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## Extraction and separation of U(VI) and Th(IV) from hydrobromic acid media using Cyanex-923 extractant

SNEHAL M. GHAG and SURESH D. PAWAR\*

*Department of Chemistry, Siddharth College of Arts, Science and Commerce,  
Dr. D. N. Road, Fort, Mumbai – 400 001, India*

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**Abstract:** A systematic study of the solvent extraction of uranium(VI) and thorium(IV) from hydrobromic acid media was performed using the neutral phosphine oxide extractant Cyanex-923 in toluene. These metal ions were found to be quantitatively extracted with Cyanex-923 in toluene in the acidity range  $5 \times 10^{-5}$ – $1 \times 10^{-4}$  M and  $5 \times 10^{-5}$ – $5 \times 10^{-3}$  M, respectively, and they are stripped from the organic phase with 7.0 M  $\text{HClO}_4$  and 2.0–4.0 M HCl, respectively. The effect of the equilibrium period, diluents, diverse ions and stripping agent on the extraction of U(VI) and Th(IV) was studied. The stoichiometry of the extracted species of these metal ions was determined based on the slope analysis method. The extraction reactions proceed by solvation and their probable extracted species found in the organic phase were  $\text{UO}_2\text{Br}_2 \cdot 2\text{Cyanex-923}$  and  $\text{ThBr}_4 \cdot 2\text{Cyanex-923}$ . Based on these results, a sequential procedure for their separation from each other was developed.

**Keywords:** solvent extraction; uranium(VI); thorium(IV); Cyanex-923; stripping; separation.

### INTRODUCTION

In last few years, organophosphorous compounds have found wide application in nuclear establishments for the extraction, enrichment and reprocessing of uranium, thorium and plutonium. The new extractant di-(2-ethylhexyl) sulphoxide (DEHSO) is used for the reprocessing of spent Th–U fuel and extraction of U(VI) and Th(IV) from dilute  $\text{HNO}_3$  media.<sup>1</sup> Preston *et al.*<sup>2</sup> performed the extraction of U(VI) and Th(IV) from  $\text{NaNO}_3$  media using *N*-alkylcarboxylic acid amide. Quantitative extraction of Th(IV) and U(VI) from  $\text{HNO}_3$  media was investigated by Dhadke *et al.*<sup>3</sup> using bis-(2-ethylhexyl) phosphinic acid (PIA-8) and bis(2-ethylhexyl) phosphoric acid (HDEHP).

\* Corresponding author. E-mail: sureshpawar\_2004@rediffmail.com  
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The extraction of U(VI) with Th(IV) was performed from nitrate media using extractants such as *N,N'*-tetrahexylbipicolinamide (THBPA) and *N,N'*-tetra(2-ethylhexyl)bipicolinamide, *O*-methyleneoxyphenyldiphosphine dioxide and butyl bis(dibutoxyphosphinylmethyl) phosphinate.<sup>4-6</sup> The separation of Th, La, Eu ions was realised using the acidic extractant bis(2-ethylhexyl) phosphoric acid (DEHPA) in cyclohexane using 2-nitrobenzo-18-crown-6 [NB18(6)].<sup>7</sup> LIX-360 (HX) in kerosene and Versalic-10 was also used for extraction studies of U(VI).<sup>8</sup> In addition, 2-hydroxy-1-naphthaldehyde thiosemicarbazone was used to study the extraction behaviour of Th(IV) from HNO<sub>3</sub> in the presence of donors, tri-octylphosphine oxide (TOPO), Calix[3]OH[3]OMe[6]arene, trioctyl amine (TOA) and DMSO in ethyl acetate solvent.<sup>9</sup>

A new chelating polymeric sorbent was developed using Merrifield chloromethylated resin anchored with di-bis(2-ethylhexyl)malonamide (DB2EHM) for the selective extraction of U(VI) and Th(IV) from an acidic stream.<sup>10</sup> The branched chain di(2-ethylhexyl) isobutyramide (D2EHIBA) was chosen as a promising alternative to tri-*n*-butyl phosphate (TBP) for the recovery of U from irradiated Th.<sup>11</sup>

## EXPERIMENTAL

### *Apparatus and reagents*

The extractant Cyanex-923, supplied by Cytec Industries Inc. Canada, was used without further purification. Known amounts of uranyl nitrate UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O and thorium nitrate Th(NO<sub>3</sub>)<sub>4</sub>·5H<sub>2</sub>O were dissolved in water and diluted to 1.0 dm<sup>3</sup> with double distilled water. All other employed chemical were of analytical grade. An Equip-Tronic model EQ-614 pH meter with a combined electrode was used for H<sup>+</sup> concentration studies and an Elico UV-Vis SL-27 spectrophotometer with 10 mm cortex quartz cuvettes was employed for the absorbance measurements.

### *Procedure*

An aliquot of solution containing U(VI) (20 µg) or Th(IV) (20 µg) was taken, diluted to 10 ml and equilibrated with an equal volume of Cyanex-923 diluted in toluene for the required shaking time of 10 min, after adjusting the acidity of the aqueous solution to 1.0×10<sup>-4</sup> and 1.0×10<sup>-3</sup> M HBr, respectively. The organic phase containing the extracted metal species was then stripped with different stripping agent, such as HCl, HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub> and HClO<sub>4</sub>. It was found that U(VI) and Th(IV) can be quantitatively stripped from the metal loaded organic phases of Cyanex-923 with 7.0 M HClO<sub>4</sub> and 2.0 M HCl solutions, respectively, as was determined spectrophotometrically using Arsenazo-I and Thoron-I indicators, respectively.<sup>12</sup> All the experiments were performed at room temperature, except when the effect of temperature on the distribution equilibria was studied.

## RESULTS AND DISCUSSION

### *Effect of acidity and reagent concentration on the percentage extraction of U(VI) and Th(IV)*

The effect of acidity on the percentage extraction of U(VI) and Th(IV) with Cyanex-923 in toluene was studied for the acidic range from 5×10<sup>-5</sup>–1.0 M HBr.

The percentage extraction is the amount of U(VI) or Th(IV) extracted into the organic phase containing Cyanex-923 in toluene. It was found that the extraction of U(VI) and Th(IV) into the organic phase of 0.0025 and 0.001 M Cyanex-923 was quantitative in the acidic range of  $5 \times 10^{-5}$ – $1.0 \times 10^{-4}$  M and  $5 \times 10^{-5}$ – $5 \times 10^{-3}$  M, respectively. Hence, all the extractions of U(VI) and Th(IV) with Cyanex-923 in toluene were performed at a fixed acidic concentration of  $1 \times 10^{-4}$  and  $1.0 \times 10^{-3}$  M HBr solution, respectively (Fig. 1).

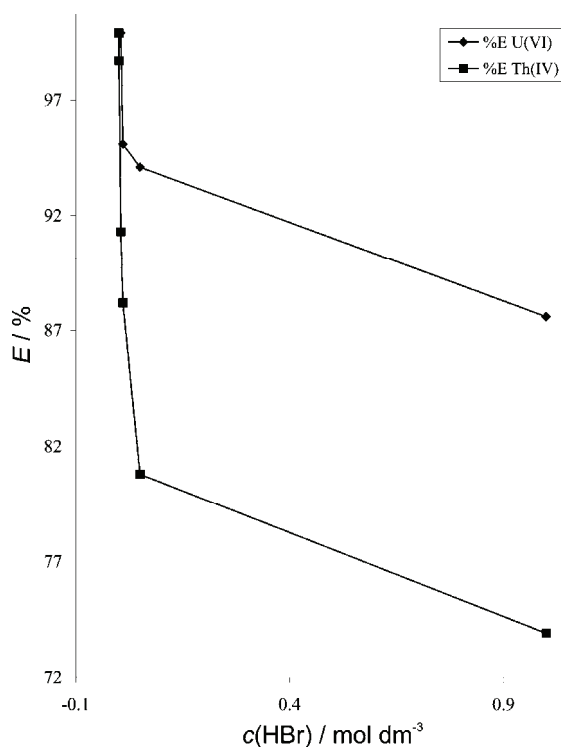


Fig. 1. Effect of acidity on the percentage extraction of U(VI) and Th(IV) with Cyanex-923.

These metal ions were extracted with varying concentrations of Cyanex-923 in toluene from  $5 \times 10^{-5}$ – $5 \times 10^{-3}$  M, while keeping the other parameters, such as pH, period of equilibration, diluent and temperature, constant. The extraction was found to increase with increasing reagent concentration. The extraction of U(VI) and Th(IV) was quantitative with  $2.5 \times 10^{-3}$  and  $1 \times 10^{-3}$  M Cyanex-923, respectively.

#### *Effect of stripping agent*

The metal ions were stripped from the metal-loaded organic phases of Cyanex-923 with different strengths of acids such as HCl, HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub> and HClO<sub>4</sub>. This process is termed percentage recovery. The complete stripping of U(VI) was observed with 7.0 M HClO<sub>4</sub> solution from the organic phase of Cyanex-923.

Th(IV) was stripped quantitatively with 2.0–4.0 M HCl and 1.0–4.0 M HNO<sub>3</sub> solutions (Table I).

TABLE I. Effect of the stripping agent on the percentage extraction of U(VI) and Th(IV) from metal-loaded organic phases of Cyanex-923 in toluene; U(VI): 20 µg; Cyanex-923: 2.5×10<sup>-3</sup> M; acidity: 1.0×10<sup>-4</sup> M; Th(IV): 20 µg; Cyanex-923: 1.0×10<sup>-3</sup> M Acidity: 1×10<sup>-3</sup> M

Acid	Acid concentration, mol dm <sup>-3</sup>							
	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0
U(VI)								
HCl	43.5	42.3	35.5	23.9	14.1	6.7	3.6	1.2
HNO <sub>3</sub>	0.0	4.3	6.1	10.4	20.8	36.2	19.6	16.6
H <sub>2</sub> SO <sub>4</sub>	1.8	3.7	15.9	18.4	–	–	–	–
HClO <sub>4</sub>	31.9	39.3	41.1	46.6	58.8	66.3	99.9	80.5
Th(IV)								
HCl	76.2	99.9	99.9	99.9	–	–	–	–
HNO <sub>3</sub>	99.9	99.9	99.9	99.9	–	–	–	–
H <sub>2</sub> SO <sub>4</sub>	3.9	17.8	27.7	46.5	–	–	–	–
HClO <sub>4</sub>	48.5	62.4	63.4	74.3	77.3	53.5	41.6	38.6

#### Effect of various diluents

The extraction of U(VI) and Th(IV) was performed with Cyanex-923 using various aromatic and aliphatic organic diluents, such as xylene, benzene, chloroform, *n*-hexane, carbon tetrachloride and cyclohexane. Quantitative extraction of U(VI) was observed with all the diluents. In the case of Th(IV), the extraction was quantitative with all solvents except for *n*-hexane and carbon tetrachloride. Toluene was considered the best diluent for the extraction of U(VI) and Th(IV) since it provided better phase separation (Table II).

TABLE II. Effect of various diluents on the percentage extraction of U(VI) and Th(IV) with Cyanex-923; U(VI): 20 µg; Cyanex-923: 2.5×10<sup>-3</sup> M; acidity: 1.0×10<sup>-4</sup> M; Th(IV): 20 µg; Cyanex-923: 1×10<sup>-3</sup> M; Acidity: 1×10<sup>-3</sup> M

Diluent	Percentage extraction ( <i>E</i> / %)	
	U(VI)	Th(IV)
Toluene	99.9	99.9
Cyclohexane	99.9	99.9
<i>n</i> -Hexane	99.9	95.0
Xylene	99.9	99.9
Chloroform	99.9	99.9
Carbon tetrachloride	99.9	97.0

#### Effect of period equilibration

The extraction equilibrium was studied for different periods of shaking ranging from 1–30 min. It was observed that a 1-min shaking period was sufficient for the quantitative extraction of U(VI) and Th(IV) with Cyanex-923 in toluene,

but there was no adverse effect when the extraction period was extended up to 30 min.

#### *Nature of the extracted species*

It was necessary to evaluate the distribution ratio ( $D$ ) while varying the extractant concentration to ascertain the nature of the extracted species. The distribution ratio is the ratio of the concentration of solute in the organic phase to the concentration of the solute in the aqueous phase. Therefore, the composition of the extracted species was first ascertained from graphs of  $\log D$  vs.  $\log R$  at a fixed HBr concentration of  $1.0 \times 10^{-4}$  and  $1.0 \times 10^{-3}$  M (Fig. 2). The slopes obtained were 1.92 and 1.83 respectively for U(VI) and Th(IV), indicating that two molecules of ligand react with one molecule of U(VI) and Th(IV). Hence, the probable ratio of the extracted species of U(VI) and Th(IV) with Cyanex-923 was found to be 1:2. Therefore, the probable extracted species of U(VI) and Th(IV) in the organic phase are  $\text{UO}_2\text{Br}_2 \cdot 2\text{Cyanex-923}$  and  $\text{ThBr}_4 \cdot 2\text{Cyanex-923}$ .

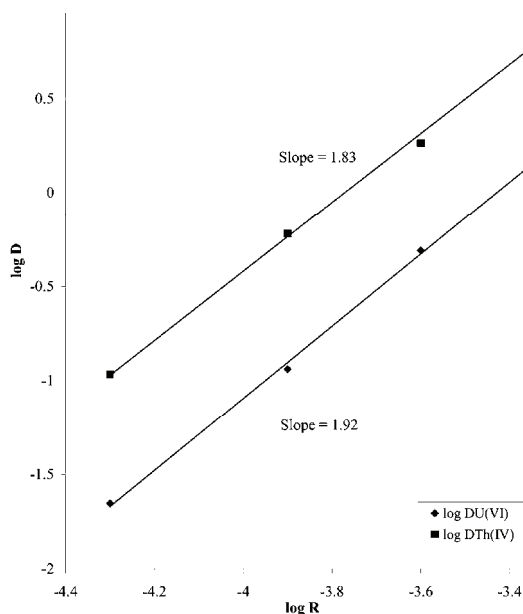


Fig. 2. Effect of reagent concentration on the distribution ratio of U(VI) and Th(IV). ( $\log R = \log [\text{Cyanex-923}]$ ).

#### *Influence of temperature*

Extractions of uranium(VI) and thorium(IV) with  $2.5 \times 10^{-3}$  and  $1.0 \times 10^{-3}$  M Cyanex-923 in toluene at a fixed HBr concentration of  $1.0 \times 10^{-4}$  and  $1.0 \times 10^{-3}$  M were performed at different temperatures (up to 343 K). The distribution ratio decreased with increasing temperature. The Van't Hoff Equation is given by:

$$\log D = - \frac{\Delta H}{2.303RT} + C$$

where  $D$  represents the distribution ratio,  $\Delta H$  is the enthalpy change for the reaction and  $C$  is a constant. The slope obtained from the plots of  $\log D$  vs.  $1/T$  were  $2.45 \times 10^{-3}$  and  $2.77 \times 10^{-3}$  K for the extraction of U(VI) and Th(IV), respectively (Fig. 3). The obtained  $\Delta H$  values were  $-46.9$  and  $-53.0$  kJ mol $^{-1}$ , respectively, indicating that the reaction are exothermic in nature.

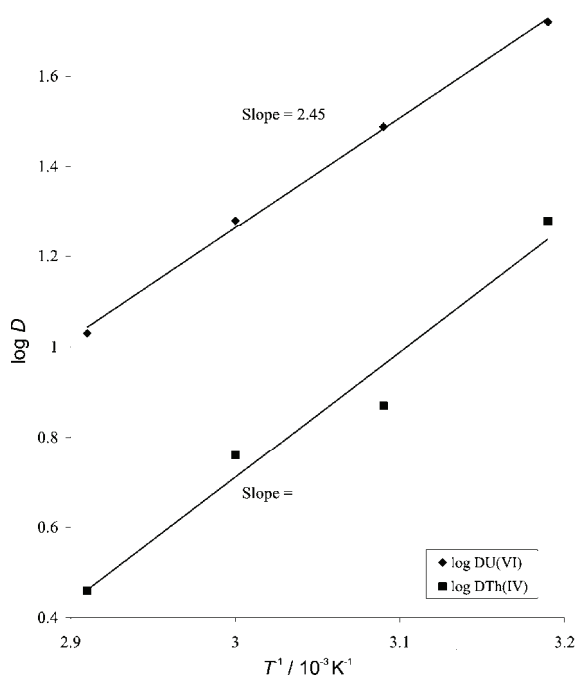


Fig. 3. Effect of temperature on distribution ratio of U(VI) and Th(IV) with Cyanex-923.

#### Effect of diverse ions

The effects of diverse ions on the extraction of U(VI) and Th(IV) by Cyanex-923 in toluene were studied at a fixed HBr concentration of  $1.0 \times 10^{-4}$  and  $1.0 \times 10^{-3}$  M, respectively. The tolerance limit was set so that the error in the percentage recovery was not more than  $\pm 2.0$  %.

In case of U(VI), ions such as thiocyanate, thiourea and  $\text{Zr}^{4+}$  interfere the most with the percentage extraction of U(VI), while ions such as  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Cs}^+$ ,  $\text{Pb}^{2+}$ ,  $\text{Rb}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Ba}^{2+}$  and  $\text{Sr}^{2+}$  are tolerated to a greater extent. In case of Th(IV), ions such as thiocyanate, thiourea,  $\text{Al}^{3+}$ ,  $\text{Be}^{2+}$  and  $\text{Zr}^{4+}$  interfere to the maximum in the percentage extraction of Th(IV), while  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Cs}^+$ ,  $\text{Rb}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$  and  $\text{SO}_3^{2-}$  are tolerated to a larger extent. The other ions which show intermediate effects are indicated in Table III.

TABLE III. Effect of various diverse ions on the percentage extraction of U(VI) and Th(IV) Cyanex-923 in toluene; U(VI): 20 µg; Cyanex-923:  $2.5 \times 10^{-3}$  M; Acidity:  $1.0 \times 10^{-4}$  M; Th(IV): 20 µg; Cyanex-923:  $1.0 \times 10^{-3}$  M; acidity:  $1.0 \times 10^{-3}$  M

Uranium(VI)						
1:32	1:27	1:22	1:17	1:10	1:6	Strongly interfere
Na <sup>+</sup> , K <sup>+</sup> , Cs <sup>+</sup> , Pb <sup>2+</sup> , Rb <sup>+</sup> , Mg <sup>2+</sup> , Ca <sup>2+</sup> , Ba <sup>2+</sup> , Sr <sup>2+</sup>	Bi <sup>3+</sup> , Zn <sup>2+</sup> , Fe <sup>3+</sup> , Cr <sup>3+</sup>	Mn <sup>2+</sup> , Co <sup>2+</sup> , Cu <sup>2+</sup> , Cd <sup>2+</sup> , Pd <sup>2+</sup> , Os <sup>8+</sup>	Rh <sup>3+</sup> , Au <sup>3+</sup> , Ru <sup>3+</sup>	V <sup>5+</sup> , Mo <sup>6+</sup> , Pt <sup>2+</sup> , Ce <sup>3+</sup> , La <sup>3+</sup>	Citrate, Oxalate	Thiocyanate, thiourea, Zr <sup>4+</sup>
Thorium(IV)						
Na <sup>+</sup> , K <sup>+</sup> , Cs <sup>+</sup> , Rb <sup>+</sup> , Mg <sup>2+</sup> , Ca <sup>2+</sup> , Ba <sup>2+</sup> , Sr <sup>2+</sup>	Mn <sup>2+</sup> , Co <sup>2+</sup> , Cu <sup>2+</sup> , Cd <sup>2+</sup>	Cr <sup>3+</sup> , Fe <sup>3+</sup> , Zn <sup>2+</sup> , Bi <sup>3+</sup>	Pt <sup>2+</sup> , Pd <sup>2+</sup> , Os <sup>8+</sup> , Ru <sup>3+</sup>	Ce <sup>3+</sup> , La <sup>3+</sup>	SO <sub>3</sub> <sup>2-</sup> , Cl <sup>-</sup> , Γ, Br <sup>-</sup> , citrate,	Thiocyanate, thiourea, Al <sup>3+</sup> , Be <sup>3+</sup> , Zr <sup>4+</sup> , V <sup>6+</sup> oxalate

#### Mutual separation of uranium(VI) and thorium(IV) with Cyanex-923

The proposed method provides mutual separation of U(VI) and Th(IV). U(VI) was separated mainly from Th(IV) and various other metal ions. This was enabled by exploiting the differences in their respective extracting and stripping conditions. In order to separate U(VI) and Th(IV), the difference in their stripping agents was exploited.

A mixture of U(VI) (20 µg) and Th(IV) (20 µg) was taken and the acidity of the solution was adjusted to  $1.0 \times 10^{-4}$  M. The final volume was made up to 10 ml by the addition of double distilled water. It was then equilibrated with an equal volume of  $1.0 \times 10^{-3}$  M Cyanex-923 in toluene by shaking it in a separating funnel for 10 min at room temperature, whereby both the metal ions were extracted simultaneously. Taking advantage of the difference in the stripping agents, both the metal ions were separated. Th(IV) was stripped first with 1.0 M HNO<sub>3</sub> followed by U(IV) with 7.0 M HClO<sub>4</sub>.

Separation of U(VI) from Th(IV) was possible. In addition, U(VI) was separated from a mixture containing Be(II), Ce(IV), La(III) and Zr(IV), while Th(IV) was separated from a mixture containing La(III), Zr(IV) and Ce(IV) (Table IV).

TABLE IV. Separation of U(VI) and Th(IV) from multicomponent mixtures with Cyanex-923 in toluene; Acidity:  $1.0 \times 10^{-4}$  HBr; Extractant, Cyanex-923:  $1.0 \times 10^{-3}$  M

No.	Metal ion	Amount taken, µg	Stripping agent	Recovery, %
1.	U(VI)	20	8.0 M HClO <sub>4</sub>	99.5
	Th(IV)	20	1.0 M HNO <sub>3</sub>	99.3
2.	U(VI)	20	8.0 M HClO <sub>4</sub>	99.5
	Be (II)	50	1.0 M NaOH	99.2
3.	La(III)	50	2.0 M HCl	99.2
	U(VI)	20	8.0 M HClO <sub>4</sub>	99.5

TABLE IV. Continued

No.	Metal ion	Amount taken, $\mu\text{g}$	Stripping agent	Recovery, %
4.	U(VI)	20	8.0 M $\text{HClO}_4$	99.5
	Th(IV)	20	1.0 M $\text{HNO}_3$	99.3
	Zr(IV)	20	0.5 M NaF	98.8
5.	U(VI)	20	8.0 M $\text{HClO}_4$	99.5
	Th(IV)	20	1.0 M $\text{HNO}_3$	99.3
	Ce(IV)	50	Unextracted	99.1
6.	La(III)	50	2.0 M HCl	99.2
	Th(IV)	20	1.0 M $\text{HNO}_3$	99.3
	Ce(IV)	50	Unextracted	99.1

## CONCLUSIONS

The U(VI) and Th(IV) were extracted quantitatively in the acid concentration range  $5.0 \times 10^{-5}$ – $1.0 \times 10^{-4}$  M and  $5.0 \times 10^{-5}$ – $5.0 \times 10^{-3}$  M, respectively. The minimum reagent concentration required for the complete extraction of U(VI) and Th(IV) was  $2.5 \times 10^{-3}$  and  $1.0 \times 10^{-3}$  M in toluene, respectively.

A thermodynamic study of the extraction reaction of U(VI) and Th(IV) with Cyanex-923 in toluene was also performed. It revealed that the extraction reaction is exothermic in nature with a value of the enthalpy change of  $-46.9$  and  $-53.0$   $\text{kJ mol}^{-1}$ , respectively.

Quantitative extraction of U(VI) and Th(IV) was not possible from  $\text{HNO}_3$  media using 4-benzoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-thione (HBMPPT). The addition of petroleum sulphoxides (PSO) was required for significant improvement in the extraction of U(VI) and Th(IV).<sup>13</sup> Cyanex-923 does not require the addition of any other extractant for the quantitative extraction.

The extraction of Th(IV) was possible with 1-phenyl-3-methyl-4-benzyl-5-pyrazolone in 1–1.5 M HCl only when ascorbic acid and ammonium oxalate were used for the extraction,<sup>14</sup> while complete extraction of Th(IV) was possible with Cyanex-923 without a salting out agent.

Tri-*n*-octylphenoxide was used for the separation of Th(IV) and U(VI), but for complete separation, the use of ascorbic acid solution together with an  $\text{NH}_4\text{I}/\text{HCl}$  mixture was required.<sup>15</sup> With the proposed method, complete separation of U(VI) and Th(IV) was realized without the addition of such mixtures.

Extraction of U(VI) with Primene JMT required a minimum reagent concentration of 0.3 M in benzene,<sup>16</sup> while Cyanex-923 required a lower concentration, *i.e.*,  $2.5 \times 10^{-3}$  M.

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## ИЗВОД

## ЕКСТРАКЦИЈА И РАЗДВАЈАЊЕ U(VI) И Th(IV) ИЗ БРОМОВОДОНИЧНЕ КИСЕЛИНЕ ПОМОЋУ CYANEX-923 ЕКСТРАКТАНТА

SNEHAL M GHAG и SURESH D PAWAR

Department of Chemistry, Siddharth College of Arts, Science and Commerce, Dr. D. N. Road, Fort,  
Mumbai – 400 001, India

У раду је систематски испитивана екстракција уранијума(VI) и торијума(IV) из бромоводоничне киселине коришћењем неутралног фосфин-оксида Суапех-923 у толуену као средству за екстракцију. Нађено је да се ови метални јони квантитативно екстрахују са Суапех-923 у толуену у осегу концентрације киселине  $5,0 \times 10^{-5}$ – $1,0 \times 10^{-4}$  и  $5,0 \times 10^{-5}$ – $5,0 \times 10^{-3}$  М, респективно, и да се из органске фазе одвајају са 7,0 М HClO<sub>4</sub> и 2,0–4,0 М HCl, респективно. Испитиван је ефекат дужине успостављања равнотеже, разблаживања, других јона и средства за одвајање на екстракцију U(VI) и Th(IV) и одређен стехиометријски састав екстрахованих врста. Екстракција се одиграва солватацијом, а вероватне екстраховане врсте нађене у органској фази су UO<sub>2</sub>Br<sub>2</sub>·2Суапех-923 и ThBr<sub>4</sub>·2Суапех-923. На основу ових резултата развијен је и секвенционални поступак за њихово раздвајање.

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