

# Recovery of Uranium from Phosphoric Acid Solutions Using Chelating Ion-Exchange Resins<sup>†</sup>

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Experimental measurements have been made on the batch extraction of uranium from phosphoric acid solutions using various chelating ion-exchange resins (RSPO, Diaion-CRP200, Diphonix, Purolite S940, Duolite ES467, and Lewatit OC 1060) and a solvent containing ion-exchange resins (Actinide-CU). The kinetic performance of ion-exchange resins was compared, and the effect of Fe(II) and Ca(II) ions on the sorption and elution performance has also been examined. The results showed that the resin Actinide-CU containing a diphosphonate extractant was very effective for removing uranium from phosphoric acid solution. However, the elution performance of this resin with both acid and carbonate eluants was poor. It is concluded that the chelating resins Diphonix, Duolite ES467, Lewatit OC 1060, and Purolite S940 give reasonable sorption of uranium in the presence of Fe(II) ions in batch sorption trials. The desorption of uranium has been performed quantitatively using carbonate eluants. Purolite S940 was used in small-scale column extractions of uranium from phosphoric acid solutions, and promising loading/elution profiles were obtained.

## 1. Introduction

Natural phosphates of sedimentary origin contain uranium concentration in the range 0.005%–0.02% U (Hodge and Popovici, 1994). In fertilizer manufacture, calcium phosphate in phosphate rock is rendered soluble by sulfuric acid attack. The phosphoric acid obtained in this way, denoted WPA (wet-process acid), usually contains 26%–28% P<sub>2</sub>O<sub>5</sub> (Hodge and Popovici, 1994). Several novel processes have been developed for the recovery of uranium from wet-process phosphoric acid, and the first industrial trials were based on solvent extraction. Three commercial processes have been developed using the following extractants: (1) A mixture of di(2-ethylhexyl)phosphoric acid (D2EHPA) and tri-octylphosphine oxide (TPOP) (Hurst and Crouse, 1971, 1973; Hurst et al., 1972; Boin, 1985). (2) A mixture of mono- and di-octylphenyl phosphoric acid (OPAP) (Hurst and Crouse, 1974, 1976). (3) Octylphenyl phosphoric acid (OPPA) (Kouloheris, 1980) and a mixture of mono- or di-octyl phosphoric acid (DOPA) and OPPA (Gorecki, 1985).

Ion exchange using polymeric resins is a well-established unit operation and has been used successfully to recover uranium from sulfate leach liquors. It has been reported that ion exchange is able to overcome some of the problems encountered in liquid–liquid extraction. Several ion-exchange resins have been developed for this particular application.

Bayer AG produced a series of polymeric resins containing the mixture D2EHPA/TOPO and D2EHPA/

TBP. These composite resins have been tested for the recovery of uranium from wet-process phosphoric acid (Gonzalez-Luque, 1982; Ketzin et al., 1985). Their capacity was found to be modest (1–3 g of U/kg of resin), but the rate of uptake was very slow (Ketzinel et al., 1985). Gonzalez-Luque and Streat found that the aminophosphonic resin, Duolite ES 467, was selective for the removal of uranium from wet-process phosphoric acid (Gonzalez-Luque, 1982; Gonzalez-Luque and Streat, 1983a,b, 1984).

It has already been reported that chelating resins containing amidoxime groups have a high capacity and selectivity for uranium in seawater (Kabay and Egawa, 1994). Fibrous adsorbents containing amidoxime groups have been used for the recovery of uranium from phosphoric acid solutions (Bernido et al., 1988). Recently, some hydrogels prepared with acrylamide/maleic acid (Saraydin et al., 1995) and acrylamide/itaconic acid (Karadağ et al., 1995) have been used in the adsorption of uranium. Resins containing dihydroxyphosphino and/or phosphano groups have been used for the second concentration process of uranium from acidic eluates of amidoxime resins (Egawa et al., 1990). Selective recovery of uranium by uranyl-templated gel-filled sorbents was reported by Chanda and Rempel (1996). Also, the uptake of uranyl ions from acidic solutions by a polyfunctional resin, Diphonix, has been investigated and shown to have considerable potential for uranium uptake (Horwitz et al., 1993; Chiarizia et al., 1996).

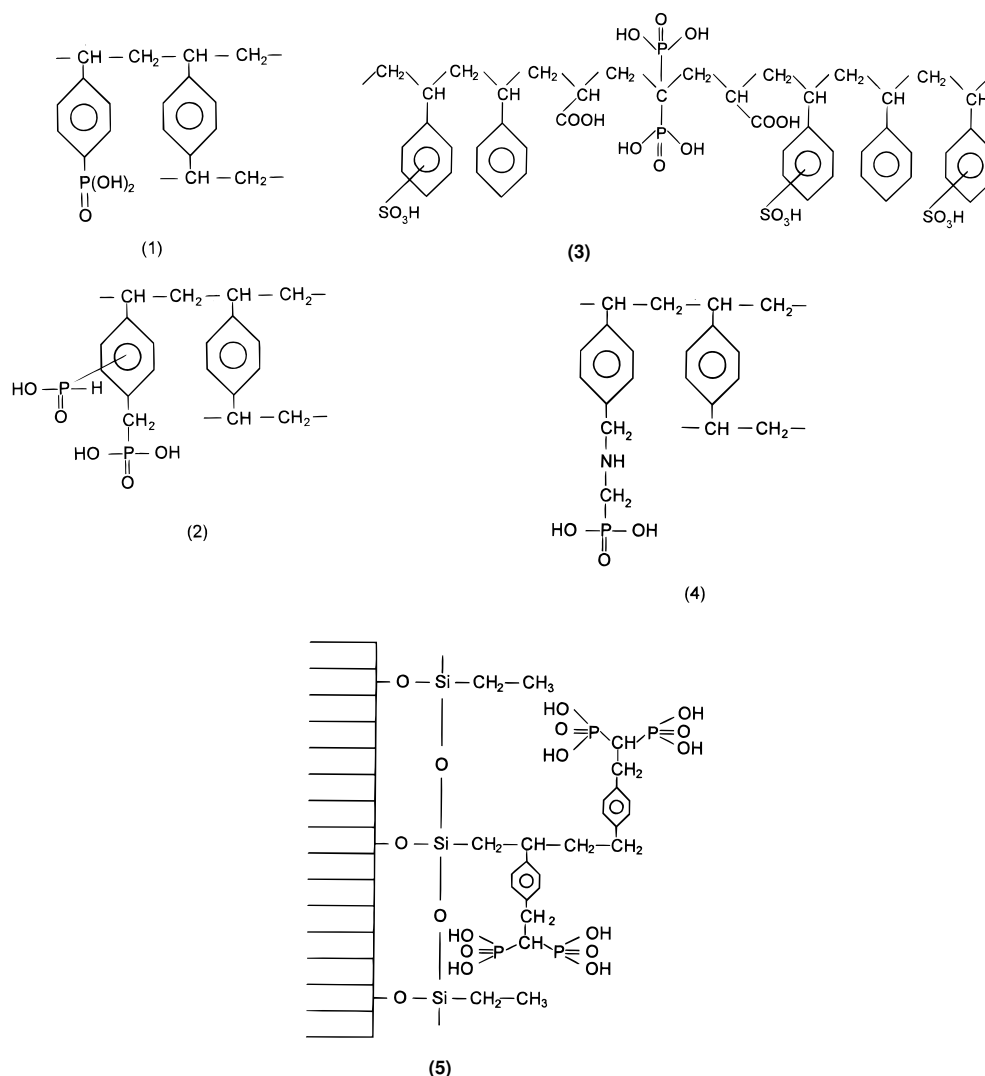
In this study, chelating ion-exchange resins containing phosphonic acid (–PO(OH)<sub>2</sub>) groups (RSPO) (1) or phosphinic (–PO(OH)H) groups (Diaion-CRP200) (2), polyfunctional ion-exchange resins (Diphonix) containing both sulfonic and gem-diphosphonic acid groups (3), aminophosphonic acid resins (Purolite S940 and Lewatit OC1060) (4), and silica-based resins containing diphos-

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<sup>†</sup> Dedicated to Prof. Hiroaki Egawa for his contributions in the field of chelating ion-exchange resins.

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phonate extractant (Actinide-CU) (5) have been used for uranium extraction from phosphoric acid solutions.

## 2. Experimental Section

**2.1. Materials.** Details of some commercial ion-exchange resins used in this work are given in Table 1. The resins RSPO and Duolite ES 467 (DES 467) were obtained from Kumamoto University, Japan, and these resins contain phosphonic acid functional groups. The resin Diaion CRP200 (D-CRP200) was kindly sent by Mitsubishi Chemical Co., Japan, and contains phosphonic functional groups. Diphonix and Actinide-CU resins were kindly given as a gift by EiChrom Industries Inc., United States. Purolite S940 (P-S940) was provided by Purolite International Ltd., United Kingdom, and contained aminomethylphosphonic acid groups. Lewatit OC1060 (L-OC1060) containing an aminophosphonic acid extractant was provided by Bayer AG, Germany. The resin samples were dried at 40 °C under vacuum. Phosphoric acid solutions were prepared from 85% *o*-phosphoric acid (analytical grade, Carlo Erba Chemical Co.), and deionized water was obtained from Teknik water purification system. The reagent  $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  was obtained from Merck. All other chemicals were reagent grade and used as received.

**2.2. Batchwise Sorption of Uranium.** Batch sorption experiments were carried out to investigate the

effects of acid concentration, contact time, temperature, presence of interfering cations, particle size, and elution condition (see Table 2). Batch equilibrations of uranium-spiked solutions in the range 10–1000 mg of  $\text{U}/\text{dm}^3$  were performed with either 50 or 100 mg of resin (the resin to solution was maintained at 2  $\text{mg}/\text{cm}^3$ ) in contact for 24 h at 30 °C. The effect of phosphoric acid in the range 0.1–3 M and the effect of time (2 and 24 h) and temperature (30 and 50 °C) were studied. Experiments in 40%  $\text{H}_3\text{PO}_4$  solution were also performed at 30 °C for 2 and 24 h, and at 50 °C for 2 h. The effect of interfering ions was performed at 40%  $\text{H}_3\text{PO}_4$  in the presence of Fe(II) (59 and 118  $\text{mg}/\text{dm}^3$ ) and Ca(II) (59  $\text{mg}/\text{dm}^3$ ) since these cations are encountered in wet-process phosphoric acid arising in the conventional acid leaching of calcium phosphate bearing minerals.

The elution of uranium from the resins Purolite S940 and Diphonix was examined using 1 M  $\text{Na}_2\text{CO}_3$  solution at 30 °C for 2 h and 1 M  $(\text{NH}_4)_2\text{CO}_3$  solution at 30 °C for 2 h for the resins Duolite ES467 and Lewatit OC1060. Stripping of uranium loaded on Actinide-CU was also studied under standard batch conditions using  $\text{Na}_2\text{CO}_3$ ,  $(\text{NH}_4)_2\text{CO}_3$ ,  $\text{NaHCO}_3$ ,  $\text{NH}_4\text{HCO}_3$ , and HCl at 30 or 50 °C for 2 or 24 h (see Table 3).

**2.3. Small-Scale Column Sorption of Uranium (Test VIII).** Glass columns of internal diameter 0.7 cm were used. Each column was packed with a 3.0- $\text{cm}^3$  wet-settled volume of resin (Purolite S940) with a

**Table 1. Characteristics of Ion-Exchange Resins**

characteristic	resin <sup>a</sup>					
	RSPO	Diaion-CRP200	Diphonix	Purolite S940	Actinide-CU	Duolite ES 467
total exchange capacity (mequiv/g)	11.22	11.30	7.31	5.50	1.36	7.80
P content (wt %)	14.78	13.20	1.64 <sup>b</sup>	N. D.	0.68 <sup>b</sup>	10.30
specific surface area (m <sup>2</sup> /g)	0.35	16.9	0 (gel)	16.0	79.9	14.1
dry volume (cm <sup>3</sup> /g)	1.32	1.91	N.D.	N.D.	N.D.	2.80
or						
dry density (g/cm <sup>3</sup> )	N.D.	N.D.	0.3	0.36	0.39	N.D.
wet volume (cm <sup>3</sup> /g)	2.71	2.91	N.D.	N.D.	N.D.	3.30
or						
wet density (g/cm <sup>3</sup> )	N.D.	N.D.	1.16	1.11	1.50	N.D.
UO <sub>2</sub> (II) sorption capacity (mmol/g)	1.23	2.27	2.3	1.48	1.10	N.D.

<sup>a</sup> N.D.: not determined. <sup>b</sup> mmol/dry g.**Table 2. Uranium Batch Sorption Tests**

test	resin	H <sub>3</sub> PO <sub>4</sub> concn. (M)	U concn. (mg/dm <sup>3</sup> )	temp. (°C)	time (h)	particle size (mm)	impurities (mg/dm <sup>3</sup> )
I (Figure 1)	RSPO	0.1–1.0	10–500	30	24	0.25–0.50	
II (Figures 2 and 3)	RSPO	0.1–3.0	100	30/50	2/24	0.25–0.50	
	D-CRP200	0.1–3.0	100	30/50	2/24	0.25–0.50	
	Diphonix	0.1–3.0	100	30/50	2/24	100–200 mesh	
	Actinide	0.1–3.0	100	30/50	2/24	100–150 μm	
	Actinide	40%	250–1000	30/50	2/24	100–150 μm	
III (Figure 4)	Actinide	40%	250	30	2/24	100–150 μm	
	Diphonix	40%	250	30	2/24	100–200 mesh	
IV (Table 4)	P–S940	40%	250	30	2/24	0.355–0.710	
	Actinide	40%	250	30	2/24	100–150 μm	Fe(II)-59 or Fe(II)-118
	Diphonix	40%	250	30	2/24	100–200 mesh	Fe(II)-59 or Fe(II)-118
	P–S940	40%	250	30	2/24	0.355–0.710	Fe(II)-59 or Fe(II)-118
	L-OC1060	40%	250	30	2/24	0.355–0.710	Fe(II)-59 or Fe(II)-118
	D-ES467	40%	250	30	2	0.355–0.710	Ca(II)-59
	L-OC1060	40%	250	30	2	0.355–0.710	Ca(II)-59
V (Table 5)							

**Table 3. Uranium Batch Stripping Tests**

test <sup>a</sup>	resin	eluant	U concn. (mg/dm <sup>3</sup> )	temp. (°C)	time (h)	particle size (mm)
VI	Actinide	Na <sub>2</sub> CO <sub>3</sub>	250	30/50	2/24	100–150 μm
		(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	250	30/50	2/24	100–150 μm
		NaHCO <sub>3</sub>	250	30/50	2/24	100–150 μm
		NH <sub>4</sub> HCO <sub>3</sub>	250	30/50	2/24	100–150 μm
		HCl	250	30/50	2/24	100–150 μm

<sup>a</sup> See the results of test VI in Table 6.

particle size range 0.355–0.500 mm. Phosphoric acid solution (1–6 M) containing uranium (0.01 M) was delivered in downflow to the column using a peristaltic pump (Atto SJ 1211) capable of delivering a flow rate of SV 10 h<sup>-1</sup> (SV, space velocity (cm<sup>3</sup>/h)/cm<sup>3</sup> of resin). The breakthrough curves were obtained by analysis of successive fractions (6 cm<sup>3</sup>) of the effluent. The fractions were collected using a fraction collector (Advantec SF 2120). The columnar elution of uranium was performed at SV 5 h<sup>-1</sup> using 1 M Na<sub>2</sub>CO<sub>3</sub>, and the elution profiles were obtained by analysis of successive 3-cm<sup>3</sup> fractions.

**2.4. Uranium Analysis.** The determination of uranium in the solution has been performed spectrophotometrically with Arsenazo III using a Jasco Spectrophotometer. The uptake of uranium (i.e., recovery (%)) and/or mg of U adsorbed/g of resin was calculated from the difference in uranium concentration in the solution before and after the sorption.

### 3. Results and Discussion

**3.1. Batchwise Sorption of Uranium from Phosphoric Acid Solutions.** **3.1.a. Effect of Acid Concentration.** It is well-documented that macroreticular chelating resins containing phosphonic (RSP, RSPO) and/or phosphinic acid groups (RCSP, RCSPO) possess

a large capacity as well as high selectivity toward hard Lewis cations such as uranyl ions (Egawa et al., 1990). Bearing this in mind, chelating resins containing phosphonic groups (RSPO) have been used for uranium uptake from phosphoric acid solutions at various concentrations. The UO<sub>2</sub>(II) capacities of the resins RSPO, Diphonix, Actinide-CU, Diaion-CRP200, and Purolite S940 were determined according to a procedure described in the literature (Kabay and Egawa, 1993). All the resins show a substantial sorption capacity for UO<sub>2</sub>(II) ions in an aqueous medium at pH 3 (see Table 1). Egawa et al. have reported that chelating resins containing phosphonic groups (RSPO) are very effective in concentrating uranium from acidic eluates of amidoxime resins (Egawa et al., 1990). In this study, a comparison of acid dependency for the uptake of uranium from the phosphoric acid solutions was studied using RSPO (Test I). The results are shown in Figure 1. Although the uptake of uranium increased with an increase in initial uranium concentration, a certain decrease in uranium recovery was observed by the increase in acidity.

**3.1.b. Effect of Sorption Time and Temperature.** Test II has been performed at various concentrations of phosphoric acid solutions. As summarized in Figures 2 and 3, an increase in sorption time from 2 to 24 h

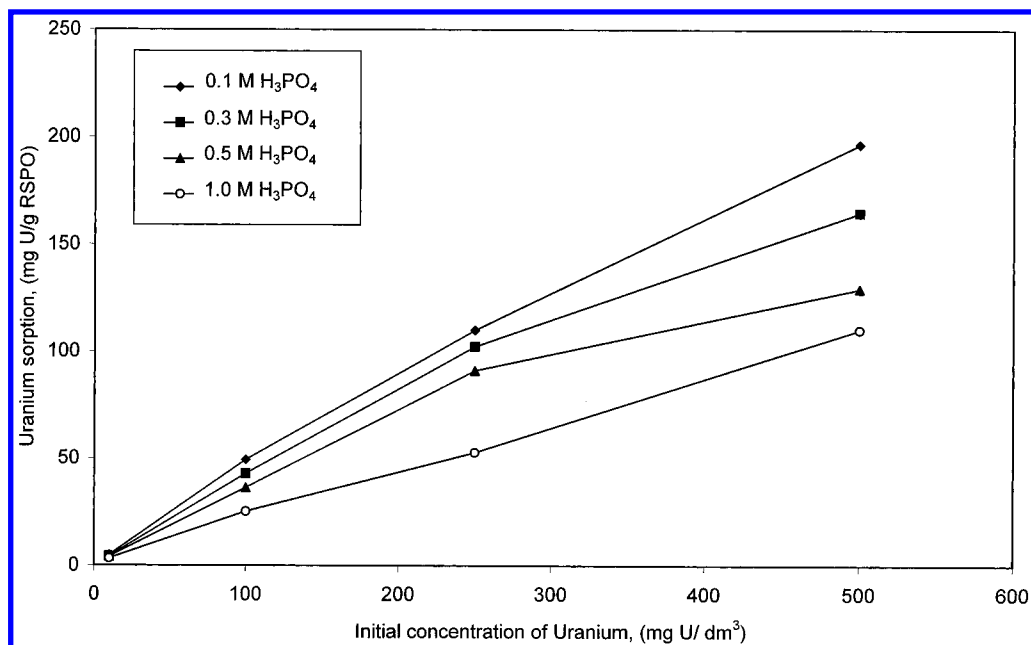


Figure 1. Effect of phosphoric acid concentration on uranium uptake from phosphoric acid solution by RSPO.

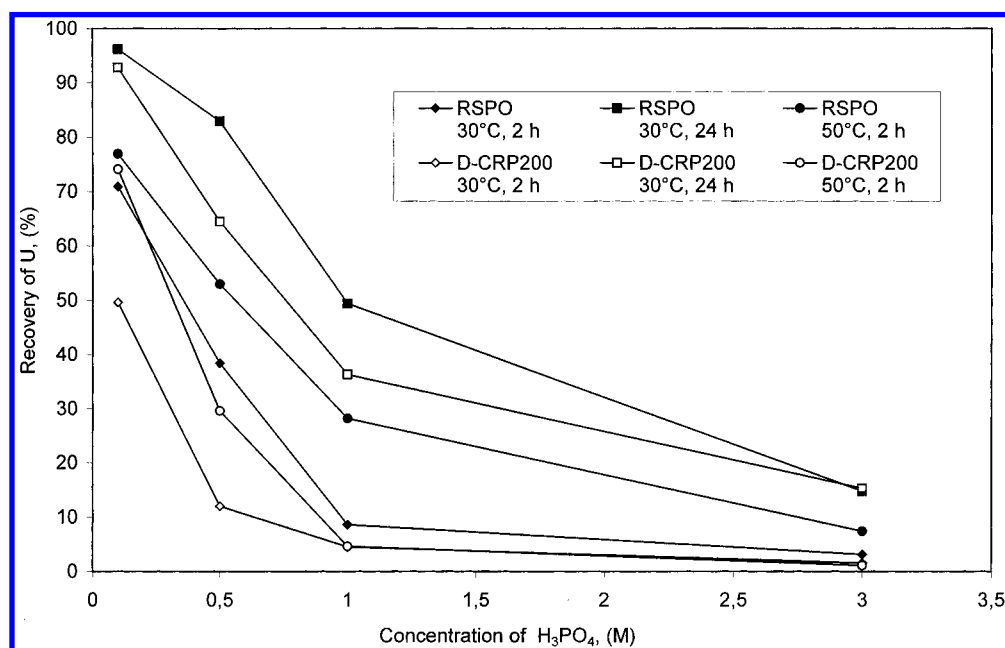


Figure 2. Effect of sorption time and temperature on uranium uptake as a function of phosphoric acid concentration.

significantly improved uranium uptake of RSPO and Diaion CRP200. Some increase was also observed for Diphonix and Actinide-CU resins. Diphonix exhibited relatively fast kinetics and gave excellent sorption of uranium from phosphoric acid in the concentration range 0.1–0.5 M. However, some reduction in uranium uptake occurred in the concentration range 1–3 M H<sub>3</sub>PO<sub>4</sub>. An increase in temperature from 30 to 50 °C significantly improved the sorption kinetics of RSPO, Diaion CRP200, and Actinide-CU, especially in the concentration range of 0.1–1 M. For Diphonix, however, the effect of temperature on the resin sorption kinetics was less apparent.

Actinide-CU is potentially the most powerful chelating resin tested in high-concentration phosphoric acid. Subsequent kinetic experiments confirmed the efficiency of Actinide-CU resin for uranium sorption in 40% H<sub>3</sub>PO<sub>4</sub> which is close to the concentration of industrial grade wet-process phosphoric acid.

The sorption isotherm for uranium was produced for this resin by using uranium solutions with concentrations of 250, 500, and 1000 mg of U/dm<sup>3</sup> (Test III). As shown in Figure 4, the isotherm barely reached saturation with a solution of 1000 mg/dm<sup>3</sup>. Also, an increase in both sorption time and temperature did not improve the uptake.

**3.1.c. Effect of Other Cations.** In Test IV, batch extractions of uranium from 40% H<sub>3</sub>PO<sub>4</sub> were performed using various chelating resins. The results are given in Table 4. The presence of Fe(II) which reduces uranium to its tetravalent oxidation state inhibits uranium uptake on Actinide-CU, Duolite ES467, Puro-lite S940, and Lewatit OC1060 at a 2-h sorption time. However, the effect of Fe(II) on uranium sorption was negligible at 24 h. This might be due to the reoxidation of uranium to its hexavalent state.

The effect of Ca(II) ions on sorption behavior of uranium was examined using both Duolite ES467 and

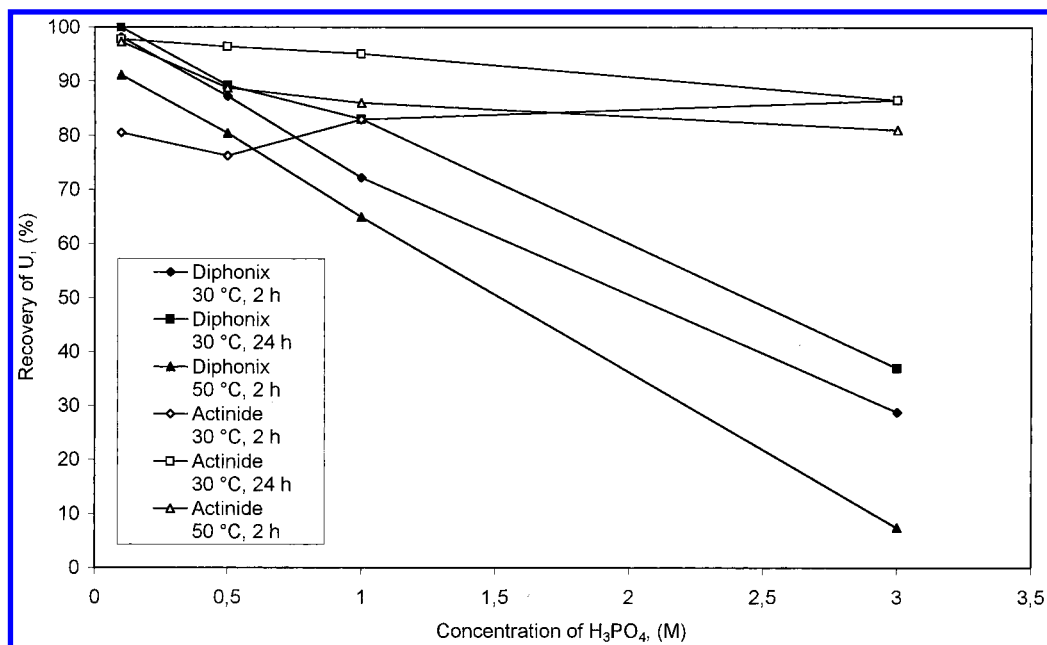


Figure 3. Effect of sorption time and temperature on uranium uptake as a function of phosphoric acid concentration.

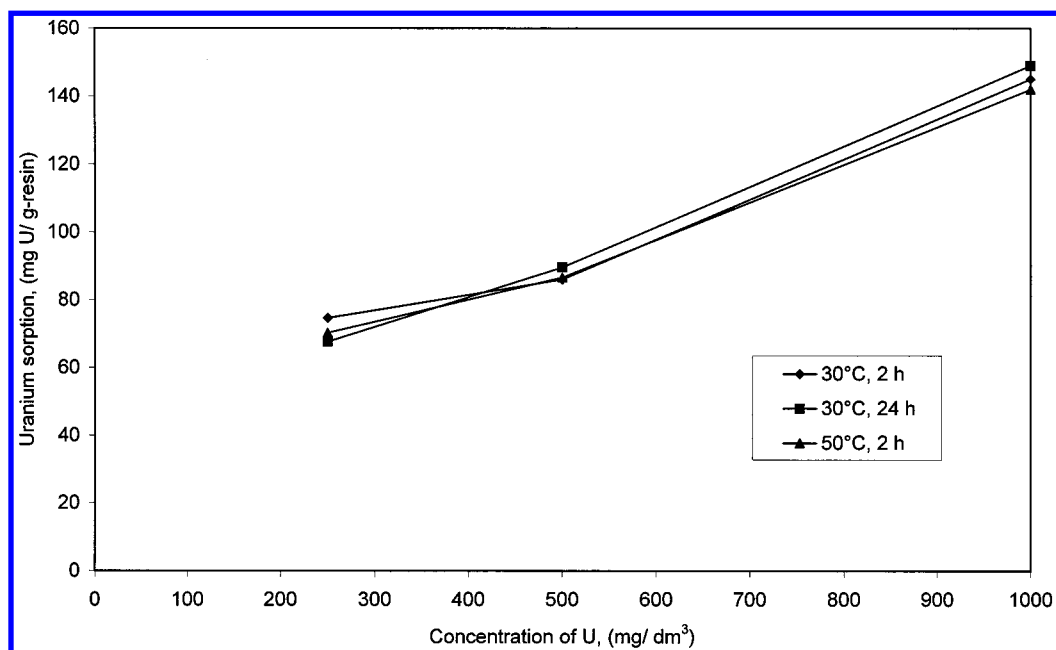


Figure 4. Effect of sorption time and temperature on uranium uptake from 40%  $\text{H}_3\text{PO}_4$  solution by Actinide-CU as a function of uranium concentration.

Table 4. Batch Sorption/Elution of Uranium

resin <sup>b</sup>	sorption time (h)	solution <sup>a</sup>					
		U in 40% $\text{H}_3\text{PO}_4$		U-Fe(II) (1:1) in 40% $\text{H}_3\text{PO}_4$		U-Fe(II) (1:2) in 40% $\text{H}_3\text{PO}_4$	
		sorption (%)	elution (%)	sorption (%)	elution (%)	sorption (%)	elution (%)
Actinide	2	38.8	N.D.	13.4	27.4	17.9	21.1
	24	36.2	30.0	26.9	N.D.	24.1	15.1
Diphonix	2	20.7	52.9	17.9	54.2	16.2	51.0
	24	18.0	49.6	22.7	43.3	22.7	48.0
P-S940	2	17.5	90.0	14.3	93.5	13.8	97.0
	24	19.2	100.0	27.5	78.3	21.7	85.0
L-OC1060	2	22.4	89.6	22.7	73.0	18.5	77.0
	24	33.6	100.0	29.0	85.0	20.9	100.0

<sup>a</sup> N.D.: not determined. <sup>b</sup> The resins Actinide-CU, Diphonix, and Purolite S940 were eluted with 1 M  $\text{Na}_2\text{CO}_3$  at 30 °C for 2 h and Lewatit OC1060 with 1 M  $(\text{NH}_4)_2\text{CO}_3$  at 30 °C for 2 h.

Lewatit OC1060 resins (Test V). The results are compared with those obtained using the solution containing only uranium in 40%  $\text{H}_3\text{PO}_4$ . The effect of

Ca(II) on uranium sorption by either Duolite ES467 or Lewatit OC1060 was negligible (Table 5).

**3.1.d. Batch Elution of Uranium.** Although Ac-



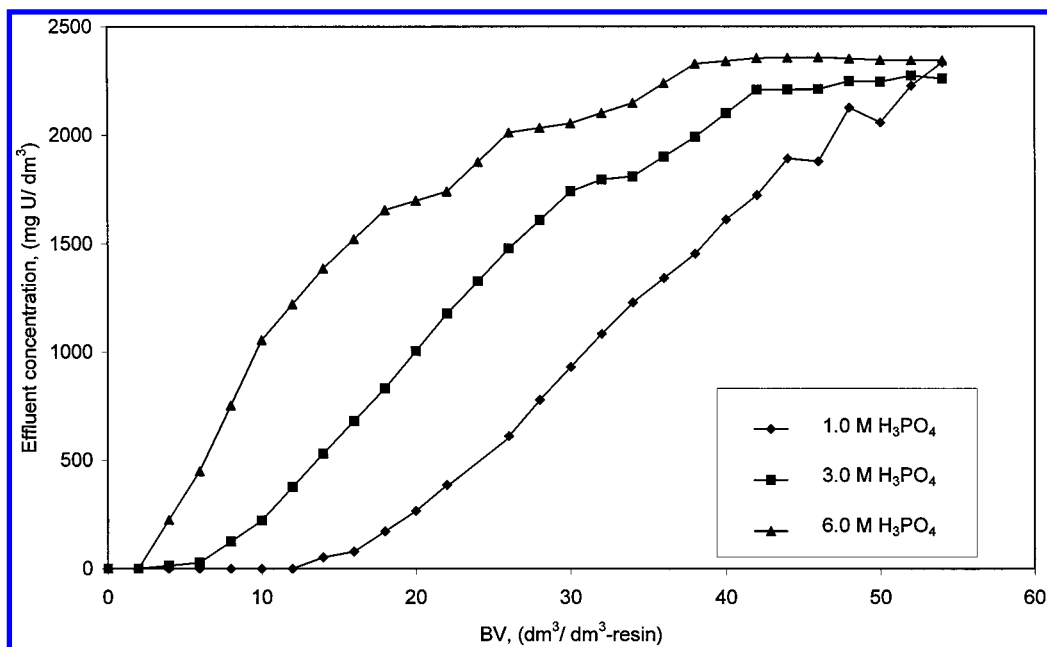


Figure 5. Breakthrough curves of uranium.

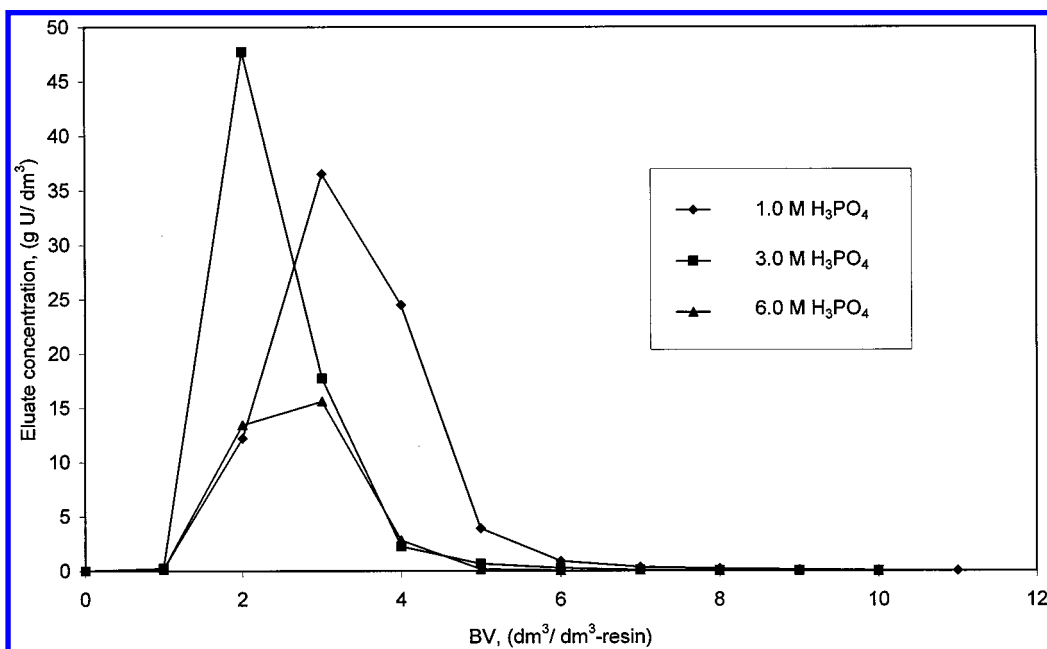


Figure 6. Elution curves of uranium.

Table 5. Effect of Ca(II) on Sorption/Elution of Uranium

resin	solution			
	U (250 mg/dm³) in 40% H <sub>3</sub> PO <sub>4</sub>		U–Ca(II) (1:1.5) in 40% H <sub>3</sub> PO <sub>4</sub>	
	U-sorption (%)	U-elution (%)	U-sorption (%)	U-elution (%)
Duolite ES-467	17.5	90.0	17.4	74.0
Lewatit OC1060	22.4	89.6	19.9	98.3

tinide-CU exhibited remarkably high uptake for uranium from 40% H<sub>3</sub>PO<sub>4</sub>, the stripping of uranium from Actinide-CU was less successful (see Table 6). This was due to the apparently high affinity of uranium for Actinide-CU. According to the producer company, EiChrom Industries Inc., the silica-based resin Actinide-CU containing diphosphonate extractant was actually designed to be nonstrippable (Horwitz and Gula, 1996).

The elution behavior of chelating resins was studied using carbonate eluants, and the results are summarized in Table 4. The elution of uranium from the

resin Purolite S940 and Lewatit OC1060 was quantitative with 1 M Na<sub>2</sub>CO<sub>3</sub> and 1 M (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>, respectively.

It was assumed that reduction of uranium to its tetravalent state would improve the elution efficiency of uranium due to the lower affinity of U(IV) for the resin. This did not improve the elution performance of Actinide-CU, Diphonix, Lewatit OC1060 and Purolite S940. An explanation for these results obtained with Actinide-CU, Diphonix, Lewatit OC1060, and Purolite S940 may be the high affinity for tetravalent uranium. The effect of Ca(II) on elution behavior of uranium was

**Table 6. Stripping of Uranium from Actinide-CU**

eluting agent	stripping condition	U-elution (%)
0.25 M Na <sub>2</sub> CO <sub>3</sub>	30 °C, 2 h	2.3
0.5 M Na <sub>2</sub> CO <sub>3</sub>	30 °C, 2 h	3.4
1.0 M Na <sub>2</sub> CO <sub>3</sub>	30 °C, 2 h	6.6
2.0 M Na <sub>2</sub> CO <sub>3</sub>	30 °C, 2 h	12.0
2.0 M Na <sub>2</sub> CO <sub>3</sub>	30 °C, 24 h	37.0
2.0 M (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	30 °C, 2 h	7.7
2.0 M (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	30 °C, 24 h	7.0
2.0 M (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	50 °C, 2 h	12.3
1 M HCl	30 °C, 2 h	1.4
6 M HCl	30 °C, 2 h	3.2

examined using both Duolite ES467 and Lewatit OC1060 resins (Test V). Both resins were quantitatively eluted with 1 M (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> solution.

**3.2. Column Sorption of Uranium from Phosphoric Acid Solution by Purolite S940.** The resin Purolite S940 was loaded using UO<sub>2</sub>(II) solution with a concentration of 0.01 M UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub> in 1, 3, and 6 M H<sub>3</sub>PO<sub>4</sub>, respectively (Test VI). The breakthrough curves are shown in Figure 5. The elution of Purolite S940 loaded with UO<sub>2</sub>(II) was performed using 1 M Na<sub>2</sub>CO<sub>3</sub>, and the resulting elution profiles are shown in Figure 6. As shown in Figure 5, the breakthrough capacity of the resin decreased with an increase in H<sub>3</sub>PO<sub>4</sub> concentration. The resins loaded with uranium were quantitatively eluted with 1 M Na<sub>2</sub>CO<sub>3</sub>. Gonzalez-Luque and Streat have reported that Duolite ES467 can be readily eluted using ammonium carbonate solution at 20 °C (Gonzalez-Luque, 1982; Gonzalez-Luque and Streat, 1984). However, to minimize the waste effluent treatment process, it is preferable to use sodium carbonate solution as an eluant since the environmental impact of ammonium salts is considerably greater (Kanno, 1980). The relative expense of using carbonate solutions as eluant demands that they must be recovered and recycled. It has been shown that electrodialysis using ion exchange membranes is an efficient technique for sodium carbonate recovery (Kanno, 1980).

## Conclusions

Batch extractions of uranium from phosphoric acid solution have been performed using various chelating ion-exchange resins (i.e., RSPO, Diaion-CRP200, Diphonix, Purolite S940, Duolite ES467, and Lewatit OC1060) and a silica-based chelating resin (Actinide-CU). The columnar behavior of the aminophosphonic resin Purolite S940 for uranium has been studied using phosphoric acids at various concentrations. The breakthrough capacity of the resin for uranium was strongly influenced by the acid strength of the phosphoric acid solution. The loaded uranium was quantitatively eluted with 1 M Na<sub>2</sub>CO<sub>3</sub>.

## Acknowledgment

This work was supported by a Grant of Avicenne Initiative (AVI-CT 94-0014) from the European Commission. We thank the European Commission for continuing support of this research. We also thank Purolite International Ltd. (England and Italy), EiChrom Industries Ltd. (United States and Scotland), Bayer AG (Germany), and Kumamoto University (Japan) for the support in providing the ion-exchange resin samples. Particular thanks are due to Prof. A. Jyo, Kumamoto University (Japan), Dr. P. Horwitz, Argonne National Laboratories (United States), Prof. S. Alexandratos,

University of Tennessee (United States), Prof. F. Cloete, University of Stellenbosch (South Africa), Prof. D. Muraviev, Autonomous University of Barcelona (Spain), and Dr. A. Zagorodni, Royal Institute of Technology (Sweden), for discussions.

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*Received for review* July 18, 1997

*Revised manuscript received* February 6, 1998

*Accepted* February 7, 1998

IE970518K