



Liquid–liquid extraction of uranium(VI) from weak sodium acetate medium using 2-octylaminopyridine: real sample analysis

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Abstract

The analytical technique has been developed for the extraction and determination of uranium(VI). This process is based on the ion-pair complex formation of uranium(VI) with 2-octylaminopyridine in xylene. Uranium(VI) is quantitatively extracted by optimizing the parameters of solvent extraction in presence of sodium acetate, such as pH, concentration of weak organic acids, strippant, shaking period, solvent study, and concentration of extractant. A mechanism of extraction was proposed based on the slope ratio analysis method. The interference of various cations and anions was also investigated. The versatility of the developed method was investigated by employing it to binary and ternary mixtures. The robustness of the method was demonstrated by determining uranium(VI) in the bone sample.

Keywords Solvent extraction · Uranium(VI) · 2-octylaminopyridine · Sodium acetate · Bone sample

Introduction

Uranium is the most vital element for nuclear energy [1]. It is universal that uranium is toxic and is being radioactive; thus the safety profiles for uranium compounds are well established [2]. Hence it is one of the elements for which world health organization (WHO) norms are most stringent (30 µg/L) in drinking water. This is because uranium is a relatively contaminating element in many surface or near-surface environments, its geological exploration requires the measurement of trace quantities of metal ions in water and other samples [3–5]. The natural source for uranium is monazite which contains a sizeable amount of uranium. Consequently, the separation purification of uranium is of great practical importance. The methodology adopted for extraction and purification from ore and nuclear fuel reprocessing has always attracted the attention of separation

scientists. The efficient and selective extraction of uranium from aqueous solution has been a subject of significant interest because of the global shortage of uranium sources and radioactive contaminants in the environment, soil, water in which versatile new materials that can separate efficiently desired ore [6–8].

A large number of modern analytical tools have been available for the determination of uranium in a wide variety of samples, The increasing availability of powerful instrumental techniques such as neutron activation analysis (NAA), energy dispersive X-ray fluorescence (EDXRF), inductively coupled plasma atomic emission spectrometry (ICP-AES); inductively coupled plasma emission mass spectrometry (ICP-MS) has enabled the analysis of complex mixtures with high accuracy and precision [9–14]. Even though these techniques provide high sensitivity and favorable detection limit, their application requires rather expensive equipment and leads to higher running cost [15]. Generally, solvent extraction and spectrophotometric technique are widely used to separate and determine uranium(VI) [16–20].

In this context, attentions have been given for the development of soft donor complexing agents as extractants for Ln/An separation. Relatively soft donor atom such as nitrogen prefers 5f elements than 4f elements, providing the higher selectivity than harder oxygen atoms. Concerning this high molecular weight, amines act as better extractant

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to oxygen-containing extractants like tri-*n*-butyl phosphate (TBP), trioctyl phosphine oxide (TOPO), and dioctyl sulfoxide (DOSO). This difference allows for preferential coordination with actinides (5f) over lanthanides. Hence amines with soft nitrogen atoms are a subject of extensive research as a potential extractant of actinides from high-level liquid waste (HLLW) [21]. The literature survey demonstrates that the solvent extraction of uranium(VI) can be carried out by various extractants like tri-*n*-butyl phosphate, trioctyl phosphine oxide and dioctyl sulfoxide [22], cyanex-272 [23]. This manuscript describes the extraction and separation of uranium(VI) from bone sample and associated elements using 2-octylaminopyridine (2-OAP) in sodium acetate medium. The advantage of this method over the earlier method is that extraction is in acetate medium which is a relatively greener approach than the earlier where extraction was carried out in mineral acids. The concentration of 2-OAP is moderate and it is synthesized in a laboratory at a low cost. The method is rapid, simple, and applicable for extraction and determination of uranium(VI) from binary, ternary mixture and bone sample.

Experimental

Apparatus

Digital spectrophotometer optimized α was used for the absorption measurement using 1 cm quartz cells. An Elico digital pH meter model LI-127 is used to measure the pH. All weighing operations were carried out by METTLER TOLEDO analytical single pan balance model ML-204/-01 having accuracy 1×10^{-4} g.

Reagents

Standard uranium(VI) solution

A solution of uranium(VI) (1 mg mL^{-1}) was prepared by dissolving 2.109 g of uranyl nitrate hexahydrate [$\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$]; (Analytical R. grade, BDH, Poole, UK) in water containing one mL of conc., HNO_3 . The solution was diluted with water to one liter in a volumetric flask. Standardized by precipitating ammonium diuranate and ignited to U_3O_8 , and prepared working solution by appropriate dilution in 0.01 M HNO_3 [24].

2-Octylaminopyridine (2-OAP)

2-OAP was synthesized by the Borsch and Petrukhin method [25] and the working extractant solution having molarity (0.05 M) was prepared in xylene.

Buffer solution: pH 8.6

Dissolved 2 g of boric acid and 2 g of potassium chloride in water followed by 6.5 mL of 1 M NaOH and diluted to 500 mL.

Arsenazo-I

Arsenazo-I (0.05% w/v) was prepared by dissolving 0.05 g of arsenazo-I (s. d. Fine –chem. limited) in water.

All reagents and metal salts used are of analytical grade and their solutions were prepared in water and mineral acid. Double distilled water was used throughout the experiment.

Recommended procedure

A solution containing 200 μg uranium(VI), was mixed with 0.01% w/v sodium acetate and pH was adjusted to 4.0 with dil. HCl and NaOH by maintaining total dilution volume to 25 mL and then transferred to 125 mL separatory funnel. 10 mL 0.05 M 2-OAP in xylene as an extractant was added in separatory funnel and equilibrated for 5 min, two phases were allowed to separate. The uranium(VI) extracted in the organic phase was back-extracted with 3 M HCl (3×10) mL as a strippant solution.

The stripped solution containing uranium(VI) was evaporated to moist dryness, then 0.5 mL of conc. perchloric acid was added to decompose organic matter and diluted to 30 mL with water. Again it was evaporated to moist dryness and extracted into 30 mL of water. The pH was adjusted to 4.0 and transferred it into 50 mL volumetric flask, 4 mL 0.05% w/v arsenazo(I) solution was added and followed by 10 mL of buffer solution (pH 8.6) and diluted up to the mark. The absorbance was measured at 600 nm against a reagent blank [27].

Results and discussion

Effect of pH

The extraction of uranium(VI) was carried by 10 mL of 0.05 M 2-OAP in xylene in the presence of 0.01 M sodium acetate from pH 1–10. The study elucidates that the quantitative extraction was achieved in the pH range 3.8–4.8 (Fig. 1). With an increase in pH, the extraction decreases because, at higher pH, uranium(VI) in acetate medium will form a less stable ion-pair complex with 2-OAP. The pH 4.0 was selected for further extraction.

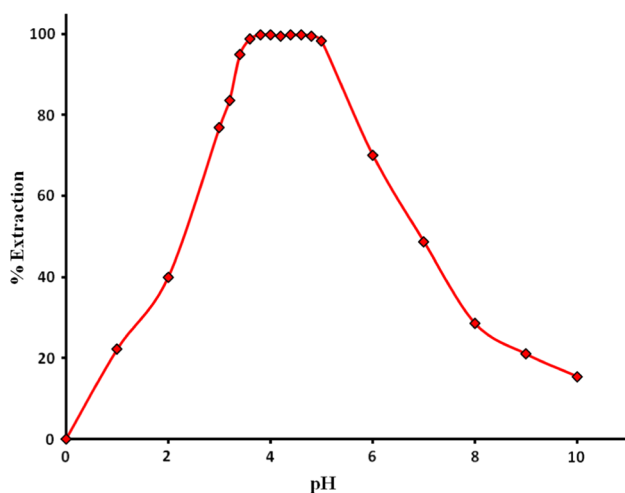


Fig. 1 Effect of pH on extraction of U(VI)-2-OAP complex. Uranium(VI)=200 μg , sodium acetate=0.01%, 0.05 M 2-OAP=10 mL, solvent=xylene, aqueous/organic = 2.5 / 1

Impact of weak organic acid concentration

The distribution ratio (D) of uranium(VI) was investigated at pH 4.0 with 10 mL of 0.05 M 2-OAP in xylene in the presence of varying concentrations of different weak organic anions like acetate, succinate, malonate, and citrate. Quantitative extraction of uranium(VI) was found from acetate and succinate media. However, acetate gives more reproducible results, and more importantly it is greener, cost-effective. The weak acid curve of sodium acetate indicates that quantitative extraction was taking place in the conc. range of 0.008 to 0.011 M. In general procedure 0.01 M sodium acetate was recommended throughout the experiment (Fig. 2). The salicylate, malonate, citrate, and oxalate do not give quantitative extraction of uranium(VI) as there was no formation of stable ion-pair complexes.

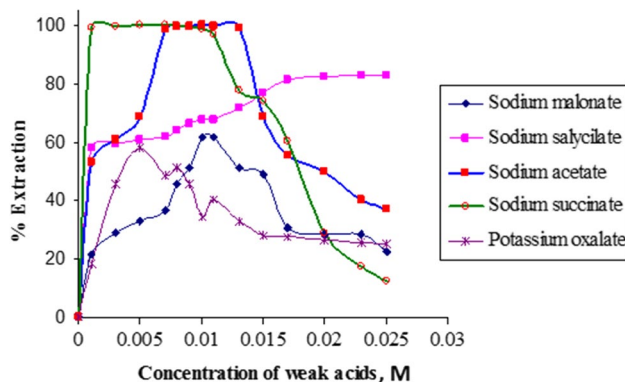


Fig. 2 Effect of weak acids concentration on extraction of U(VI)-2-OAP ion-pair complex. Uranium(VI)=200 μg , pH=4.0, 0.05 M 2-OAP=10 mL, solvent=xylene, aqueous/organic = 2.5/1

Effect of 2-OAP concentration

The concentration of 2-OAP was studied in a concentration range 0.001 to 0.5 M to optimize extraction conditions. The study reveals that extraction increases up to 0.035 M and remained constant up to 0.06 M. However further increase in reagent concentration, extraction decreases this might be due to the formation of stable 2-OAP-acetate species. Therefore for quantitative extraction 10 mL 0.05 M of 2-OAP was used throughout the experiment (Fig. 3).

Solvent study

The use of a suitable solvent is very vital in solvent extraction. The various solvents were studied such as amyl alcohol, 1,2-dichloroethane, dichloromethane, xylene, nitrobenzene, n-butanol, kerosene, methyl-*isobutyl*ketone, chloroform, toluene, benzene (Table 1). The extraction of uranium(VI) was found to be quantitative in xylene and nitrobenzene with 0.05 M 2-OAP. It was found that there is any significant relation between dielectric constant and percentage extraction, xylene was selected as solvent for extraction of uranium(VI) which has low cost and showed clear phase separation.

Influence of stripping agents

Stripping is the reverse of extraction or back extraction which directly affects the extraction of uranium(VI). The back extraction uranium(VI) from the loaded organic phase was performed with stripping agents such as HNO_3 , HCl , CH_3COOH , NH_3 , NaOH for the quantitative extraction of uranium(VI) with 10 mL of 0.05 M 2-OAP. There was quantitative recovery of uranium(VI) with HCl

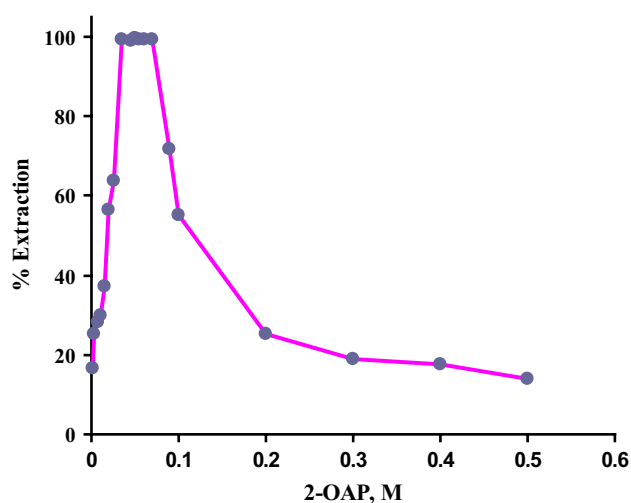


Fig. 3 Effect of 2-OAP concentration on extraction of uranium(VI). Uranium(VI)=200 μg , pH=4.0, sodium acetate=0.01%, solvent=xylene, aqueous/organic = 2.5/1

Table 1 The solvent study for quantitative extraction of U(VI)

Solvent	Dielectric constant, ϵ	Percentage extraction, %E	Distribution ratio, D
Amyl alcohol	13.90	48.7	2.37
1,2-Dichloroethane	10.50	54.1	2.95
Dichloromethane	9.08	64.9	4.63
Xylene	2.30	100	∞
Nitrobenzene	34.82	94.8	45.95
<i>n</i> -Butanol	17.80	34.0	1.29
Kerosene	1.80	67.5	5.20
Methyl <i>isobutyl</i> ketone	13.11	84.5	6.51
Chloroform	4.40	63.9	4.43
Toluene	2.38	79.4	9.62
Benzene	2.28	44.5	4.96

in the range 2.5–4 M. Hence 3 M HCl, was recommended for further study (Fig. 4). The stripping mechanism of uranium(VI) is represented.

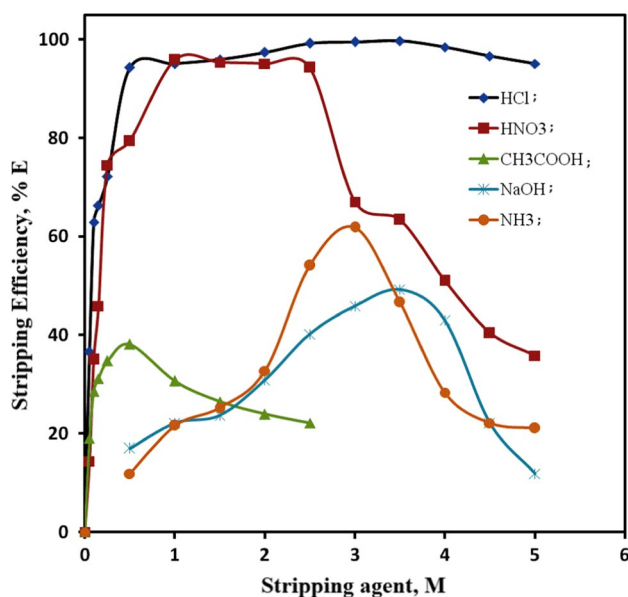
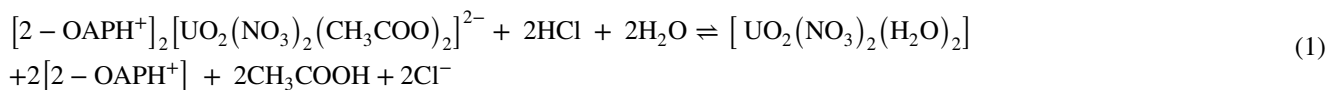


Fig. 4 Influence of stripping agent on extraction of uranium(VI). Uranium(VI)=200 μg , pH=4.0, 0.05 M 2-OAP=10 mL, solvent = xylene, aqueous/organic = 2.5/1

Effect of organic to aqueous volume ratio

The different volumes of aqueous phase to organic phase was investigated by keeping organic phase volume constant. The investigation was carried out in the range of 1:1–1:30. It was found that 1:1–1:5 ratios give quantitative extraction of uranium(VI). Beyond 1:5 ratio the distribution ratio decreases because of lack of 2-OAP extractant due to an increase in volume (Fig. 5). Therefore 1:2.5 ratio of organic to aqueous was recommended for the proposed method for practical suitability to avoid the losses of chemicals.

Metal loading capacity

Extraction of uranium(VI) was examined as a function of metal loading capacity at various concentration of uranium(VI) in the range of 100–1500 μg per 10 mL. The quantitative extraction took place in the range 100–900 μg . It means that to extract up to 900 μg of uranium(VI), 10 mL 0.05 M 2-OAP was enough and thereafter extrac-

tion decreases. Illustrating that there was less availability of 2-OAP. Therefore the loading capacity of 10 mL of 0.05 M 2-OAP was 900 μg of uranium(VI).

Stoichiometry of extracted species

The extraction mechanism of uranium(VI) was proposed by examining the experimental data and based on the slope

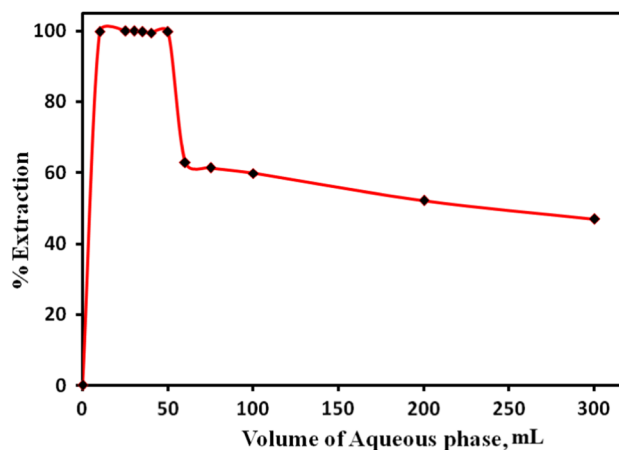


Fig. 5 Effect of organic to aqueous volume ratio on extraction of uranium(VI). Uranium(VI)=200 μg , pH=4.0, 0.05 M 2-OAP=10 mL, solvent = xylene, sodium acetate = 0.01%

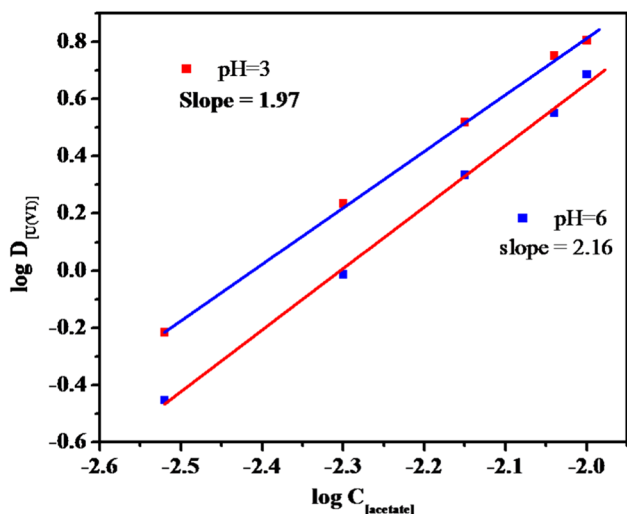


Fig. 6 Stoichiometry of uranium(VI), Plot of Log $D_{[U(VI)]}$ versus Log $C_{[malonate]}$. Uranium(VI)=200 μ g, pH=4.0, 0.05 M 2-OAP=10 mL, solvent = xylene, aqueous/organic = 2.5/1

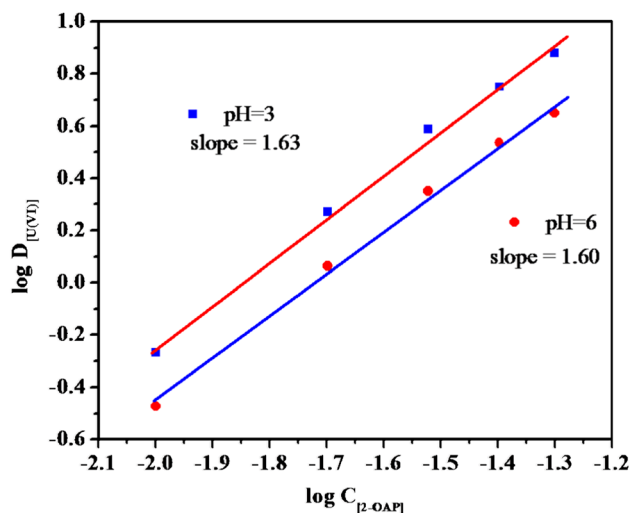
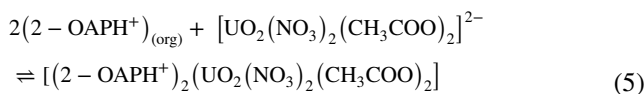
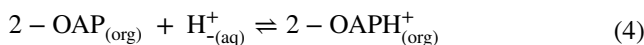
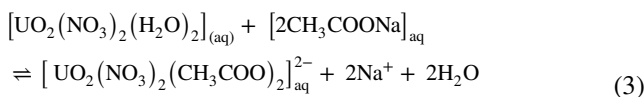
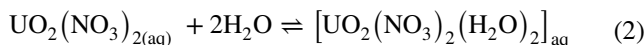


Fig. 7 Stoichiometry of uranium(VI), plot of Log $D_{[U(VI)]}$ versus Log $C_{[2-OAP]}$. Uranium(VI)=200 μ g, pH=4.0, 0.05 M 2-OAP=10 mL, solvent = xylene, aqueous/organic = 2.5/1

ratio analysis method. The plot of $\log D_{[U(VI)]}$ versus $\log C_{[acetate]}$ at fixed pH 3 and 6 was linear with slopes 1.97 and 2.16 respectively. This indicates that two acetate ions react with one UO_2^{2+} species (Fig. 6).

The plot of $\log D_{[U(VI)]}$ versus $\log C_{[2-OAP]}$ at fixed pH 3 and 6 showed a slope 1.63 and 1.60 respectively. Which illustrates that two moles of 2-OAP take part in reaction with one mole of uranium(VI) (Fig. 7). Therefore the slope analysis method proposes the possible composition of species as 1:2:2 (Metal:acetate:2-OAP). The probable mechanism of extraction based on the slope ratio analysis method as follows



The effect of foreign ions

The interference of different foreign cations and anions on the extraction of uranium(VI) was examined with 0.01 M sodium acetate and at pH 4. The study showed that there was a sufficient tolerance limit for anions and cations. However the Th(IV), Zr(IV), Pb(II), Mn(VII) showed interference which was eliminated by the use of sequestering agent and tolerance limit was enhanced to cause allowed $\pm 2\%$ error in the recovery of uranium(VI) (Table 2).

Table 2 The influence of foreign ions on extraction of uranium(VI)

Tolerance limit in mg	Foreign ions added
1	Th(IV) ^a , Zr(IV) ^b , Pb(II) ^c , Mn(II) ^a
2	Co(II), Sn(II), Ca(II), Ni(II), Al(III), Zn(II), Fe(III), Nd(III), Bi(III), Mg(II), Se(IV), Sr(III), Te(IV)
3	Ba(II), Cd(II), V(V), Ag(I), Sb(III), In(III), Ru(III), Ga(III), Hg(II)
4	Mo(VI), Ce(IV), Tl(III), La(III), W(VI), Mn(II), Cu(II)
10	Ascorbate, iodide, malonate, salicylate, tartarate, thiourea EDTA
15	Thiosulphate, chloride, bromide, nitrate, succinate
25	Citrate, thiocyanate, nitrite, sulphate

a = citrate, b = oxalate, c = thiosulphate

Applications

Separation of uranium(VI) from associated metal ions

The versatility of the proposed method was checked by carrying out extraction of uranium(VI) with various associated metal ions such as Zr(IV), Ce(IV), Y(III), Th(IV), La(III), Nd(III), Sm(III), Cd(II), Pb(II), Ba(II) and Ru(III) with 10 mL of 0.05 M 2-OAP in xylene from 0.01 M acetate medium. A very few metal ions like Zr(IV), Th(IV), Pb(II) have interfered which are masked by suitable masking agents. Added metal ion was determined by a reported method using chromogenic reagent spectrophotometrically [24, 26–28] whereas uranium(VI) was determined spectrophotometrically by aresnazo-I method (Table 3).

Separation of uranium(VI) from ternary mixture

A ternary mixture of uranium(VI) with Th(IV), Zr(IV); La(III), Ce(IV); Cd(II), Pb(II); Th(IV), Fe(II); Th(IV), Pd(II); Y(III), La(III); Ru(III), Pd(II); Sm(III), Nd(III); Pb(II), Th(IV); Zr(IV), Ru(III); were prepared and are subjected to extraction by general procedure. Associated metal ions remained in the aqueous phase whereas from loaded organic phase containing uranium(VI) was stripped with 3 M HCl (3 × 10) mL and determined spectrophotometrically by arsenazo-I (Table 4). The ions like Zr(IV) and Th(IV) have interfered but their interference was removed by using a suitable masking agent.

Table 3 Binary mixture separation of uranium(VI) from associated metal ions

Metal ions	Amount taken in µg	Average recovery in %	Relative standard deviation, %	Chromogenic ligand
U(VI)	200	99.7	0.13	Arsenazo—I
Zr (IV) ^a	400	99.1	0.15	Alizarin red—S
U(VI)	200	99.5	0.08	
Ce(IV)	300	99.8	0.09	Formaldehyde
U(VI)	200	99.7	0.10	
Y(III)	75	99.9	0.05	Arsenazo—I
U(VI)	200	99.5	0.12	
Th(IV) ^b	40	99.9	0.08	Arsenazo—III
U(VI)	200	99.7	0.07	
La (III)	250	99.5	0.16	Arsenazo—I
U(VI)	200	99.7	0.12	
Sm(III)	200	99.5	0.11	Arsenazo—I
U(VI)	200	99.7	0.05	
Pb(II) ^c	80	98.4	0.09	PAR
U(VI)	200	99.7	0.13	
Cd(II)	40	99.7	0.16	PAR
U(VI)	200	99.5	0.07	
Ru(III)	400	99.7	0.11	Thiourea
U(VI)	200	99.7	0.07	
Fe(II)	400	99.6	0.16	Ammonium thiocyanate
U(VI)	200	99.7	0.13	
Nd(III)	200	99.7	0.06	Arsenazo—I
U(VI)	200	99.5	0.08	
Pd(II)	250	99.7	0.11	4-ChloroPTPT

*Average of five determinations

a = 10 mg of oxalate, b = 10 mg citrate, c = 10 mg thiosulphate

Table 4 Separation of U(VI) from synthetic mixture

Metal ions	Amount taken in µg	Recovery of U(VI)* %	Relative standard deviation %
U(VI)	200	99.7	0.7
Th(IV)	40		
Zr(IV)	400		
U(VI)	200	99.5	0.13
La(III)	500		
Ce(IV)	500		
U(VI)	200	99.5	0.09
Cd(II)	500		
Pb(II)	500		
U(VI)	200	99.2	0.11
Th(IV)	500		
Fe(II)	500		
U(VI)	200	99.7	0.17
Th(IV)	500		
Pd(II)	500		
U(VI)	200	99.7	0.16
Y(III)	500		
La(II)	500		
U(VI)	500	99.5	0.19
Ru(III)	500		
Pd(II)	500		
U(VI)	200	99.2	0.09
Sm(III)	500		
Nd(III)	500		
U(VI)	500	99.2	0.11
Pb(II)	500		
Th(IV)	500		
U(VI)	200	99.5	0.17
Zr(III)	500		
Ru(III)	500		

*Average five determinations

Determination of uranium(VI) from bone sample

The beauty of the proposed method was that we are successfully separated and determined uranium(VI) from a bone sample with 0.05 M 2-OAP as an extractant. The bone sample was collected carefully and crushed to suitable fineness, dried perfectly by keeping it in an oven at temperature 363 K for about two hours. The sample was cooled and 0.900 g was transferred in beaker, 2 mL conc. perchloric acid was added and followed by 5 mL conc. nitric acid and heated up to moist dryness on hot plate. The residue was extracted in water and diluted to 25 mL with water. One mL of sample solution was employed for the extraction of uranium(VI) by the proposed method and determined uranium(VI) by arsenazo-I method (Table 5).

Conclusion

The solvent extraction of uranium(VI) from weak organic acid media by 2-OAP as an extractant was investigated. The optimum conditions for the extraction of uranium(VI) were investigated. The extraction mechanism was proposed on the basis of slope ratio analysis method. The beauty of proposed method was that it was successfully employed for the determination of uranium(VI) from a synthetic mixture of associated metal ions and bone sample. The method is simple, reliable and reproducible. It does not require sophisticated instrumentation. The extraction of uranium(VI) was carried out from acetate medium, this shows that the method is eco-friendly belong to green chemistry approach.

Table 5 Separation of uranium(VI) from bone sample

Sample	Reported value of uranium(VI) µg /g	Uranium found by proposed* method µg /g	Relative standard deviation %
Animal bone sample	360	359.5	0.008
	360	359.0	0.007
	360	359.0	0.01
	360	359.3	0.006
	360	359.5	0.009

*Average of five determination

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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