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# Radiochemical Procedure for Measurement Specific Activities of Uranium Isotopes in Asphaltite Bioleaching Liquor by Alpha-Particle Spectrometry

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Keywords	Abstract
Radiochemical Separation Bioleaching Liquor Uranium Measurement Alpha-Particle Spectrometry	An analytical procedure for the alpha-source preparation is investigated in the study to determine uranium radioisotopes specific activities in the bioleaching liquor leach from asphaltite samples taken from the Silopi region in Türkiye. The purpose is to develop a new radiochemical method that can provide the alpha-particle spectrometry requirements. The proposed analytical procedure includes sample preparation, radiochemical purification of uranium from bioleaching liquor, alpha-source preparation on a substrate and alpha-particle spectrometric analysis of isolated uranium. The method is valid for availability using the z-test, relative bias, and relative uncertainty outlier tests. The results show that the proposed analytical procedure is successfully implemented for measuring the activity concentration of uranium with high accuracy and precision in the bioleaching liquor.
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## 1. INTRODUCTION

The rapid increase of nuclear power plants leads to the progressive depletion of high-grade reserves of uranium ores and also increases tailing generation produced by uranium mining activities (Abhilash & Pandey, 2013; Sarkar, 2019). The sustained depletion of high-grade uranium reserves and raised awareness about environmental degradation due to the conventionally extracted methods give rise to the development of a new extraction technique that is efficient, less polluting, and economic to recover uranium from low-grade deposits and secondary sources. Biological leaching is simple, efficient, less polluting, and relatively less expensive including low energy consumption. Therefore this technique is a suitable alternative procedure and rapidly expanding worldwide to leach uranium from low-grade sources (Pal et al., 2010; Abhilash et al., 2013). The micro-organisms used in the bioleaching process catalyze the oxidizing and reducing reaction of uranium along with related metals and hence the uranium solubility. Most uranium deposits consist of a mixture of uranium-bearing minerals in either U(IV) or U(VI) state. The solubility of uranium varies depending on the oxidation state. The oxidation state being U(VI) is the most soluble form of uranium. The bioleaching mechanism of uranium is mainly controlled by the common action of iron (III) and protons produced by the micro-organisms. They use either iron or sulfur as an energy source for their growth. U(IV) is oxidized to the soluble form of uranium by ferric ions, and the rate of oxidation increases when micro-organisms like *Acidithiobacillus ferrooxidans*. Kinetics of uranium leaching occurs much more rapidly when the redox potential (Fe(III)/Fe(II) ratio) of the leaching solution is high (Eisapour et al., 2013).

Asphaltite is a petroleum-derived substance that is classified as a solid aromatic asphaltic oil containing mineral matter primarily sulfates, carbonates, silicates, and sulfides. Variations in oil deposits during migration

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and solidification result in the formation of asphaltic substances (Ballice, 2002; Hiçyılmaz & Altun, 2006; Saydut et al., 2008). Apart from compositions originating from petroleum, these substances are associated with mineral matter consisting of valuable metals like molybdenum, nickel, vanadium, and radioactive elements such as uranium and thorium. Although the abundance of uranium and thorium is in the low range, a cost-effective method of extraction can make asphaltite a viable alternative source. Biological leaching can play a crucial role as a cost-effective method. Hence, the measurement of radionuclide activities in the bioleaching liquor is very important in determining uranium recovery.

The uranium activity of the leaching liquor is measured by alpha-particle spectrometry which is among the most accurate and precise methods for alpha-emitting radionuclides (Aggarwal, 2016). Nevertheless, the spectrometric measurement requires very pure, uniform, and thin alpha sources. Because of competing significant salt contributions, known as "matrix effects," determining the specific activity of uranium in bioleaching liquor is usually exceedingly difficult. These effects are especially troublesome since they strongly depend on the composition of bioleaching solution and mineralogical components in asphaltite.

The study is aimed to develop a radio-analytical technique for evaluating the activity concentration of uranium radioisotopes in the leaching liquor obtained by bioleaching of asphaltite with a mixed culture containing iron oxidizers, sulfur oxidizers, and a few archaeal species. The radiochemical separation of the radionuclide of interest from all other interfering elements, radioactive or non-radioactive in the leaching liquor, is essential before the measurement of alpha sources since the interfering elements create a lower spectral resolution and higher detection limit (Reis et al., 2011; Zhu et al., 2015; Reis & Monterio, 2020).

## 2. MATERIAL AND METHOD

The radio-analytical method is conducted by four main steps; i) source preparation, ii) radiochemical separation, iii) alpha-source preparation, and iv) counting of alpha-source. Every step may and can contribute to the error propagation in the measurement of the specific activity. To overcome the propagation, the study presents improvements in source preparation and radiochemical separation methods to avoid the interfering elements in the alpha spectrum of the bioleaching liquor.

### 2.1. Instrumentation and Calibration

Alpha-particle spectrometry was performed for the measurement of the specific activity of uranium radioisotopes. Uranium sources were counted by a Passivated Implanted Planar Silicon (PIPS) detector with 450 mm<sup>2</sup> of active surface area. An electroplated mixed standard alpha source consisting of 1.67 Bq (5.2%) for <sup>238</sup>U; 1.63Bq (4.9%) for <sup>234</sup>U, 1.91 Bq (5.5%) for <sup>239</sup>Pu, and 1.81 Bq (5.5%) for <sup>241</sup>Am provide by Eckert & Ziegler Isotopes Products was used for the energy calibration of spectrometry.

### 2.2. Reagents and Tracer

The analytical grade reagents taken from Merck were used in this study. The UTEVA chromatographic resin with 100-150 µm was supplied by Eichrom Technologies, Inc. (Darien, Illinois, USA). The yield of the radiochemical separation technique was determined by using <sup>232</sup>U standard reference solution as a tracer. The National Institute of Standards and Technology (NIST) traceable radionuclide, <sup>232</sup>U (SRM 4324B) with 38.22 Bq g<sup>-1</sup> was purchased in the form of UO<sub>2</sub>Cl<sub>2</sub> dissolved in 2M HCl solution. The specific activity of <sup>232</sup>U was reduced to 0.1090 Bq g<sup>-1</sup> ± 0.0085% (k=1) by diluting with 2M HCl.

### 2.3. Sample Description

A mixed culture of acidophilic mesophiles consisting of iron and sulfur oxidizers with a few archaeal species was used to leach uranium from asphaltite. 9K nutrient medium [(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 15.0 g; KCl, 0.5 g; K<sub>2</sub>HPO<sub>4</sub>, 2.5 g; MgSO<sub>4</sub> 7 H<sub>2</sub>O, 2.5g; Ca(NO<sub>3</sub>)<sub>2</sub> 4 H<sub>2</sub>O, 0.05 g for 5L solution] supplemented with 4.5 g/L of Fe<sup>3+</sup> and tetrathionate (K<sub>2</sub>S<sub>4</sub>O<sub>6</sub>) solution is used for the cultivation of micro-organisms and bioleaching of asphaltite samples. The bioleaching process was conducted in the leaching medium containing an iron-free 9K mineral salt medium with mixed microbial culture at 1.0 and 1.5 pH values.

Analyzing two spiked tap water samples provided by the IAEA-Cu-2010-04 ALMERA Proficiency Test Exercise was used to validate the proposed analytical approach. The pH values of water samples were determined by using Opaus ST300-G Model portable pH meter. If needed, the pH of water samples was adjusted to 2.00 by adding concentrated  $\text{HNO}_3$  to ensure that the trace elements and radionuclides were kept in the sample.

## 2.4. Analytical Procedures

### 2.4.1. Sample Preparation

The Eichrom ACW02 (Eichrom, 2014a) analytical process, which is used to separate and analyze uranium in water, was improved to include uranium isotope analysis in the bioleaching liquor. This method was chosen because of its efficiency and reliability for the measurement of actinides in the water. The sample preparation approach and radiochemical separation procedures were previously reported and used in various alpha-particle spectrometry tests (Seferinoğlu et al., 2014). A sub-sample of 50 mL of bioleaching liquor was taken with a volumetric flask. The quantification of radiochemical recoveries and correction of results to improve precision and accuracy was performed with  $^{232}\text{U}$  isotopes as a tracer. A weighed aliquot of  $^{232}\text{U}$  standard solution with an analytical balance calibrated with SI traceable weights was added to the sample at the beginning of the procedure; 0.1045 Bq was put in total. A calcium phosphate precipitation was performed to concentrate the actinides that exist in bioleaching liquor. 0.5 mL of 1.25M  $\text{Ca}(\text{NO}_3)_2$  was added to the liquor and heated with stirring at the medium setting for 60 min. After adding 2 drops of phenolphthalein indicator and 1 mL of 3.2M  $(\text{NH}_4)_2\text{HPO}_4$  solution, enough concentrated  $\text{NH}_4\text{OH}$  was added and heated for another 30 min to co-precipitate uranium with calcium phosphate ( $\text{Ca}_3(\text{PO}_4)_2$ ). The precipitate was cooled to room temperature and allowed to settle until the solvent would be separated by decantation or centrifugation. After discarding the supernatant, the precipitate was washed with an amount of deionized water approximately twice the volume of precipitate and centrifuged again for about 5 or 10 min at 2500 rpm. The residue was dissolved with 5 mL of concentrated  $\text{HNO}_3$  and evaporated to incipient dryness. The residue was re-dissolved with 10 mL of the solution of 3M  $\text{HNO}_3$ -1.0M  $\text{Al}(\text{NO}_3)_3$ . The effect of phosphate on the separation of uranium isotopes from other interfering radioisotopes like neptunium and thorium is very important. The phosphate anion readily complexes with tetravalent actinides such as Th (IV) and Np (IV) ions. The phosphate complexes are not extracted from the UTEVA resin used for isolating uranium isotopes. 1.0 M  $\text{Al}(\text{NO}_3)_3$  is added to the leaching solution for counteracting the phosphate effects. Aluminum effectively ties up the phosphate, so this step prevents phosphate interference with neptunium/thorium uptake by the resin. The radiochemical separation step of Th (IV) and Np (IV) ions from the UTEVA resin was improved by the addition of Al. Another important problem is the iron deposition on the substrate material of alpha-source. The iron present in the liquor is coming from the 9K nutrient medium supplemented with  $\text{Fe}^{3+}$  for the cultivation of micro-organisms and from the pyrite content of asphaltite. The iron deposition should be prevented in the analysis of uranium isotopes because the iron easily contributes to the deposition of uranium on the substrate materials during the electrodeposition process. This increases the thickness of the deposit and inhibits the uranium deposition on the substrate materials. For the counteracting of iron effects, 2 mL of freshly prepared 0.6M ferrous sulphate solution, 1M ammonium thiocyanate indicator, and ascorbic acid should be added to the liquor prior to the radiochemical purification steps of uranium. Hence, three-valent of Fe ions and also tetra-valent of Pu ions are reduced to iron (II) and to plutonium (III) to avoid the retaining of iron and plutonium isotopes on UTEVA resin. The uranium in the solution was then purified by the chromatographic resin.

### 2.4.2. Radiochemical Purification of Uranium

The radiochemical separation step must be performed to isolate the uranium isotopes from other radionuclides with unresolvable alpha energies such as  $^{241}\text{Am}$  and  $^{238}\text{Pu}$ ,  $^{237}\text{Np}$ , and  $^{234}\text{U}$  exist in the liquor with UTEVA resin prior to the measurement. UTEVA resin selectively absorbs the uranium isotopes from interfering elements. The UTEVA resin column placed on a vacuum box has been pre-conditioned by adding 5 mL of 3M  $\text{HNO}_3$  before transferring the sample to the column. The bioleaching liquor was poured into the resin reservoir, and the solution was allowed to drain before being washed with 5 mL of 3M  $\text{HNO}_3$  solution. A 5 mL solution of 8M  $\text{HNO}_3$  was also run through the column. Uranium isotopes with tetravalent ions like Np and Th isotopes were absorbed by the resin, while other interfering elements passed through the column. The resin should be converted from nitrate to the chloride system by adding 5 mL of 9M  $\text{HCl}$  solution for the elution of plutonium,

thorium, and neptunium from the resin. 20 mL of 5M HCl-0.05M oxalic acid solution was added into the column and allowed to drain. This rinse removed any plutonium, thorium, and neptunium retained in the resin. Uranium was finally stripped with 15 mL of 1.00 M HCl solution. The purified uranium fraction was evaporated to dryness.

### 2.4.3. Alpha-Source Preparation and Measurement

Alpha-particle spectrometry requires a uniform and thin source to ensure that the alpha particles are not absorbed by radioactive materials deposited on the substrate and counted with high-resolution spectra. Electrodeposition is one of the most common source preparation methods for radiometric measurement routinely (Crespo, 2012). The Eichrom SPA02 analytical procedure was performed to prepare thin and uniform alpha sources (Eichrom, 2014b). The electrodeposition method with a sulfate system on a stainless steel disc was used in this study. The polished stainless steel disc was used as a substrate for uranium deposits along with a cathode in the electrolytic cell. The evaporated uranium solution was dissolved in the mix solution including 2.5 mL of 5.0 wt% NaHSO<sub>4</sub>, 2.0 mL of deionized water, 5.0 mL of 15 wt% of Na<sub>2</sub>SO<sub>4</sub>, and then added 1.0 mL of ammonium oxalate to the electrolysis cell. After electrodeposition at 0.75 A for 90 min, the reaction was ended by adding 2.0 mL of 25 wt% KOH. The disc was rinsed with NH<sub>4</sub>OH, ethanol, and acetone sequentially. After drying the alpha source at 200°C for 5 min, the count rate of uranium radioisotopes was measured by a PIPS detector.

## 3. RESULTS AND DISCUSSION

The proposed analytical procedure in the study comprises one of the evaluation parameters for determining the uranium recovery from asphaltite by the bioleaching process. The contribution of the leaching liquor is not easily determined by alpha-particle spectrometry due to its matrix effects. The proposed analytical procedure is comprised of four steps: i) sample preparation to get rid of some elements and salt interfering properties during the radiochemical separation including electrodeposition, ii) radiochemical separation to isolate the uranium from other interfering radionuclides such as <sup>237</sup>Np, <sup>238</sup>Pu, and <sup>232</sup>Th on the alpha spectrum, iii) alpha-source preparation on the substrate disc, and finally iv) measurement of alpha-source. Sample preparation and radiochemical steps allow obtaining a pure uranium fraction via chemical and radiochemical ways. One of the main troubles is the formation of the phosphate ions and tetravalent actinides (especially Np and Th radionuclides) complexes. The complexes are easily up taken by UTEVA resin and are not extracted from the column. This can be counteracted via adding Al in the sample separation step because Al is selectively bounding with phosphate anion. Another difficulty is the iron deposition during the electrodeposition process. Iron ions contribute to uranium deposition on the substrate materials and cause the inhibition of uranium deposition. Therefore, the reduction of Fe<sup>3+</sup> ions must be reduced to Fe<sup>2+</sup> by ferrous sulphonate before the radiochemical separation. Consequently, the proposed procedure separates radionuclides that can interfere with the measurement of uranium by alpha-particle spectrometry.

### 3.1. Validation of Analytical Procedure

The proposed analytical procedure was inspected by testing two spiked tap water supplied by IAEA-Cu-2010-04 ALMERA Proficiency Test Exercises. The main aim was to elucidate the applicability of the proposed method to the measurement of activity concentration of uranium radioisotopes in the bioleaching liquor obtained from asphaltite. The evaluation of the results was made in terms of the relative bias, z-score, u-test, trueness, and precision. The results for uranium isotopes concentration compared to recommended values given by the proficiency test were shown in Table 1. The relative bias for spike tap water1 (the so-called STW1) and spike tap water2 (the so-called STW2) are found as 0.00%, -6.38% for <sup>234</sup>U, and 2.86%, -6.45% for <sup>238</sup>U, respectively. The value of the u-test parameter was assigned to 2.58 for the 99% confidence level. The u-score values for STW1 and STW2 samples are found as 0.00, -1.29 for <sup>234</sup>U, and 0.19, -1.25 for <sup>238</sup>U, respectively. Overall, the results of relative bias, z-score, and u-score showed that measured specific activities and their uncertainties were within the recommended reference interval. In this study, the measured results were also evaluated according to the acceptance criteria for trueness and precisions.

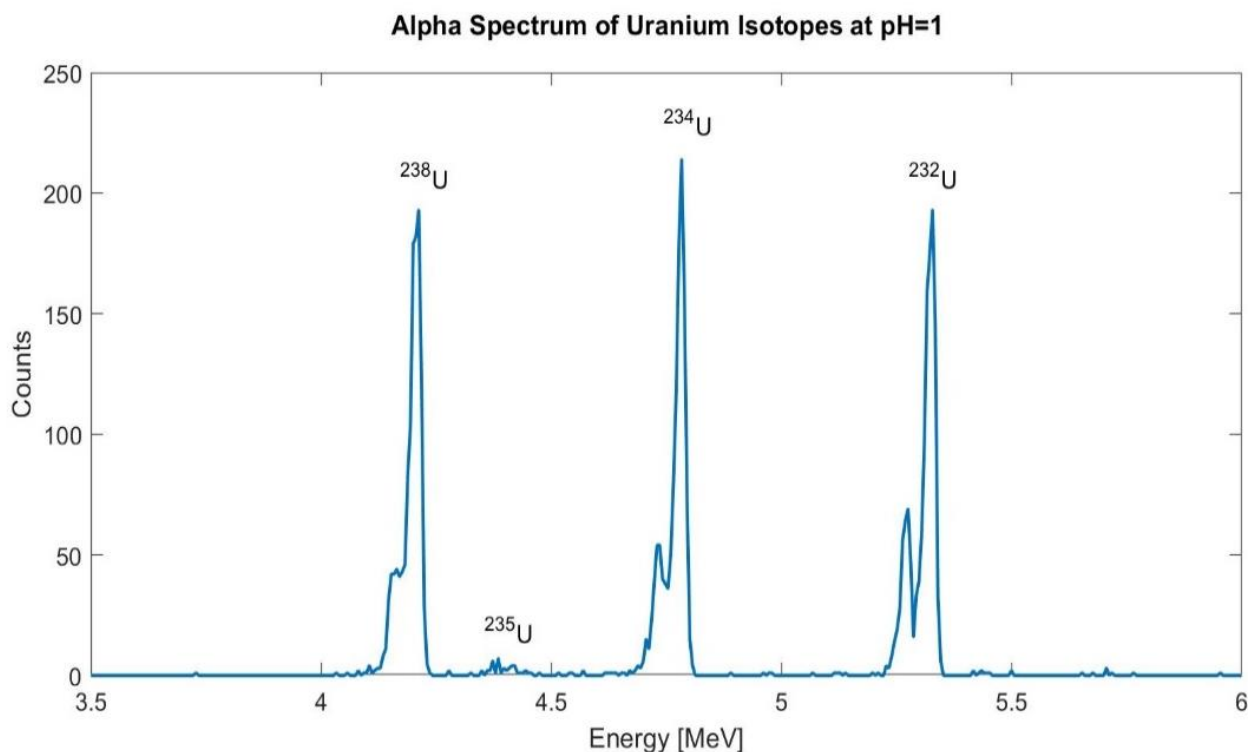
**Table 1.** The measured results of  $^{234}\text{U}$  and  $^{238}\text{U}$  radioisotopes compared to the recommended values of the activity concentration in the spike tap water samples taken from the proficiency test organized by the IAEA

Institution	Sample	Analyte	Measured Value (Unc. k=1) (Bq kg <sup>-1</sup> )	Relative Unc. k=1	Recommended Value (Unc. k=1) (Bq kg <sup>-1</sup> )	Relative Unc. k=1	Relative Bias (%)	Z Score	U Test	A1*	A2	P* (%)	FS*
IAEA-Cu 2010-04 Almera	STW1	$^{234}\text{U}$	1.30 (8)	6.15%	1.30 (3)	2.31%	0.00%	0.00	0.00	0.00	0.22	6.57	A
		$^{238}\text{U}$	0.72 (6)	8.33%	0.70 (2)	2.86%	2.86%	0.06	0.19	0.02	0.16	8.81	A
	STW2	$^{234}\text{U}$	0.44 (3)	7.14%	0.47 (1)	2.13%	-6.38%	-0.13	-1.29	0.03	0.08	7.14	A
		$^{238}\text{U}$	0.29 (2)	7.41%	0.31 (1)	3.23%	-6.45%	-0.13	-1.25	0.02	0.06	7.61	A

The final score is assigned “Acceptable” status if it passed both trueness and precision criteria. The criteria for trueness is passed if the A1 value is smaller than the A2 value. For precision criteria (P), the limited value was determined as 15% for uranium radionuclides. If the P value  $\leq 15\%$ , the precision criteria value passes. The trueness and precision criteria were met, as shown in Table 1. Consequently, the measured specific activities of  $^{234}\text{U}$  and  $^{238}\text{U}$  were found in agreement with recommended values assigned by IAEA.

### 3.2. Determination of Activity Concentration of Uranium in the Bioleaching Liquor

The validated analytical procedure was implemented to determine the specific activity of uranium isotopes in the bioleaching liquor collected from bioleaching of asphaltite at pH values of 1.00 and 1.50. The uranium isotopes spectrum of bioleaching liquor at pH=1.00 was presented in Figure 1. The regions of interest were adjusted between 3.95-4.46 MeV for  $^{238}\text{U}$ , 4.37-4.60 MeV for  $^{235}\text{U}$ , 4.69-4.86 for  $^{234}\text{U}$ , and 5.13-5.39 MeV for  $^{232}\text{U}$ . The spectrum shows that the peak resolution is sufficient (no overlapping peaks) to skip spectral deconvolution calculations. Therefore, the peak area for each radionuclide was straightforwardly determined.

**Figure 1.** The alpha spectrum of the bioleaching liquor collected from the leaching of asphaltite at pH=1



The experiments were done with three replicates. The specific activities and the uncertainties of  $^{234}\text{U}$ ,  $^{235}\text{U}$ , and  $^{238}\text{U}$  isotopes were presented in Table 2. The specific activities at pH=1.00 range between 21.80 (2.80) BqL<sup>-1</sup> and 23.21 (2.59) BqL<sup>-1</sup> for  $^{234}\text{U}$ , 0.71 (0.25) BqL<sup>-1</sup> and 0.86 (0.25) BqL<sup>-1</sup> for  $^{235}\text{U}$ , and 20.50 (2.82) BqL<sup>-1</sup> and 22.07 (2.27) BqL<sup>-1</sup> for  $^{238}\text{U}$ . The specific activities at pH=1.50 were found less than those at pH=1.00.

**Table 2.** The specific activities of uranium isotopes in the bioleaching liquors collected from the asphaltite bioleaching process at pH values are 1.00 and 1.50

Bioleaching liquor	pH Value	Specific Activity of $^{234}\text{U}$		Specific Activity of $^{235}\text{U}$		Specific Activity of $^{238}\text{U}$	
		A (BqL <sup>-1</sup> )	Unc. (BqL <sup>-1</sup> , k=2)	A (BqL <sup>-1</sup> )	Unc. (BqL <sup>-1</sup> , k=2)	A (BqL <sup>-1</sup> )	Unc. (BqL <sup>-1</sup> , k=2)
ABL1	1.00	21.80	2.80	0.71	0.22	20.50	2.82
ABL1	1.00	22.05	2.20	0.85	0.23	21.41	2.45
ABL1	1.00	23.21	2.59	0.86	0.25	22.07	2.27
<b>Mean</b>		<b>22.35</b>	<b>2.53</b>	<b>0.81</b>	<b>0.23</b>	<b>21.33</b>	<b>2.51</b>
ABL2	1.50	14.34	1.39	0.53	0.11	14.50	1.43
ABL2	1.50	14.50	1.45	0.55	0.09	15.10	1.48
ABL2	1.50	15.40	1.48	0.57	0.10	15.70	1.51
<b>Mean</b>		<b>14.75</b>	<b>1.44</b>	<b>0.55</b>	<b>0.10</b>	<b>15.10</b>	<b>1.47</b>

\*ABL1 and ABL2 mean that asphaltite bioleaching liquor at pH=1 and pH=1.50, respectively.

The specific activities were observed in good agreement with each other. No outlier values were found in any of the measurements. The results indicated that the proposed analytical method is applicable to measure a reliable and accurate specific activity of uranium isotopes in the bioleaching liquor by alpha-particle spectrometry.

Radiochemical recovery of the proposed method for  $^{238}\text{U}$  and  $^{234}\text{U}$  isotopes was calculated by the count rate of the tracer ( $^{232}\text{U}$ ). The detector efficiency ( $\epsilon$ ) was determined as 0.150 by the relative solid angle method. The radiochemical recoveries for two isotopes ( $^{234}\text{U}$  and  $^{238}\text{U}$ ) were found in approximately the same range from 60% to 68%.

#### 4. CONCLUSION

The results obtained for two IAEA-Cu-2010-04 ALMERA proficiency test samples indicated that the proposed analytical method is applicable to determine the specific activities of uranium isotopes in the bioleaching liquor matrix with high accuracy and precision. The evaluated results in terms of the relative bias, z-score, and u-score showed the measured activities and uncertainties were in good agreement with reference values assigned by IAEA.

The procedure was tested on bioleaching liquors obtained from the asphaltite bioleaching process at various pH levels. The results of uranium isotopes were in keeping with each other and resulted in no outlier. In the alpha-particle spectrometric measurements, the sample preparation and radiochemical separation stages removed all interfering components, whether radioactive or not. The mean specific activities and their uncertainties for  $^{234}\text{U}$ ,  $^{235}\text{U}$  and  $^{238}\text{U}$  radioisotopes at pH=1.00 were found as 22.35 (2.53) Bq L<sup>-1</sup>, 0.81 (0.23) Bq L<sup>-1</sup>, and 21.33 (2.51) Bq L<sup>-1</sup>, respectively. When the pH value was increased to 1.50, the specific activities were changed to 14.75 (1.44) Bq L<sup>-1</sup> for  $^{234}\text{U}$ , 0.55 (0.10) Bq L<sup>-1</sup> for  $^{235}\text{U}$ , and 15.10 (1.47) Bq L<sup>-1</sup> for  $^{238}\text{U}$ .

Consequently, for samples acquired by bioleaching processes, alpha spectrometry produces a distinctive spectrum. Thus the uranium recovery in the bioleaching processes for low-level uranium resources can be evaluated by the proposed analytical procedure.

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## CONFLICT OF INTEREST

The author declares no conflict of interest.

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