Fabrication of thin uranium source for alpha spectrometry using one shape of Pt-anode

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ABSTRACT

One shape of Pt-anode (a rod of platinum) with a diameter of 6 mm was used, in electrodeposition procedure and studied its effects on chemical yield. A series of initial experiments were managed to evaluate three parameters to determine their significant effect on chemical yield, deposition efficiency. The electrodeposition time was the first of the parameters which studies, optimum time was found to be 80 minutes. The experiments showed that there is an inverse relation between the last two parameters, the deposition diameter and distance between electrodes, especially when this type of anode was used. This means that when the diameter of the deposition is increased, the distance between the electrodes must be reduced to obtain the highest chemical yields. The best diameter was 17 mm, which was the second parameter. The third parameter was the distance between anode and cathode 1 cm was the perfect distance to obtaining of highest chemical yield, which was 90 ± 3%.

KEYWORDS: Uranium, Alpha spectrometry, Electrodeposition, Chemical yield.

1. INTRODUCTION

Uranium element is a naturally radioactive element that is existent in the earth’s crust an average abundance (0.1-20) mg/Kg (2005). Uranium contains three natural isotopes (238U, 235U, 234U), the 238U isotope is the most abundant by weight 99.27% with the 235U and 234U isotopes constituting about 0.72% and 0.0054%, respectively. The human intervention in the use of this element causes an imbalance in the balance of its isotopes. The numerous uses of this element of electric power generation, military uses, especially the use of depleted uranium in the manufacture of armor projectiles produce large amounts of radioactive waste, taking into account the toxicity of radionuclides, which have to continuously control the presence of such waste in the environment, even if at rare levels.

There are many techniques used to measure the concentration of uranium, but is an alpha spectrometry technique of the important techniques in the measurement of uranium concentrations and predicting components. The major advantage of alpha spectrometry is its high sensitivity due to the high-yield alpha decay process, low background and using the chemical separation to eliminate the other possible interferences (Stastna, 2010).

For the success of Alpha spectrometry technique in measuring uranium concentration in low concentrations at high accuracy, uranium should be deposited in the form of a thin and homogeneous layer, where this is the critical and important parameter in the measurement of alpha-isotopes emitters. This parameter is responsible for the important spectral properties like counting statistics and energy resolution (Jobbagy, 2011). The thin and homogenous uranium source can produce very good resolution if the source detection by semiconductor detector (Dumitru, 2013).

As for the subject of uranium electrodeposition, several methods of work have been developed that describe the main parameters responsible for obtaining a thin and homogeneous uranium layer, these parameters: electrodeposition time, type of electrolyte, current density, pH of solution, distance between electrodes and others (Salar Amoli, 2006).

The main goal of the present study is to know the influence of anode shape (rod of platinum) on the main parameters, electrodeposition time, diameter of electrodeposition and distance between electrodes and how these parameters can be employed to obtain good spectral properties and thus verify the attempt to obtain maximum chemical yield.

2. EXPERIMENTAL

Chemicals and backings: All chemicals (NH₄OH, (NH₄)₂SO₄, H₂SO₄, HNO₃) used in this study were analytical grade reagents. The electrolyte solution (NH₄)₂SO₄ was prepared by dissolving 33.035 gm of this material with distilled water and complete the volume to the mark in a volumetric flask 250 ml, concentration of electrolyte solution 1M (Hallstadius, 1984). pH of this electrolyte solution was adjusted between (2.1-2.4), using sulfuric acid and ammonia solution (Maya, 2004).

A stock solution 1000 ppm of uranium was prepared by dissolving 2.109 gm of uranyl nitrate UO₂(NO₃)₂.6H₂O, in distilled water containing 1 ml conc. HNO₃. Dilute the solution with distilled water to mark in a 1-litre volumetric flask (Van Loon, 2013). Several dilutions were done with distilled water lowering uranium concentration to 1 ppm, depending on specific activity of natural uranium these concentrations corresponding to 25.28 Bq/mg. Discs of polished mirror stainless steel were used as sources substrate.
Electrodeposition cell design and backing: The device of the experiments of uranium deposition is a locally manufactured Teflon cell, the diameter of the deposition is 20 mm. This cell is so tight that it does not allow the leakage of liquids while it working containing two electrodes: Cathode or negative electrode is a disk of stainless steel, anode or positive electrode is a platinum rod. The coating solution must immerse both electrodes (Dus, 2011). Fig. 1, represents the schematic diagram of the electrodeposition cell.

Cell preparation and uranium electrodeposition: At first, the cell is tested to prevent liquid leakage by placing water inside it and leaving the cell for 20 minutes to be sure of it. Then it was cleaned with distilled water and acetone. The stainless steel disc before placing inside the cell is also washed with soap to degrease, then with distilled water and acetone.

As it described earlier, three parameters were studied in this work which is as following:

**Time of electrodeposition:** In order to know the effect of this parameter on uranium deposition, thereby of using five different times (20, 40, 60, 80 and 100) minutes (Beesley, 2009). At the beginning of each experiment with certain time, 5 ml of 1 ppm uranium was taken by using mechanical pipette the solution was in 10 ml beaker, and then evaporated the liquid to dryness, the salt was dissolved with of 3-4 ml plating solution (uranium electrodeposition electrolyte) and transferred the solution to a plating cell. Wash the beaker with two additional portions of the plating solution. The total volume should not exceed 15 ml. The wires was Connected to electrodes and switch on the power supply, the current was set 1 Amp. And the deposition diameter 20 mm; after the time expires 1 ml of conc. Ammonia was added, and then switched off current. Disassemble the cell, the disc was washed with distilled water, then with ethanol. The reverse side of the disc was marked with an identification number and the date of electrodeposition. Each planchet in a covered petri dish was stored. The disc using alpha spectrometry was measured.

**Electrodes Distance:** This parameter is the main function of the factors affecting of uranium deposition and chemical yield for the process of precipitation. The experiment procedure was by using four distance between the cathode and anode 0.5, 1, 1.5 and 2 cm (Jobbagy, 2012). At the beginning of each experiment with certain distance, the experiment was carried out as mentioned in section (Time of electrodeposition), deposition time 80 minute and deposition diameter 17 mm.

**Deposition Diameter:** To study this parameter was used four different diameters of the deposition 11, 14, 17 and 20 mm. At the beginning of each experiment with certain diameter, the experiment was carried out as mentioned in section (Time of electrodeposition); deposition time 80 minutes and the distance between the electrodes 1 cm. Voltage starts near 9 V DC and gradually decreases to about 6.5 V.

**Measurement set-up:** Alpha-spectrometer (Alpha Analyst Integrated Alpha Spectrometer, Canberra) is a device used for measuring the alpha particles emitted from radioactive elements. Which contains a vacuum chamber connected to the rotary vacuum pump, the chamber contains a set of shelves (for carrying source) and semiconductor detector (PIPS detector +60 volt, activating area 450 mm²), coupled with low noise preamplifiers, amplifiers, and a multichannel analyzer. The energy resolution (FWHM) of the measurements was up to ~ 25keV and the counting efficiency for the detector was estimated by means of calibration standards to be ~ 6 %. Device connected to PC computer (software genie 2000 version 3.1), this computer is responsible for the private management of the program relative alpha particles.

3. RESULTS AND DISCUSSIONS

Many parameters are very important in electrodeposition processes, some of these are critical, some are not. The three parameters which were studied had significantly influence of chemical yield. The influence of deposition time is shown in Fig. 2.

![Figure 2. Chemical yield as a function of electrodeposition time](image_url)
As can be seen from the figure above, the chemical yield increases by the time of electrodeposition until reaching (80 min) time, where the highest yield was obtained, then the uranium electrodeposition yield decrease in a time greater than 80 minutes.

Experiments in this study showed that electrodeposition diameter is a critical parameter that has a significant effect on the chemical yield. This can be seen clearly in the Fig.3.

**Figure 3. The relation between chemical yield and diameter of electrodeposition**

As noted from the results of electrodeposition diameter, small diameters 11, 14 mm had a relatively smallest chemical yield due to the phenomenon of self-absorption obtained as a result of the small diameter of electrodeposition, thus the deposition of uranium in the form of layers of one above the other where upper layer to block and attenuate alpha particles of Lower layer so chemical yield was reduced. As for large diameters 17, 20 mm, it was found that the chemical yield was the highest possible due to the disappearance relatively of the phenomenon of self-absorption and the distribution of the deposit homogeneously on stainless steel disc.

The results of the third parameters study showed that this parameter has an important influence on the results of deposition of uranium, especially on the chemical yield, which is due to the anode geometry (platinum rod) where the deposition process, in this case, is truncated cone, also effect of the current density distribution in electroplating electrolyte during the electrodeposition process, thus affects homogeneity of the source of alpha particles. Fig.4, showed the results of the distance between electrodes and their relationship with the chemical yield.

**Figure 4. The relation between electrodes distance and chemical yield**

Results in fig. 4 show that highest chemical yield can obtained of 1 cm distance and that means that the distance of 1 cm is the optimum distance for uranium electrodeposition.

Experiments proved that the chemical yield varies according to the diameter of the deposition and the distance between the electrodes. It is observed that the deposition diameter (20 mm) with the perfect distance between the electrodes was 0.5 cm. While in the case of the diameter of the deposition 17 mm, the optimum distance between the electrodes is 1 cm. The reason is due to the path that uranium ions took down to cathode electrode where there precipitates. Fig.5 represents the relationship between different diameters and distances between electrodes as a function of the chemical product.

**Figure 5. The relationship between different diameters and distances between electrodes as a function of the chemical product**

Finally, the optimum conditions for deposition of uranium can be listed in table.1, for the purpose of obtaining a source of alpha particles that can be measured using Alpha spectroscopy technique.

<table>
<thead>
<tr>
<th>Parameters</th>
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<tbody>
<tr>
<td>Deposition time</td>
<td>80 minute</td>
<td>pH of electrolyte</td>
<td>2.14</td>
</tr>
<tr>
<td>Deposition diameter</td>
<td>17 mm at 1 cm electrodes distance</td>
<td>Volume of electrolyte</td>
<td>15 ml</td>
</tr>
<tr>
<td></td>
<td>20 mm at 0.5 cm electrodes distance</td>
<td>Current</td>
<td>1.0 A</td>
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Table.1. Optimum parameters of uranium electrode-position using rod of platinum
In Fig. 6, displayed the spectrum of alpha particles achieved from natural uranium electrodeposition, where peaks of isotopes ($^{238}$U, $^{235}$U, $^{234}$U) can be observed.

4. CONCLUSION

Although the used anode (rod of Platinum) is not of the specifications of electrodes that given a high chemical yield, where most of the research in this field proved that the electrodes of the anodes with optimum specifications were shaped (spiral, mesh), but experiments in this research shows that a platinum rod can be used as anode, so that uranium can be deposited by using it. High chemical yield in addition to high resolution can obtain. Three parameters were studied for the purpose of reaching the perfect parameters while several sources of natural uranium were prepared by electrodeposition. Effect of electrodes distance was studied, optimum distance depended on the diameter of deposition (17 mm at 1 cm electrodes distance, 20 mm at 0.5 cm electrodes distance), deposition time was 80 minute, pH was 2.14, current 1.0 A.

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REFERENCES


