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Sorption behaviour of well-defined oxidation states

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OXIDATION STATES

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SUMMARY

The sorption of the actinides Am(III), Th(IV), Np(V), Pa(V), U(VI) and Pu has been studied as a function of pH (2-12) for two nuclide concentrations (10^{-7}-10^{-9} M) (only one for Pa and U) in the systems Al_2O_3 - 0.01 M $NaClO_4$ and SiO_2 - 0.01 M $NaClO_4$. Distribution coefficients have been determined by a batch technique after various contact times (6h - 6w) at constant temperature (25 °C) in systems equilibrated with air. The observed sorption behaviour indicates a predominantly physical adsorption mechanism, where pH of the aqueous phase is the principal chemical parameter of influence. The sorption is highly related to the degree of hydrolysis, with a maximum in the pH-region where neutral species dominate and with a reduction of the sorption under conditions when anionic species (hydroxides or carbonates) would exist in solution. This is particularly the case for U(VI) at pH above 7-8 when anionic carbonate complexes would be formed. Plutonium is predominantly tetravalent under the present conditions, as indicated by the sorption behaviour.

CONTENTS

1.   INTRODUCTION

The interaction between actinides in environmental waters and exposed
solid materials is largely dependent on the chemical state of the
element in question (1). Of particular importance are

o   redox conditions; various oxidation states are possible in the
     environmental pH-range: Th(IV), Pa(V), U(IV)-U(V)-U(VI), Np(IV)-
     Np(V), Pu(III)-Pu(IV)-Pu(V), Am(III), Cm(III) (2),

o   complexation, especially with hydroxide (hydrolysis) and carbonat

Much of the sometimes contradictory experimental observations concern-
ing the sorption and distribution of actinides in geologic systems can
probably be related to poor characterization and control of important
chemical parameters such as

o   pH,

o   Eh (redox potential),

o   concentration of complexing anions (including carbonate from the
     water-air exchange),

o   presence of solid agents with non-characterized chemisorbing
     properties or considerable exchange capacities (pH-dependent).

The purpose of the present study is to get information on the sorption
behaviour of actinides under well-defined conditions, using actinides
in single oxidation states (Am(III), Th(IV), Np(V), U(VI)) as referenc
systems. Hereby it is possible to illustrate the importance of charac-
terizing the redox conditions of e.g. a potential geologic repository
site, as well as the effect of changes in Eh or pH on the retention of
specific actinides. Some preliminar results from this study have pre-
viously been presented (3).

2.   ACTINIDE REDOX CHEMISTRY AND HYDROLYSIS

2.1.   Oxidation states

The actinides (here denoted by An) exhibit multiple oxidation states
(from 3 to 7) in the early part of the series (Th, Pa, U, Np, Pu, Am),

but beyond plutonium (Am, Cm, etc.) the lower oxidation states are predominant (2,4).

Thorium exists only as Th(IV) in aqueous solutions.

For protactinium the most stable oxidation state is Pa(V) in the presence of air, although Pa(IV) can exist in aqueous solutions under highly reducing conditions.

For uranium, U(VI) will dominate entirely in aerated solutions, although both U(IV), and possibly also U(V) would exist under reducing conditions. The lowest oxidation state, U(III), is slowly oxidized by water with the evolution of hydrogen.

For neptunium and plutonium all the oxidation states III, IV, V and VI can exist in aqueous solutions, although Np(III) is easily oxidized. Under oxic conditions Np(V) would dominate entirely, while both Pu(IV) and Pu(V) could exist, as well as Pu(III) (at low pH). The distribution between Pu(IV) and Pu(V) would largely be affected by the carbonate concentration.

Americium is most stable as Am(III), although the easily reduced Am(VI) and Am(V) also can exist in aqueous systems, as well as Am(IV) in the presence of high concentrations of complexing agents. Only Am(III) would be expected in aerated waters in the absence of other oxidants than oxygen from the air.

Curium would largely exist as Cm(III).

Thus, in aqueous solutions equilibrated with air ($Eh \geq 0.8-0.06$ pH) and in the pH-range of possible environmental interest (3-4 to 11-12) the following oxidation states would be expected to dominate entirely (1,4-6):

    An(III):Am
    An(IV) :Th
    An(V) :Np,Pa
    An(VI) :U

For plutonium, at least in the intermediate pH-range 5-6 to 8-9 in the absence of any carbonate other than from the carbon dioxide in the

air, the expected dominating oxidation state would be Pu(IV), possibly with some minor contribution of Pu(V) at high pH.

2.2. Hydrolysis and speciation

The actinides are generally forming strong complexes with oxygen containing ligands, and the aqueous chemistry is dominated by hydrolysis reactions in the environmental pH-range, as well as by carbonate complexation for species in the penta- and hexavalent state, Table 1.

Table 1 Hydrolysis and carbonate complex formation constants ($\log K_1$) for the actinides (An) (25°C, I = 0) (2,6).

|		$\log K_1$	
		L = OH$^-$	L = CO$_3^{2-}$
An(III)L	Pu	a	a
	Am	6.5	6
An(IV)L	Th	10.8	
	Pa	14.8[c]	
	U	13.4	
	Np	12.5	
	Pu	13.5	
An(V)O$_2$L	Pa	9.5[c]	
	U	(4)	(5)
	Np	5.1	5.9
	Pu	4.3	b
An(VI)O$_2$L	U	8.2	10.1
	Np	8.9	b
	Pu	8.4	(9)

[a] Same as for Am assumed

[b] Same as for U assumed

[c] Ref. 7

The elements exhibit similar behaviour when they are in the same oxida
tion state. The differences in complex formation constants for in-
organic complexes of environmental interest between the actinides
uranium, neptunium, plutonium and americium in the same oxidation
state are, for the higher complexes, often within the uncertainty of
the data (2). Thorium forms significantly weaker hydroxide complexes
than the other tetravalent actinides (size effect). Protactinium dif-
fers from the other actinides both in the tetra- and pentavalent state;
Pa(V) is more similar to An(IV) than to An(V), in terms of hydrolysis
behaviour.

The actinide speciation in aerated solutions are given in Fig. 1.
Evidently hydrolysis would become significant at pH above 6 for americ-
ium(III), pH 1 for thorium(IV), pH 7-8 for neptunium(V) and pH 4-5 for
uranium(VI). Carbonate complexes would dominate in aerated solutions
at pH above 8 for americium(III), pH 8 for neptunium(V) and pH 5-6 for
uranium(VI). The speciation in aqueous systems with variation of Eh,
pH and $P(CO_2)$ is discussed elsewhere (2,6).
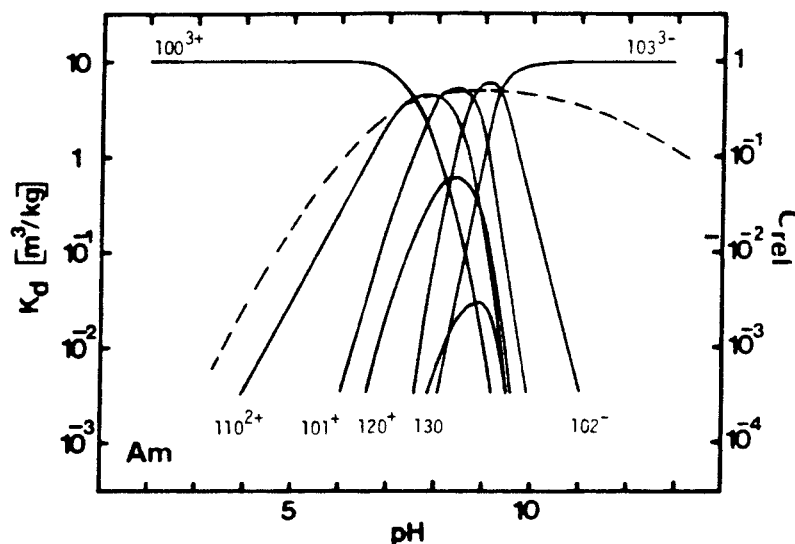
Figure 1 Speciation and distribution coefficients for americium(III),
thorium(IV), neptunium(V) and uranium(VI). Distribution data
as in Fig. 2-5 (Al_2O_3; Initial concentration: SII (SI for
U)(cf. Table 2); 0.01 M $NaClO_4$).Relative concentration (C_{rel})
calculated from data in ref. 2.(Each concentration curve
identified by xyz^n, corresponding to $An_x(OH)_y(CO_3)_z^n$).
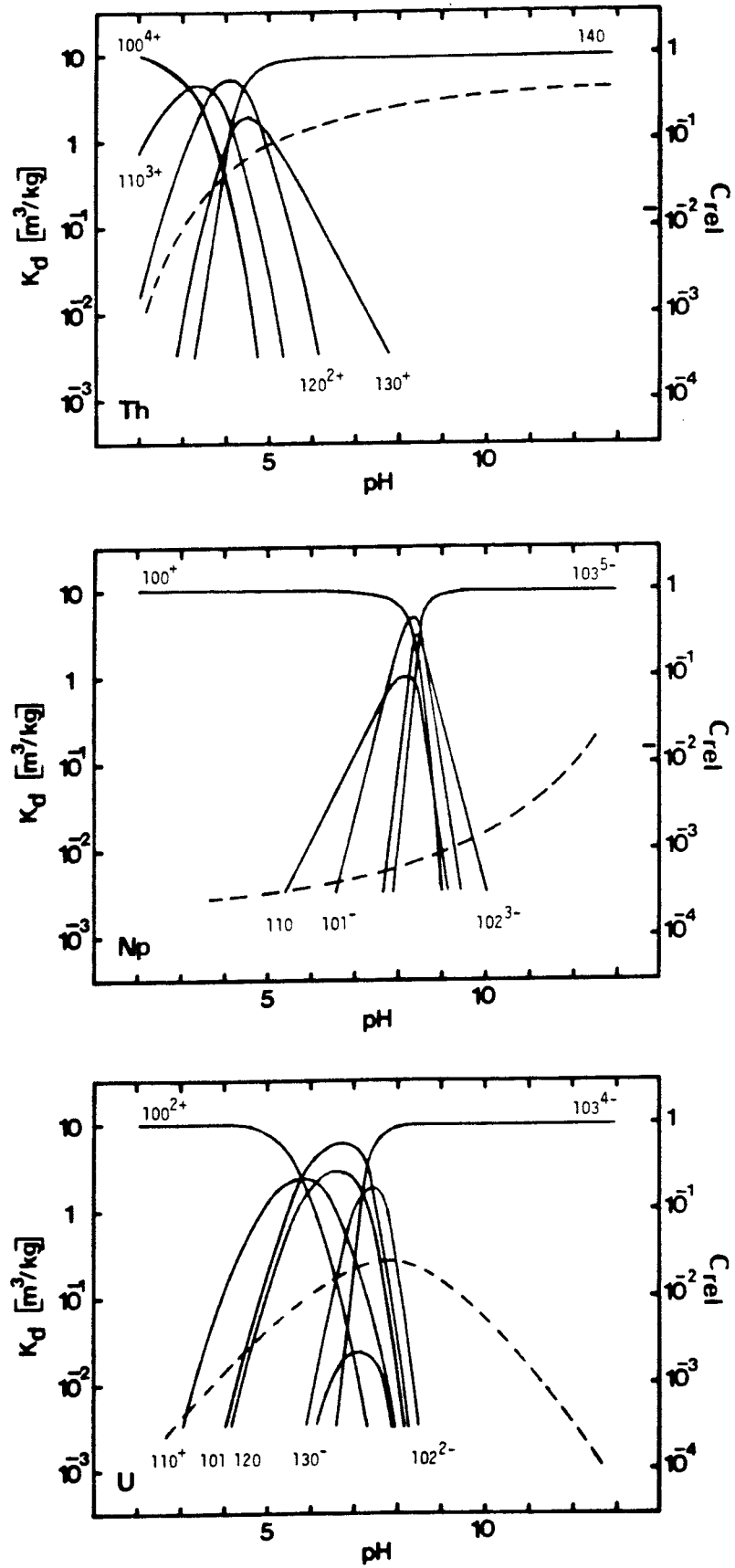
Figure 1 Speciation and distribution coefficients for americium(III),
Thorium(IV), neptunium(V) and uranium(VI)(Cont.)

3.   EXPERIMENTAL

3.1.   Radionuclides

Radionuclides according to Table 2 where used in this study. The sorption studies were performed at two different nuclide concentrations for those of the elements where long-lived as well as short-lived isotopes were available. All nuclides were stored in stock solutions of 1-2 M HCl.

Table 2   Radionuclides used in the distribution experiments.

Nuclide	Initial concentration[a]	
	SI, Mx10^7	SII, Mx10^9
^{232}Th+^{234}Th	3.0	2.5
^{233}Pa	-	0.004
^{233}U	2.1	-
^{235}Np+^{237}Np	1.9	1.9
^{237}Pu+^{239}Pu	0.6	0.6
^{241}Am	2.9	2.3

[a]Concentration in the solution at the start; the concentration in the stock solutions were about 10^{-5} M, except for ^{233}Pa.

The short-lived isotopes ^{235}Np and ^{237}Pu were obtained from Harwell, UK, and ^{234}Th and ^{233}Pa recovered from ^{238}U and ^{237}Np, respectively, by the use of a sorption procedure (8).

The concentrations of ^{233}U were measured by liquid scintillation technique (alpha-activity), while all the other nuclide concentrations were determined from measurements of the gamma-activity.

3.2.   Distribution measurements

The distribution coefficient (K_d, mol/kg solid per mol/m^3 solution) was measured by a batch technique (9,10) under the conditions given in Table 3. The thoroughly sieved and washed solid sorbent and the

aqueous phase were contacted, and the active stock solution (typically 0.1 ml per batch of 20 ml) was added. The distribution coefficients were, after an initial pH-adjustment (with NaOH), determined as a function of contact time. No further pH-adjustments were made. Totally 8-12 experiments at various pH but otherwise identical conditions were run simultaneously for each system. The solid and aqueous phases were separated by centrifugation (27000 g for 0.5 h) and samples from the water phases were taken. The distribution coefficients were calculated from

$$K_d = [(C_o - C)/m]/(C/V) \qquad (1)$$

where C_o = initial element concentration in solution (mol/l)

C = element concentration after certain contact time (mol/l)

m = mass of sorbent (kg)

V = volume of the water phase (m^3)

No corrections were made for sorption on the vessel walls (made of polypropylene). This should not be required, when considering that the surface areas of the exposed fine-grained solids were several orders of magnitude larger than the surface area of the vessel.

All experiments were performed in aerated systems.

Table 3 Conditions for the distribution measurements.

Solid sorbents:	Cryst. Al_2O_3 and SiO_2; grain size 0.090-0.125 mm
Solutions:	0.1 and 0.01 M $NaClO_4$; variation of pH (2-12)
Radionuclides:	See Table 2
Experimental conditions:	Temperature: 25 ± 1 $^{\circ}$C
	Solid/liquid: 0.20 g/20.0 ml
	Contact time: 6h, 1d, 1w, 4 or 6w

4. RESULTS AND DISCUSSIONS

The measured distribution coefficients (K_d) vs pH for Am(III), Th(IV), Np(V), U(VI), Pa(V) and Pu are given in Fig. 2-7. The curves correspond roughly to a contact time of one week.

Generally, the K_d vs pH-curves exhibit three separate stages:

o   increasing K_d with increasing pH; from very low values in the low pH-region to a high level within a fairly narrow pH-range, e.g. from 0.005 to 1.9 (corresponding to 5% and 95% sorption, respectively) within three pH-units for Am(III), Th(IV) and Pu;

o   high and fairly constant K_d in the intermediate pH-region, particularly for Am(III), Th(IV), Pa(V) and Pu;

o   slightly decreasing K_d (Am(III), Th(IV), Pa(V), Pu) or a more pronounced decreasing K_d (U(VI)) with increasing pH in the high pH-region

Only the K_d-increasing stage is observed for Np(V) within the pH-range of the present study (pH<12).
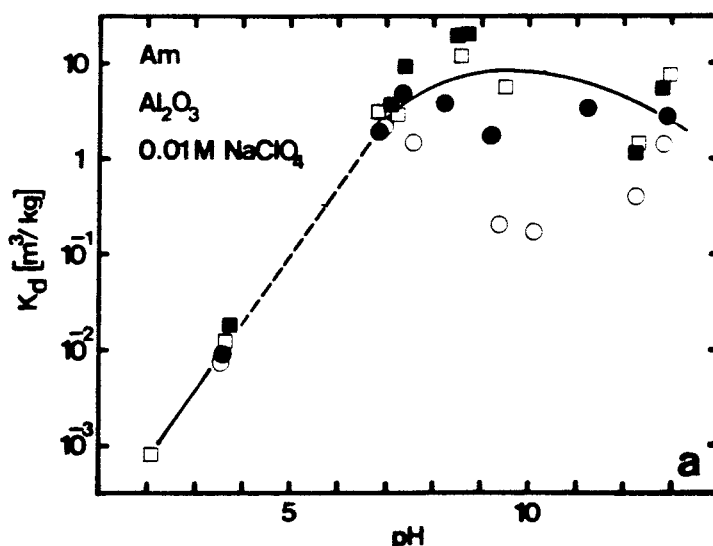
Figure 2 Distribution coefficients for americium(III).

O6h,   ●27h,   □1w,   ■6w

a. SI, b.-d. SII.
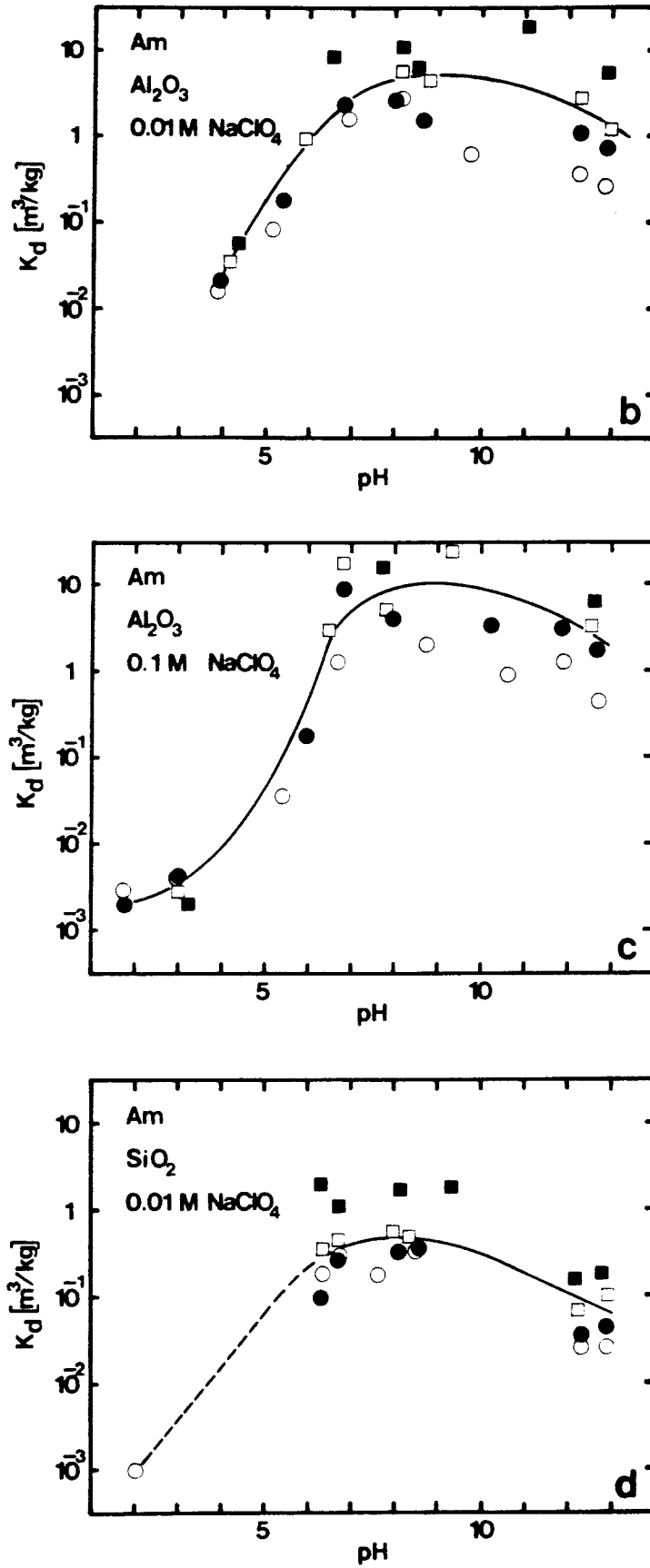
Figure 2 Distribution coefficients for americium(III)(Cont.).

○6h, ●27h, □1w, ■6w.
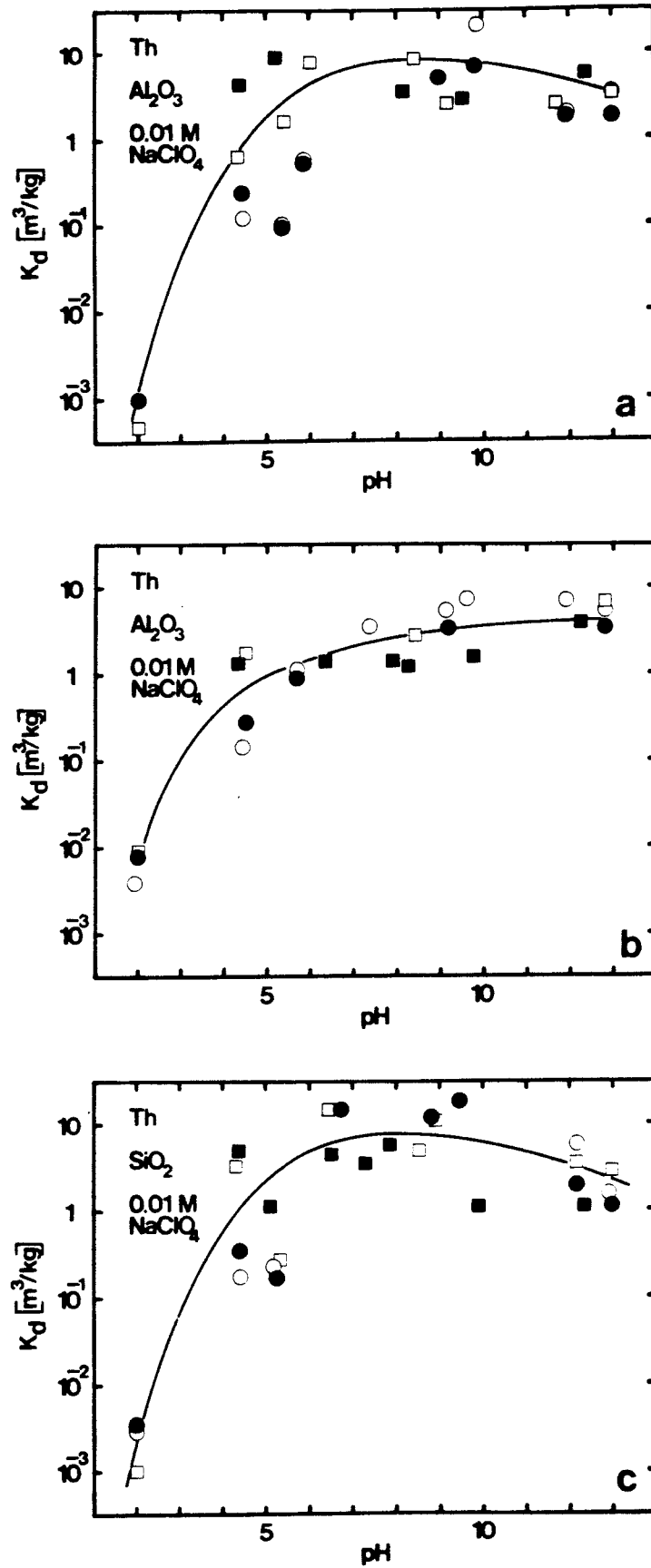
Figure 3 Distribution coefficients for thorium(IV).

○6h, ●27h, □1w, ■6w.

a. SI, b,c. SII.
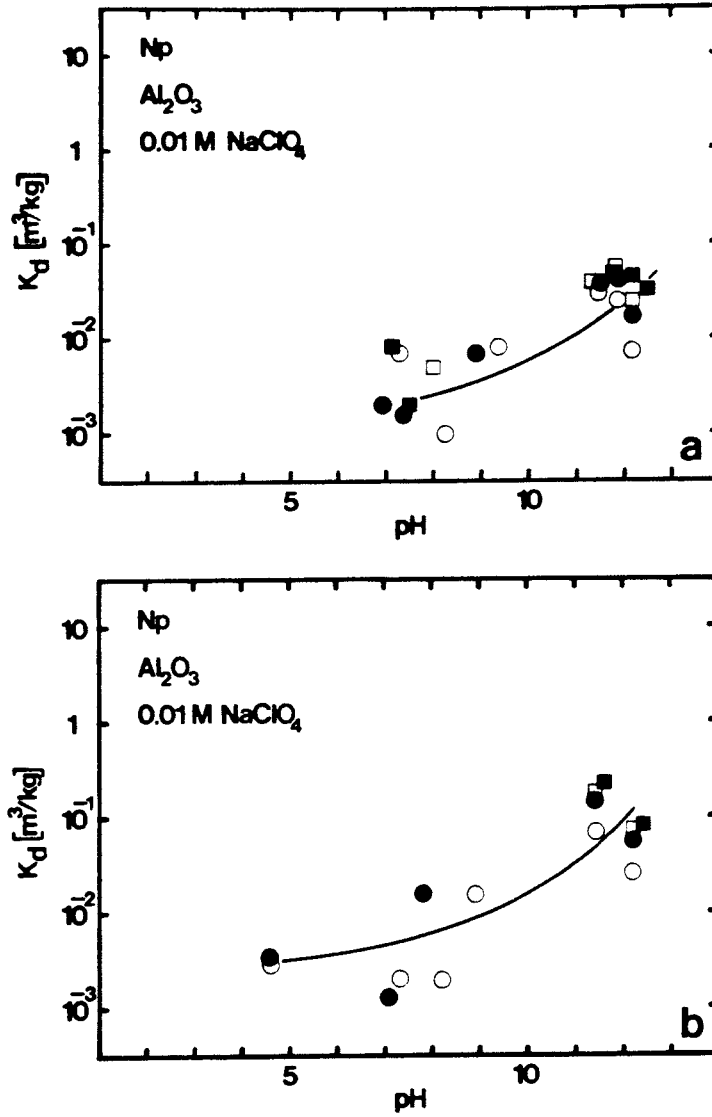
Figure 4 Distribution coefficients for neptunium(V).

○6h,   ●27h,   □1w,   ■4w.

a. SI, b. SII.
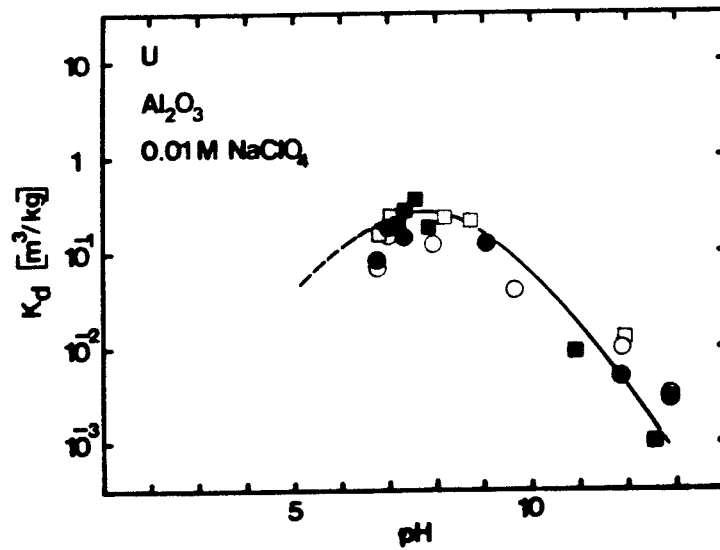
Figure 5 Distribution coefficients for uranium(VI).

○6h,   ●27h,   □1w,   ■6w.
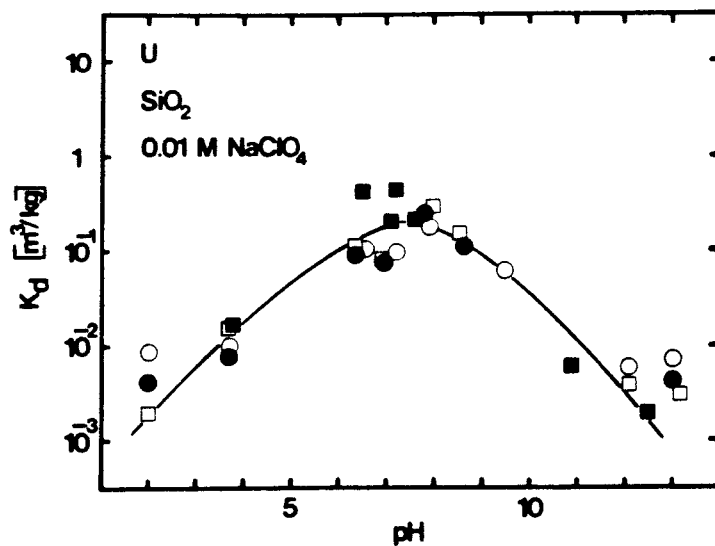
Figure 5 Distribution coefficients for uranium(VI)(Cont.).
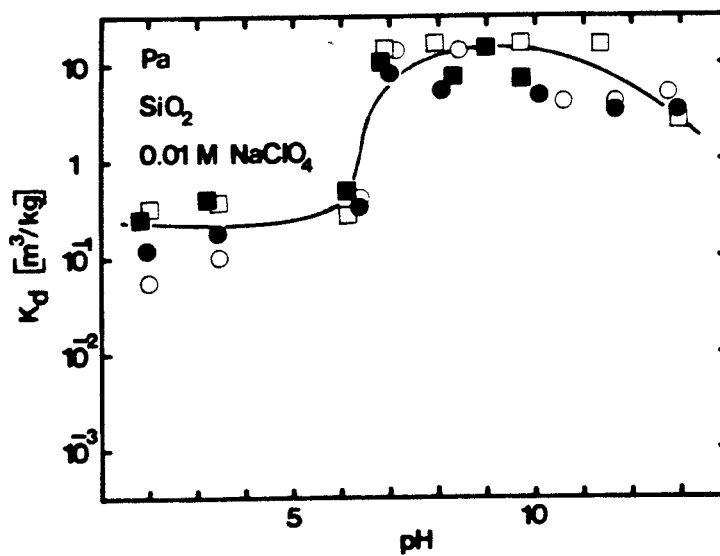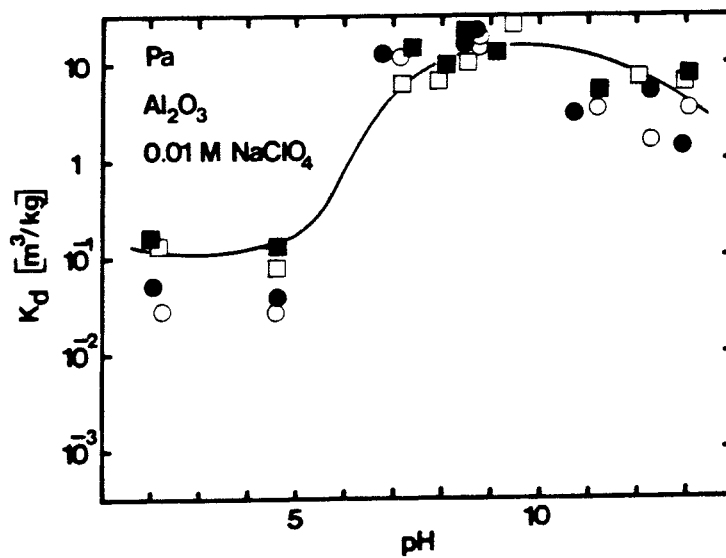
Figure 6 Distribution coefficients for protactinium(V).

○6h,   ●27h,   □1w,   ■6w.
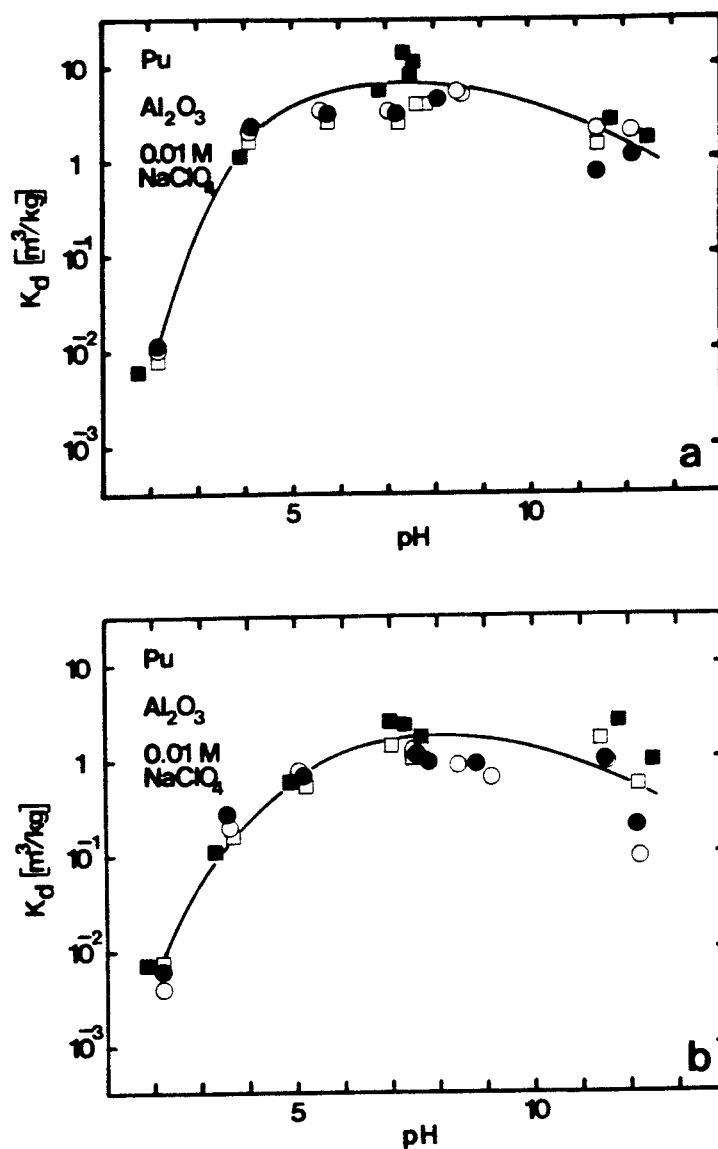
Figure 7 Distribution coefficients for plutonium.

○6h, ●27h, □lw, ■6w.

a. SI, b. SII.

Obviously, the degree of hydrolysis, determined by pH of the system, has a large influence on the distribution between the solid and aqueous phases. The increase in sorption with increasing pH seems to reflect the progressive hydrolysis, as illustrated in Fig. 1, and the sorption maximum roughly corresponds to the pH-range where neutral species would exist. This is particularly the case for the Th(IV)-systems, where no formation of anionic hydroxy species or carbonates would be expected (2,7).

The significant decrease in distribution with increasing pH observed for Am(III) and Pu, and the pronounced decrease observed for U(VI) could reflect the formation of anionic species (carbonates and hydroxides). The non-existence of a similar behaviour in the Np(V)-system either indicates a sorption behaviour in this system different from the behaviour of U(VI), or more likely errors in the calculated speciation of Fig. 1 (to high carbonate complex formation constants), as previously pointed out (6).

The general sorption behaviour in the Pu-system strongly indicates that plutonium is predominantly tetravalent under the present conditions (aerated solutions without excess carbonate). Possibly a minor fraction could be either Pu(III) (at pH below 5) or Pu(V) (at pH below 5-6 or above 8-9), considering the general sorption behaviour of Am(III), Th(IV), and Np(V), and the similarity in chemical behaviour of different actinides in the same oxidation state. The predominance of Pu(IV) in the intermediate pH-range with some possible contributions of Pu(III) and Pu(V) is in good agreement with calculated speciation (2,6).

The hydrolysis dependence of the sorption is further illustrated in Fig. 8, showing pH corresponding to 10% and 50% sorption vs the first hydrolysis constant (log K_1). Here, the assigned log K_d-value might be too high in the plutonium system (Pu(IV) assumed), considering the possible existence of a minor fraction of less hydrolyzed Pu(III) or Pu(V). Considerably lower pH-values for the two selected sorption levels would be expected for Np(V) and U(VI) in the absence of carbonate (carbon dioxide-free atmosphere).

The differences in observed distribution coefficients between the two nuclide concentrations (cf. Table 2) are minor. Slightly higher values are obtained in the high-concentration systems (SI) for thorium and plutonium. However, these systems would be very close to saturation. The solubility product of $An(OH)_4(s)-AnO_2(s)$ would be exceeded in the absence of any sorbing surfaces (2). Slightly higher distribution coefficients were observed in the low-concentration systems (SII) for neptunium. Similar observations have previously been made for uranium (1).
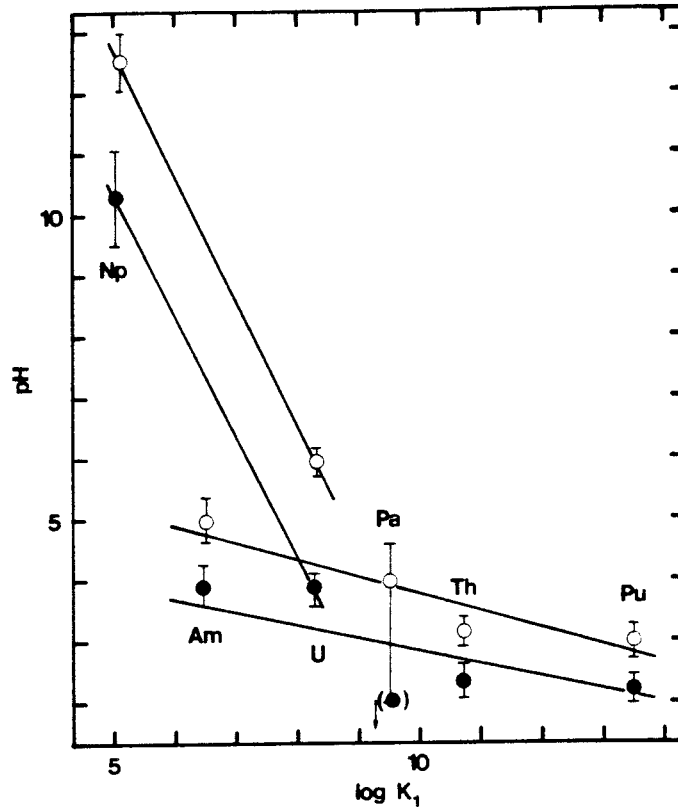
Figure 8 Hydrolysis dependence of the distribution.

Distribution data from Fig. 2-5.

O 50% sorption (K_d = 0.1 m^3/kg).

● 10% sorption (K_d = 0.01 m^3/kg).

There is generally a significant increase of the sorption with the contact time, (e.g. from 6h to 1d, 1w and to 4-6w) in the systems where carbonate complexes would not dominate. This can not solely be attributed to the delay due to diffusion into microfissures and crystal lite boundaries in the solid grains (radii around 50 μm) but rather to slow kinetics in the formation of sorbing higher hydrolysis products or colloidal aggregates. An increased ionic strength appears merely to speed up these processes (11).

In general, three basic kinds of sorption can be distinguished (12):

Physical adsorption processes are due to non-specific forces of attraction between the sorbent and the solute. This type of sorption results in the binding of the solute to solid surfaces in several consecutive layers. This would be a reversible, fairly rapid and largely concentration independent process.

Electrostatic adsorption processes (ion exchange) are due to the action of attractive coulomb forces between charged particles in solution and in the solid. This is often a reversible, fairly rapid and highly concentration dependent process.

The action of chemical forces between a solute and a sorbent would lead to chemisorption processes, e.g. when anions in the solid lattice could form strong complexes with the solute. This would be a highly element specific, partly irreversible, fairly slow and concentration dependent process.

All three of these basic sorption mechanisms can be observed in the interactions of actinides with geologic materials under environmental conditions (1). However, the sorption mechanism can largely be considered as a physical adsorption process, when

o   hydrolyzed species or other complexes dominate in the aqueous phase (at pH above 5-6 for Am(III), 3-4 for Th(IV), 7-8 for Np(V), 4-5 for U(VI), 2-3 for Pa(V) and 3-4 for Pu in aerated systems),
o   no chemisorbing agents are present.

The properties of the solid in terms of exchange capacities have only a minor influence on the sorption behaviour under these conditions. (Cf. examples given in ref. 1 and 3: the distribution coefficient for Am(III) in bentonite-systems (cation exchange capacity 750-800 meq/kg) and Al_2O_3-systems (cation exchange capacity < 1 meq/kg) are very similar at pH 8.5).

5.   CONCLUSIONS

The observed actinide sorption behaviour in the present experiments indicate a predominantly physical adsorption mechanism. The major chemical parameters that would have a significant influence on the actinide distribution between a solid and aqueous phase are

o   the redox conditions (determining the oxidation state),
o   pH (determining the degree of hydrolysis),
o   presence of complexing agents (e.g. carbonate).

The sorption of cationic or neutral hydrolyzed actinide species would be pronounced on exposed solid phases, even when the exchange capacity of these solids are low. However, the formation of anionic species (hydroxides, carbonates) would reduce the sorption.

Plutonium is largely tetravalent in aerated aqueous systems of environ-mental pH in the absence of other complexing agents than the hydroxide as well as the carbonate from the carbon dioxide-water equilibrium.

Further studies of actinide sorption on low-capacity solids in the presence of high concentrations of complexing agents are in progress. Results from these studies, as well as discussions on sorption mecha-nisms and the various sorption models suggested in the literature will be presented in a subsequent report.

6.   ACKNOWLEDGEMENTS

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