk concentration (25, 26). The direction of extraction is given by:

$$c_i = c_i \left(1 - \frac{c^*_{i,a}}{c_{i,eq}}\right) + c^*_{i,a} \qquad (5)$$

The initial concentration $c^*_{i,a}$ depends on all the individual coefficients (24, 25, 26) and is therefore, in most

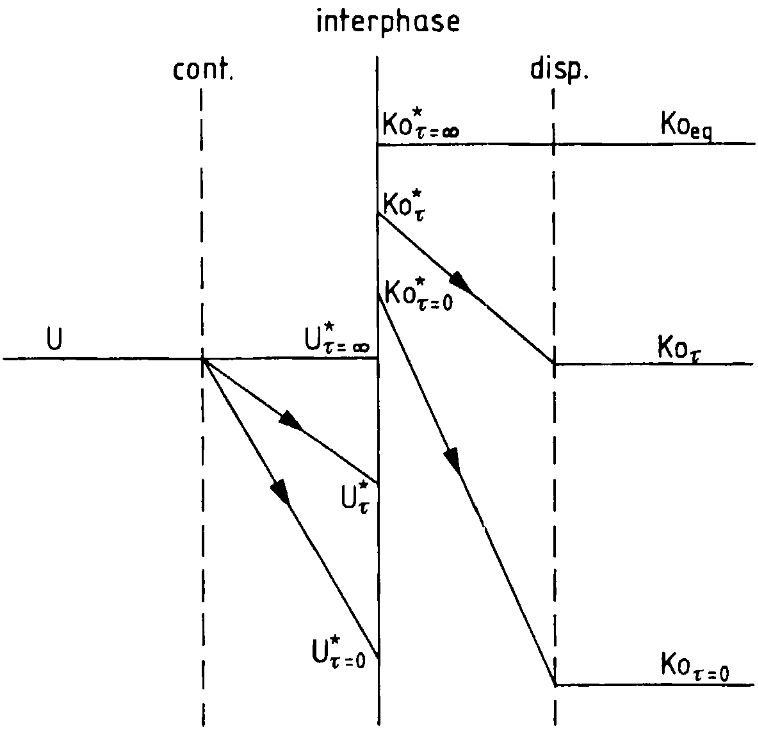

FIGURE 3. Development of the concentration profile during the droplet rise (sketch only).

cases, undefined. $c_{i,eq}$, the equilibrium concentration inside the droplets is only established after a longer contact time with the continuous phase and is determined by the respective concentrations in that phase (26).

Equation 5, combined with the selected transport equation,

$$\dot{n}_{Ko} = \beta_{Ko}(Ko^* - Ko) \qquad (6)$$

gives

$$\dot{n}_{Ko} = -\beta_{Ko} \frac{Ko^*_a}{Ko_{eq}} (Ko_{eq} - Ko) \qquad (7)$$

which can be integrated (10,11,12):

$$\tau F_s \beta_{Ko} \frac{Ko^*_a}{Ko_{eq}} = - \ln \left(\frac{Ko_{eq} - Ko_E}{Ko_{eq} - Ko_K}\right) \qquad (8)$$

With equations 1, 7 and 8 an expression for the rate $\dot{n}$,

$$\dot{n}_{Ko} = - \frac{Ko_{eq}}{F_s \tau} \left(1 - \frac{Ko}{Ko_{eq}}\right) \ln \left(1 - \frac{\dot{V}_K}{\dot{V}_d} \cdot \frac{dKo_K / dt}{Ko_{eq} - Ko_K}\right) \qquad (9)$$

can be obtained. Thus, for $Ko_K = Ko = 0$ the equation for the initial rate is given by

$$\dot{n}_{Ko_a} = - \frac{Ko_{eq_a}}{F_s \tau} \ln \left[1 - \left(\frac{dKo_K}{dt}\right)_{t=o} \cdot \frac{\dot{V}_K}{\dot{V}_d Ko_{eq_a}} \right] \qquad (10)$$

Because $\dot{n}_{Ko,a} = \dot{n}_{U,a}$ and $\overset{\circ}{n}_U = \beta_U(U-U^*)$ for $U_a^* \ll U_a$ it is possible to derive the coefficient β_U. The coefficients β_{Kx} and β_{Ko} could be obtained in a similar way using the measurements from reextractions (26).

On a plot of the values of β_i against the reciprocal throughput a linear extrapolation leads to an intercept on the β-axis corresponding to the "real" β-coefficient which is not influenced by end effects (34). The coefficients obtained are listed in Table 2.

DISCUSSION OF THE β-VALUES.

For $U_a \leq 10^{-2}$ mol/l the coefficients obtained are independent of the concentration at the plate. However, with increasing concentration of uranium, interfacial instabilities give rise to increasing values of β with U_a and β_U becomes dependent on the concentration U_a and the contact time of the droplets.

Because nitric acid transfer itself produces instabilities, the β_U-coefficients for $U_a \leq 10^{-2}$ mol/l have to be normalized with respect to the concentration of nitric acid. Such a correction is possible without any assump-

TABLE 2

Mean Value of the Mass Transfer Coefficient β_U at Various Uranium Concentrations.

U mol/l	h cm	β_U cm/s
$2{,}4 \cdot 10^{-4}$	20	$1{,}35 \cdot 10^{-2}$
$2{,}0 \cdot 10^{-3}$	20	$1{,}35 \cdot 10^{-2}$
$2{,}0 \cdot 10^{-3}$	9	$1{,}35 \cdot 10^{-2}$
0,020	20	$1{,}35 \cdot 10^{-2}$
0,10	20	$1{,}60 \cdot 10^{-2}$
0,10	9	$1{,}60 \cdot 10^{-2}$
0,50	20	$1{,}60 \cdot 10^{-2}$
0,50	9	$2{,}30 \cdot 10^{-2}$

The accuracy of the mass transfer coefficients amounts to approx. ± 10% at small uranium concentrations and up to ± 20% at U = 0,5 mol/l and h = 9 cm.

tion being made (26). The resulting standard value for β_U with $T_a = 1{,}09$ mol/l and $\beta_U = 1{,}10 \cdot 10^{-2}$ cm/s indicates (see Table 2) that the effect of the nitric acid instability on the uranium transfer is small.

With dodecane as a solvent phase the values of β_{Kx} and β_{Ko} are independent of the corresponding concentrations (26) (see Figure 4) i.e., the corresponding mass transfer in the direction of reextraction the interface is stable.

For β_H and β_T ß-conditions could not be realized. Therefore, we have used the estimated values listed in Table 3.

These estimations are not crucial since it can be shown that the mass transfer is insensitive to these coefficients (24, 25, 26). The so-called standard values for

TABLE 3

"Standard Values" of the Individual Mass Transfer Coefficients.

	Continuous Phase		Disperse Phase		
	β_U	$\beta_H{}^*$	$\beta_T{}^*$	β_{Ko}	β_{Kx}
Mass Transfer Coefficient cm/s $\cdot$ 10^2	1,1	2,12	1,09	0,35	1,05

*β_H computed from β_U (both continuous phase) and β_T from β_{Kx} (both disperse phase) with $\beta_1/\beta_2 = \sqrt{D_1/D_2}$.

the coefficients, needed for the following calculations, are also listed in Table 3.

Correlations.

The above values of the individual transport coefficients can be compared with the corresponding values calculated from known correlations for the mass transfer at single droplets. With the properties of the contacted phases and the rising velocity of the droplets one can conclude that the droplets in our system are in a transition region between circulation and oscillation (35). Selected correlations for this state of flow have been used to calculate β-values which are in a reasonable agreement with our experimental values (26) (see Table 4). This corroborates the kinetic model and the concept of the β-condition. By applying the model and the suitable correlations it should be possible to estimate transport coefficients for other droplet diameters.

TABLE 4

Comparison of Mass Transfer Coefficients Calculated from Theory with Experimental Values (Continuous Phase).

Mass transfer component	U	Kx
Continuous phase	H_2O	Dodekane, 30 Vol % T
Column height cm	9; 20	18,5
$ß_{exp} \cdot 10^2$ cm/s	1,1	1,1*

Correlation	$ß_{exp}/ß_{theo}$	$ß_{exp}/ß_{theo}$
Yamaguchi,Katayama (36) Circulating droplets	0,95	1,25
Abachi (37) Circulating droplets	0,83	1,00
Higbie (38) Circulating droplets	0,55	0,76
Boussinesq (39) Circulating droplets	0,55	0,76
Yamaguchi et al (40) Oscillating droplets	0,63	0,92

*Value of Schweyer (42)

EFFICIENCY OF THE MODEL.

In this context the decisive question is whether the ß-coefficients, always related to initial states, are suitable to describe the mass transfer for various concentrations and both directions of fluxes. To answer this question numerical calculations were performed using the coefficients listed in Table 3. The numerical procedure was quasi-stationary i.e., the model equations were used to calculate the increase of the concentration inside the droplet, beginning with any concentration c_K at the inlet into the plate ($\tau = 0$) and successively increasing τ by small steps of up to 0,1 s until the contact time τ was reached (26).

The calculations with β_i (Table 3) $f_1\beta_i$ or $f_2\beta_i$ (f_1, f_2 are variable coefficients which were used to examine the sensitivity of the overall results to β_i) can be compared with the corresponding experimental values for c_E with the following characteristic results:

1) For the reextraction of Kx and Ko good agreement is obtained between calculation and experiment (for example see Figure 4).
2) For the extraction of nitric acid alone and for the coextraction of uranium and nitric acid agreement between calculation and experiment is restricted to the second part of the conversion which starts at a time of approximately 400 s for both components Ko and Kx (see Figure 5). In the first part of conversion, when the system is far from equilibrium, the measured concentrations are greater than the calculated values. At $t = 0$ the experimental value corresponds, approximately, to a calculated value using $2\beta_i$.

These are representative examples for a broad series of relevant measurements.

The reason for the observed deviations between calculation and experiment is the interfacial instability produced by mass transfer of nitric acid and uranium. It is known that such instabilities disappear as the system approaches equilibrium (41). Thus, in the so-called second part of the conversion the influence of instability seems to be negligible. For high uranium concentrations the instabilities produced can be identified with a marked "kick"-behaviour of droplets, suspended at a capillary (41). The instability caused by the pure nitric

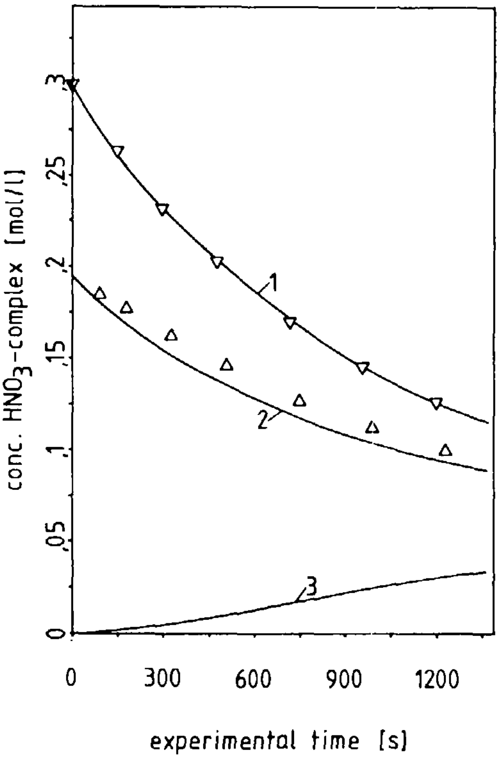

FIGURE 4a. Reextraction of nitric acid:
1: Mixing vessel concentration
2: Calculated droplet concentration c_E
△ Measured droplet concentration c_E
3: Calculated equilibrium concentration of the dispersed phase.

U_a=0 mol/l; U_∞=1,90·10^{-4} mol/l; H_a=0 mol/l; H_∞=0,37 mol/l; K_U=15,7 $(mol/l)^{-4}$; K_H=0,40 $(mol/l)^{-2}$; h=20cm; τ =1,70 s; d_P=0,267 cm;

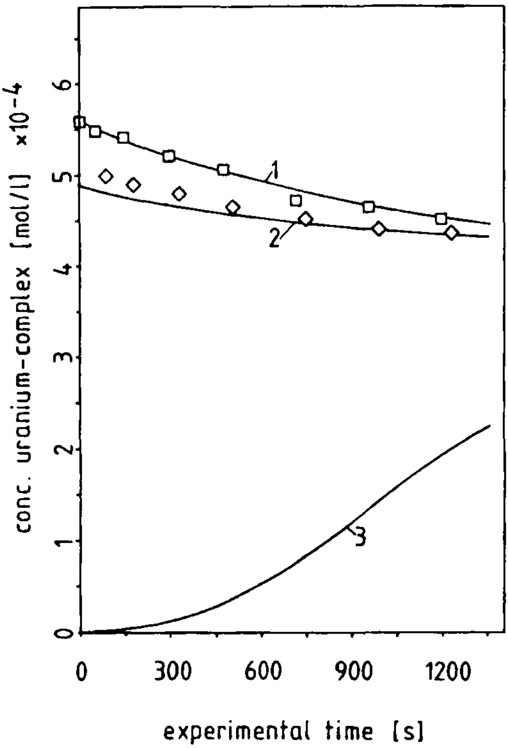

FIGURE 4b. Reextraction of uranium: conditions analogue FIGURE 4a.
◇ Measured droplet concentration c_E.

acid transfer is comparably small as can be seen from the acceleration of a simultaneous benzene transfer. This interpretation of the deviations is supported by the fact that they become small if the contacted solvent phase is loaded with Ko at the start of the experiment (Figure 6).

The most striking indication for the usefulness of the proposed model is the prediction of the overshooting nitric acid concentration. In accordance with the measurements at droplets (10, 26) and in the stirred cell (24) this effect is limited to high uranium concentrations in the water phase. For swarms of droplets this effect

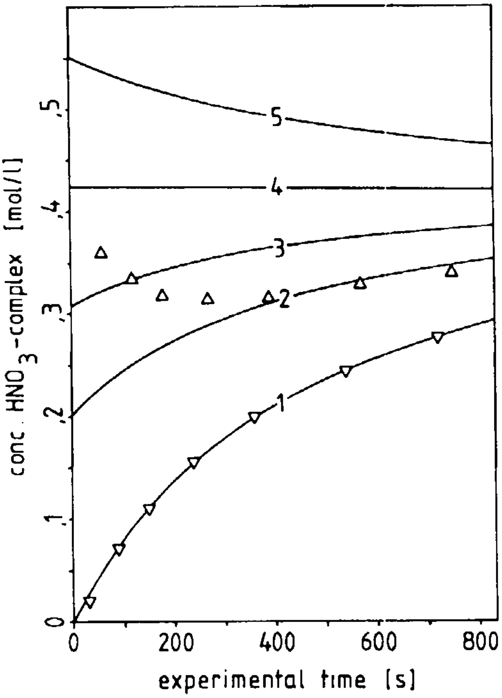

FIGURE 5a. Extraction of nitric acid:

1: Mixing vessel concentration

2: Calculated droplet concentration c_E

△ Measured droplet concentration c_E

3: Calculated droplet concentration c_E, $f_1=1,75$

4: Calculated droplet concentration c_E, $f_2=3,00$

5: Calculated equilibrium concentration; for the uranium complex (FIGURE 5b) the equilibrium concentrations lie well out of the area shown in the figure.

$U_a=2,00\cdot10^{-3}$ mol/l; $U_\infty=6,37\cdot10^{-5}$ mol/l; $H_a=2,48$mol/l;$H_\infty=1,81$mol/l; $K_U=12,2(\text{mol/l})^{-4}$; $K_H=0,190$ $(\text{mol/l})^{-2}$; h=20 cm; $\tau=1,70$ s; $d_P=0,243$ cm;

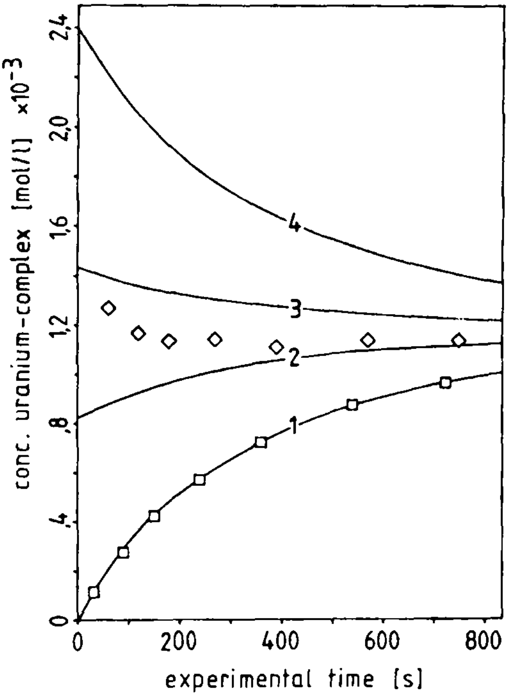

FIGURE 5b. Extraction of uranium: conditions analogue FIGURE 5a.
◇ Measured droplet concentration c_E.

is shown in Figure 7: the calculated and the measured curve for c_E shows mass transfer of nitric acid into the solvent phase although c_K is larger than c_{eq}.

The above kinetic model should be suitable for calculating concentration profiles for columns. The use of such a model to calculate the concentration profiles for plutonium in a PUREX-medium by including the coupled plutonium-flux would be considerably more effective than the alternative, empirical, procedure of determining the axial concentration of plutonium. Furthermore, we are convinced that the development of column kinetics is essential if the mass transfer system is of the coextraction type described here.

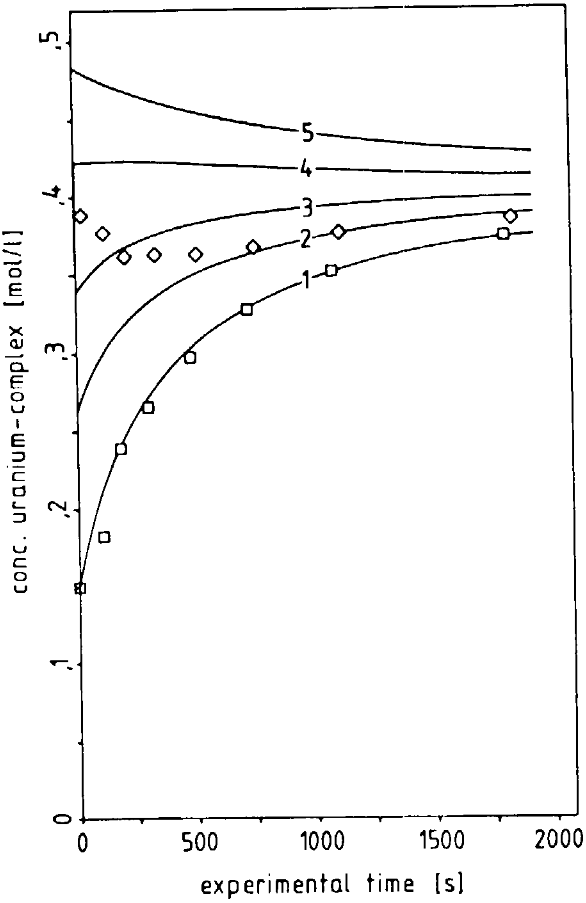

FIGURE 6. Extraction of uranium and nitric acid, experiment with precharged organic phase, only the concentration curve of the uranium complex is plotted.

1: Mixing vessel concentration.

2: Calculated droplet concentration Ko_E

◇ measured droplet concentration Ko_E

3: Calculated droplet concentration Ko_E, $f_1 = 2{,}00$

4: Calculated droplet concentration Ko_E, $f_2 = 4{,}00$

U_a=0,516 mol/l; U_∞ = 0,111 mol/l;

H_a=2,40 mol/l; H_∞ =2,69 mol/l; Ko_a=0,149 mol/l;

Ko_∞ =0,405 mol/l; Kx_a=0,314 mol/l;

Kx_∞ =0,148 mol/l; K_U= 24,7 $(mol/l)^{-4}$;

K_H=0,143 $(mol/l)^{-2}$; h=21 cm; τ =1,84 s;

d_P=0,245 cm.

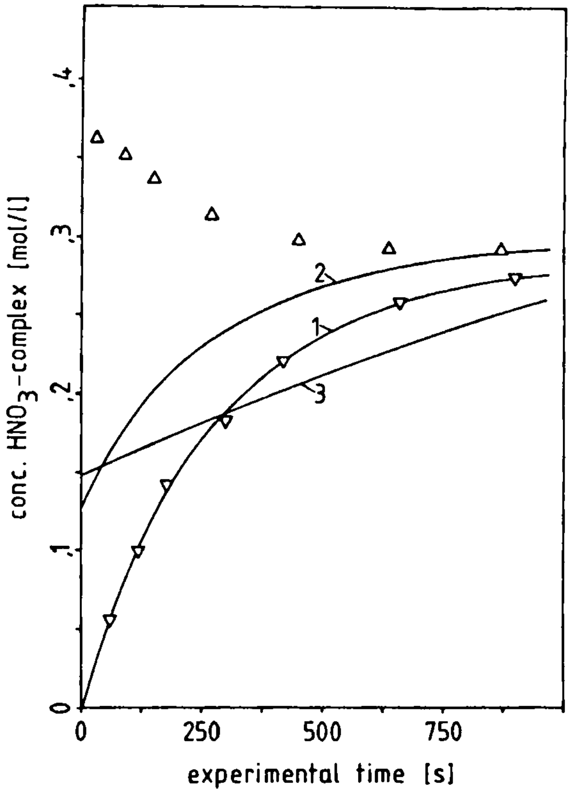

FIGURE 7. Extraction of uranium and nitric acid; only the concentration curve of the nitric acid complex is plotted.

1: Mixing vessel concentration

2: Calculated droplet concentration Kx_E

△ Measured droplet concentration Kx_E

3: Calculated equilibrium concentration

U_a=0,262 mol/l; U_∞=0,0146 mol/l;H_a=2,38 mol/l; H_∞=1,97 mol/l; Ko_a=0 mol/l; Ko_∞ =0,149 mol/l; K_U = 11,2 $(mol/l)^{-4}$; K_H = 0,167 $(mol/l)^{-2}$ h = 20 cm; τ = 1,91 s; d_P = 0,230 cm.

SYMBOLS

Symbol	Dimension	Explanation
c	mol/l	Concentration
D	cm	Diameter
d_P	cm	Droplet diameter
$F_s = 6/d_P$	1/cm	Specific interfacial area
h	cm	Column height
H	mol/l	Nitric acid concentration, water phase
K_H	$(mol/l)^{-2}$	Equilibrium constant of the nitric acid complexation
K_U	$(mol/l)^{-4}$	Equilibrium constant of the uranium complexation
Ko	mol/l	$UO_2(NO_3)\cdot 2T$-concentration, solvent phase
Kx	mol/l	$HNO_3\cdot T$-concentration, solvent phase
$\dot{n}$	mmol/s cm^2	Specific molar flux
N	mol/l	NO_3^--concentration, water phase
Re	-	Particle Reynolds number
t	s	Experimental time
T	mol/l	Tributyl phosphate concentration, solvent phase
TAM		(2-(2-Thiazolylazo)-5-dimethylaminophenol
U	mol/l	Uranium concentration, water phase
V_K	cm^3	Volume of the mixing vessel
$\dot{V}_d$	cm^3/s	Flow rate of the dispersed phase.

Greek Symbols

Symbol	Dimension	Explanation
β	cm/s	Individual mass transfer coefficient
τ	s	Droplet contact time

Indices

a	Start
E	Single plate outlet
eq	Equilibrium
i	i-component
K	Mixing vessel
∞	Final equilibrium of an experiment
tot	Total

Superscripts

*	Interphase

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