

# Spectrophotometric determination of uranium using chromotrope 2R complexes

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**Abstract** A simple, fast and reliable spectrophotometric method for the determination and microextraction of trace amounts of uranium using chromotrope 2R as a chelating agent and 1-butyl-3-methylimidazolium hexafluorophosphate ionic liquid (IL) was used as an extractant solvent. Influence variables such as pH, volume of ligand and IL were inspected by full factorial design. In the view of Pareto chart a contour plot was studied to examine the significant variables and their interactions. The detection limit and the preconcentration factor were found to be 0.87 and  $50 \mu\text{g L}^{-1}$ , respectively. The developed method was successfully applied to ore samples.

**Keywords** Uranium · Chromotrope 2R · Ionic liquid based micro-extraction · Multivariate determination · UV–Vis spectrophotometry

## Introduction

Uranium and its compounds are known as one the dangerous radioactive element [1] and it can be harmful because of its toxicity. It occurs naturally in soil, rock and water at low level and is commercially extracted from uranium-bearing minerals such as uraninite [2]. The quantitative determination of uranium in igneous rocks is of great interest and important for geological investigation

[3]. However the small amount of radioactive element can cause acute toxicological effects in humans and animals [4, 5].

Several methods have been developed for the determination of uranium such as instrumental neutron activation analysis (INAA) [6], inductively coupled plasma mass spectrometry (ICP-MS) [7, 8], capillary zone electrophoresis (CZE) [9], alpha spectrometry [10, 11] and spectrophotometry [12]. Although all these techniques have also some drawbacks such as high level of sensitivity, lack of selectivity, presence of complex matrix, poor precision and accuracy, because it requires well-controlled experimental conditions, and presence of complex matrix.

Spectrophotometric methods are widely used for the determination of trace U, due to its simplicity, rapidity, inexpensive and reliable accuracy [13]. A number of chromogenic reagents were used such as chromazurol [14], 4-(2-pyridylazo) resorcinol [15], 8-quinolinol [16], Arsenazo III [17], and Chromotrope 2R [18].

Multivariate technique has been widely used in analytical chemistry for heavy metal determination. In the multivariate technique a full factorial design (FFD) is well established in preconcentration and separation techniques [19–21].

In this study, an ionic liquid (IL) based dispersive liquid–liquid microextraction (IL-DLLME) procedure has been developed for the determination of uranium in ore sample using chromotrope 2R as a chelating agent. The optimization of procedure was carried out through a multivariate approach. The three factors [pH (P), ligand volume (LV) and IL volume] were selected as effective parameters for the extraction of U with two levels for each one. A FFD was applied to identify the effects of main variables on the % recovery of U and finding out possible interactions between the main factors.

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## Materials and methods

### Apparatus

A Hitachi Model 150–20 UV–Vis double beam spectrophotometer carrying a 10 mm optical path cell was used for the measurement of U. A model Nel pH 900 (Ankara-Turkey) with glass electrodes was used for the pH adjustment.

### Chemicals and reagents

Chemicals and reagents were used of high purity analytical grades (Merck, Darmstadt, Germany). Nitric acid (HNO<sub>3</sub>-65 %), perchloric acid (HClO<sub>4</sub>-60 %), hydrogen fluoride (HF-38–40 %) and 1-butyl-3-methylimidazolium hexafluorophosphate [C<sub>4</sub>MIM][PF<sub>6</sub>] IL were used and obtained from Merck, (Darmstadt, Germany). A stock standard solution (1,000 µg mL<sup>-1</sup>) of U was also purchased (Merck, Darmstadt, Germany). While working standards of corresponding metal ion was prepared from the dilution of stock standard solution with distilled water. A solution of chromotrope 2R (Fluka, Buchs, Switzerland) was prepared with the addition of an appropriate amount of 0.1 g of the reagent in 100 mL distilled water.

### Ionic liquid based dispersive liquid–liquid microextraction procedure

For the preconcentration of U(VI), 20 mL of aliquots containing 100 µg L<sup>-1</sup> U(VI) were taken into 50 mL conical bottom vials. The pH of the solution was adjusted at desired pH value of 8.0 by the addition of HCl (0.01 mol L<sup>-1</sup>) and NaOH (1.0 mol L<sup>-1</sup>). After adjusting of pH, 100–500 µL of LV, and 50–200 µL of IL was added into mixtures. The mixture solutions were diluted up to 20 mL with distilled water and were kept at room temperature for the formation of U chelates. Afterward, the solution was centrifuged for 3 min at 3,000 rpm. After centrifugation, the aqueous layer was decanted and the sample solution containing uranium(VI) was diluted with 400 µL of ethanol and finally was determined by UV–Vis spectrophotometer.

### Analysis of ore samples

The ore samples were collected from Kayseri, Turkey. A 0.2 g of triplicate ore samples were weighed in 100 mL of beaker and added 10 mL of HNO<sub>3</sub> followed by heating on hot plate for 2 h at 90 °C. After that 5 mL of HClO<sub>4</sub> was added to this mixture and then left the sample solution for overnight. Thereafter, HNO<sub>3</sub> (10 mL) and HClO<sub>4</sub> (5 mL) were added into the mixture and was heated overnight at

90 °C. After cooling, the resulting solutions were filtered through a 0.45 µm pore size membrane filter (Millipore Corporation, Bedford, MA, USA) into a 25 mL conical bottom flask and were diluted with distilled water. Afterwards, adjusted pH of digested ore samples to desired pH and then were subjected to developed procedure as described in “IL based dispersive liquid–liquid microextraction procedure” section and subsequent determination of uranium(VI) by UV–Vis spectrophotometer.

### Statistical software

A FFD was used for experimental design analysis and to evaluate the results by using of computer program Minitab 13.2 (Minitab Inc., State College, PA, USA).

### Experimental design

For the optimization of proposed procedure, a two level factorial design was carried out [22, 23]. A FFD was used for sequential experimentation and gives the practical information about the imperative factors. Thus the three most significant factors such as P, IL and LV were evaluated by FFD in which eight experiments were performed to optimize above discussed three variables versus % recovery of U. For further assessment of optimal conditions of variables and their significant interactions were studied by Pareto chart.

## Results and discussion

### Optimization strategy by full factorial design

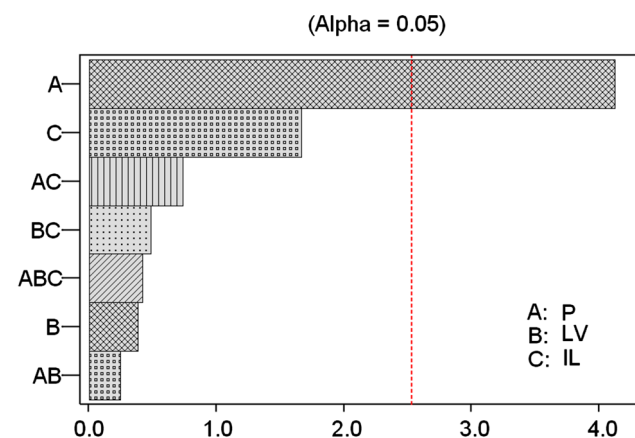
In preliminary experiments, FFD was performed involving 8 experimental runs. The variables at low (–) and high (+) levels are listed in Table 1. This design was used for the screening of the main variables such as P, IL and LV which influencing on the analytical signal of U and estimation of the corresponding response surface by a proposed procedure based on the advantages of standardized testing [24]. From the results of the FFD (Table 2), it was clearly seen that the high P(+), IL and high amount of ligand provided significantly high extraction efficiency for U. In the present

**Table 1** Factors and levels are used in FFD

Variables	Low (–)	High (+)
P	2.0	8.0
Chromotrope-2R chelating agent (LV) (µL)	100	500
IL (µL)	50	200

**Table 2** Design matrix for FFD

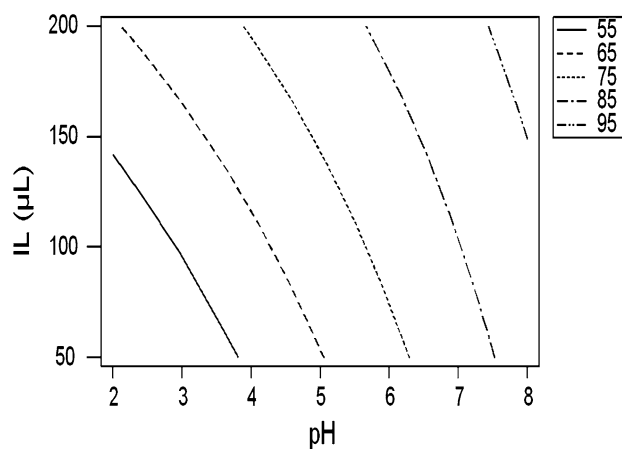
Experiments	P	LV	IL	% Recovery
	A	B	C	
1	–	–	–	35.1 ± 1.1
2	+	–	–	85.6 ± 2.3
3	–	+	–	45.4 ± 1.2
4	+	+	–	92.2 ± 2.5
5	–	–	+	68.1 ± 1.3
6	+	–	+	95.5 ± 2.6
7	–	+	+	60.5 ± 1.4
8	+	+	+	101.0 ± 2.7



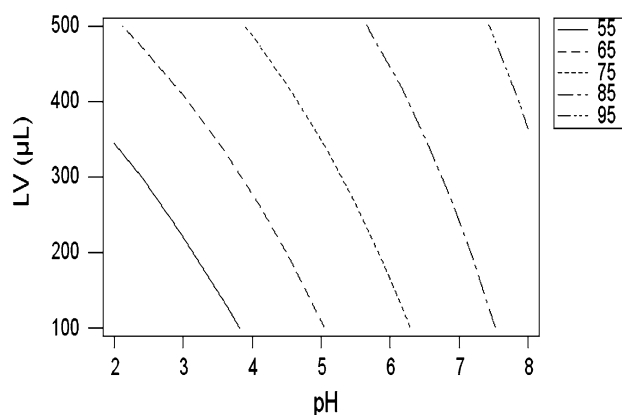
**Fig. 1** Pareto chart of effects versus % recovery of U

study, pH plays a very important role in metal chelate formation and the consequent extraction efficiency of analytes [25–33]. The results showed that the analytical signal of uranium(VI) increased with the increasing of pH and whereas remaining factors were at low (–) level. At high pH, % recovery was achieved 85.6 % and whereas other factors LV and IL were at low level (–) (experiment 2).

The selected three significant variables P, LV and IL (experiment 8) showed optimum % recovery of U at high level of each factors, 8.0, 500 and 200 μL, respectively. Therefore, U(VI) CT-2R complex will become stronger at high pH 8.0. However, in the present study FFD with eight experiments were performed to explore the significant variables and their interactions versus % recovery of U as summarized in Table 2 and their interactions can be visualized by Pareto chart using standardized effect at 0.05 *P* values (Fig. 1). According to the Pareto chart, the effect of P and IL were shown to be most significant at *P* values of 0.05. Since there were eight experiments were performed at two levels for optimization of recovery and estimation of the corresponding response surface. Figures 2



**Fig. 2** Response contour plot for % recovery of U versus P and IL



**Fig. 3** Response contour plot for % recovery of U versus P and LV

**Table 3** Influences of foreign species on the determination of uranium (μg mL<sup>-1</sup>)

Foreign species	Added	Tolerance limit	% Recovery
Ca <sup>2+</sup>	CaCl <sub>2</sub>	2,500	98.8
Cu <sup>+</sup>	Cu(NO <sub>3</sub> ) <sub>2</sub> ·3H <sub>2</sub> O	25	99.3
F <sup>-</sup>	NaF	25	97.4
Fe <sup>3+</sup>	Fe(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O	1,000	97.9
Mg <sup>2+</sup>	Mg(NO <sub>3</sub> ) <sub>2</sub>	1,500	98.8
Na <sup>+</sup>	NaCl	1,500	98.3
Ni <sup>2+</sup>	Ni (NO <sub>3</sub> ) <sub>2</sub>	25	98.8
NO <sub>3</sub> <sup>-</sup>	KNO <sub>3</sub>	1,000	99.3
PO <sub>4</sub> <sup>3-</sup>	Na <sub>3</sub> PO <sub>4</sub> ·12H <sub>2</sub> O	1,000	98.3
SO <sub>4</sub> <sup>2-</sup>	Na <sub>2</sub> SO <sub>4</sub>	1,500	100.7
Zn <sup>2+</sup>	Zn (NO <sub>3</sub> ) <sub>2</sub>	25	99.8

**Table 4** Addition recovery/test procedure for determination of U in ore sample ( $n = 4$ ,  $\mu\text{g g}^{-1}$ )

Added	Found	% Recovery
0.0	$2.29 \pm 0.05$	–
2.0	$4.21 \pm 0.12$	99.4
4.0	$6.25 \pm 0.11$	99.8
8.0	$10.2 \pm 0.17$	98.1

and 3 represented the contour diagram of the estimated response surface. The figure clearly indicates the significance of P and IL at high levels and its interaction.

### Interferences study

Due to the “Matrix Effects” are an important problem in the instrumental detection of analytes at trace levels [31, 34–39], the effect of interference ions on analytical response of U was studied under optimized conditions. The solution containing  $100 \mu\text{g L}^{-1}$  and different concentrations of alkali, alkaline earth metals and some metal ions were prepared separately and then was applied the proposed procedure as illustrated in “IL based dispersive liquid–liquid microextraction procedure” section. The results showed that the proposed method was fairly free from foreign interfering species as shown in Table 3.

### Analytical figure of merit

Under the optimized conditions, the analytical performance of the developed procedure was evaluated. The linear range was  $0.5\text{--}10 \mu\text{g mL}^{-1}$  with the correlation coefficient ( $R$ ) of 0.998. The preconcentration factor (PF) was 50 when sample volume 20 mL and final volume  $400 \mu\text{L}$ . The limit of detection (LOD) was calculated as equivalent of three times of standard deviation of blank signals was  $0.87 \mu\text{g L}^{-1}$ . The limit of quantification (LOQ), ten times of the standard deviation of the blank signals was  $2.90 \mu\text{g L}^{-1}$ .

### Validation of proposed methodology

In order to validation of IL-DLLME methodology, the accuracy of the proposed method was evaluated by standard addition recovery/test method to determine U in ore samples. The proposed procedure was applied on four replicates of ore samples as discussed in “IL based dispersive liquid–liquid microextraction procedure” section. Under the optimized experimental conditions, the spiking of ore samples was performed with a known amount of 2.0, 4.0 and  $8.0 \mu\text{g g}^{-1}$ , respectively. The results are listed in Table 4. A good agreement exists between the results of

**Table 5** Comparison of IL-DLLME procedure with other reported analytical techniques for U determination ( $\mu\text{g L}^{-1}$ )

Techniques	Analytes	PF	LOD ( $\mu\text{g L}^{-1}$ )	References
Spectrophotometry	U(VI)	100	5.0	[40]
Spectrophotometry	U(VI)	325	300	[41]
Spectrophotometry	U(VI)	400	1.0	[42]
ICP-OES	U(VI)	100	0.3	[43]
ICP-AES	U(VI) and Th(IV)	143	6.14	[44]
Spectrophotometry	U(VI)	50	0.87	Present study

PF preconcentration factor, LOD limit of detection

the proposed method and can be applied successfully to real samples.

### Comparison of IL-DLLME with other analytical methodologies

A comparison of developed IL-DLLME procedure with the other reported analytical methodologies is summarized in Table 5 [40–49]. Our method has comparatively low detection limit.

### Conclusion

A two-level FFD was used for the screening of the main variables which affect extraction efficiency of U(VI). Thus the effects of significant variables versus the % recovery of U were evaluated by FFD. Moreover, pareto chart was used to identify important variables and their interactions between the significant variables. The developed procedure was really more effective, rapid, and environmental friendly. This technique provides good repeatability and reproducibility with high LODs and PF and could be successfully applied for determination of U in ore samples without interfering ions.

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