PREPARATION ALPHA SOURCE U²³⁴, U²³⁸ FOR ALPHA SPECTROSCOPY BY ELECTROPLATING USING ISOPROPYL ALCOHOL

^aLe Cong Hao, ^aLuu Xuan Tinh, ^aChau Van Tao, ^bNguyen Van Dong, ^aHo Nha Nghi, ^aPham Thuy Dung, ^aPhan Thanh Liem.

^aFaculty of Physics, University of Science Ho Chi Minh City ^bFaculty of Chemistry, University of Science Ho Chi Minh City Email: <u>lchao@phys.hcmuns.edu.vn</u>

Abstract:

Over the past few decades, many methods for the preparation of alpha source of ²³⁸U, ²³⁴U by electrodeposition technique for alpha spectroscopy analysis have been developed but none have been specifically designed to facilitate in a field situation. This article describes a simple preparation technique of uranium source by electrodepositing using isopropyl alcohol that is easily realized. In order to obtain optimum conditions for deposition, several parameters influencing the quality of layers such as current, distance between the anode and cathode and the deposition time were investigated. With optimal parameters, the ²³⁸U, ²³⁴U isotopes can be deposited quantitatively on a stainless steel disk within 20 to 40 minutes with nearly 100% yield and good energy resolution.

1. Introduction

Our laboratory use alpha and gamma spectroscopy to analyze many environmental and biological samples for uranium, plutonium, americium and thorium. Because of the low-level nature of these samples, subsequent radiochemical separators are generally done sequentially from a single sample, so alpha spectroscopy is proposed for analyzing. For the alpha spectrometric determination of some nuclides it is necessary for the deposited sample to be as thin as possible. The usual methods for preparing the α source are vaccum evaporation [1], electrospraying [2,3], painting with an organic solution [4,5], electrodeposition [6-12], and directrop [13] deposition. From the point of rapidity and safety of the treatment, we have investigated electrodeposition as method for preparing α source. For the electrodeposition of actinides in electrolyte buffer, several procedures have been reported. For examples, NaHSO₄ – H₂SO₄ – NH₄ buffer system [14], ammonium oxalate-ammonium sulphate [15], NaHSO₄ – Na₂SO₄ [16]and electrodeposition of alpha –emitting nuclides from mixed oxalate-chloride lectrolyte has been shown relatively accurate results [17] but none have been specifically designed to facilitate in a field situation and not so easy to use. In this work, it has been found that isopropyl alcohol as a solvent is very suitable for the electrodeposition of uranium to make the uranium target for acceralator experiments, producing of ⁹⁹Mo for medical purposes, and standard source of ²³⁸U and ²³⁴U for energy and detector efficiency calibration. This article describes the details of a simple preparation technique of uranium source (target) by electrodepositing using isopropyl alcohol.

2. Experiment

2.1 Reagents

All reagents were used throughout the work consisted of uranyl acetate powder, distilled water, nitric acid (HNO₃) 0.1 M, isopropyl alcohol ((CH₃)₂CHOH) and acetone (CH₃COCH₃). Also in this work, glasses, pipettes (3ml, 5ml and 50 μ l), filtered paper and gloves were used.

2.2 Preparation of sample solution

A stock solution of ²³⁸U, ²³⁴U was prepared by dissolving 0.035 g uranyl acetate powder in10ml of 0.1 M HNO₃, then was heated and stirred until fully dissolved to make solution. This work have been done at room temperature and taken a few minutes.. After that, the solution should be kept in the dark because uranium acetate is sensitive to light and deposits easily when it exposes light.

2.3 System of the electrolysis

As in shown following Fig.1, the electroplating was made of: teflon tube (height: 6cm, outer diameter of bed area: 3cm, inner diameter of bed area: 2.2 cm, overall capacity: 22 ml), stainless steel tube (height: 2.8 cm, outer diameter of bed area: 3.75 cm, inner diameter of bed area: 3.1 cm) surrounds teflon tube, electrodes, which cathode was made from stainless steel and anode from a platium wire, DC power supply with a floating votage of 0 to 300 V, abtained from Lybold company.



Fig.1: Schematic diagram of the electrodeposition cell

2.4 Instruments

The alpha-spectrometric system was a Canberra, model 7401 with the two detector (PIPS) type A1200-37Am. The measured sample was mounted at the chamber which was vacuumed at least 0.01 torr.

2.5 Procedure

The energy and efficiency calibration were done by using a custom electrodeposited alpha –source prepared and certified by Analytics Inc., containing ²³⁸U ²³⁴U, ²³⁹Pu, ²⁴¹Am

Before the electrodeposition, the stainless steel disks have to be cleaned by wiping the surface with acetone followed by pressing to smoother the surface. The clearing step is necessary because the leftover of the grease on the surface may affect the deposition process and finally affect the uniformity and efficiency of target.

From the stock solution of 238 U 234 U, an aliquot of 50 µl was added to 5 ml isopropyl alcohol then mixed with 100 µl nitric acid 0.1 M in a electrodeposition cell. And another work with the same process was done with 100 µl. By adjusting the voltage gradually to ensure that the current can keep in acertain value, the distance between cathode and anode was fixed. The deposition was performed onto polished stainless steel disks, which acted as the cathodes of the electrolysis cell, while the anode was platinum (Pt) which shape consists of a plane surface with Pt- rings.. The use of Pt ensured that it did not appreciably corrode during the electrodeposition. After the deposition the disk was cautiously rinsed with water and acetone and dried under the bulb.

3. Results and discussion

To determination the optimum conditions for deposition, a lot of standard samples with different conditions such as uranium concentration, voltage, current and time electrodeposition were investigated. The dependence of the deposition efficiency on the distance between cathode and anode was considered. From experience, the optimum distance between the two poles was considered to be 4 - 5 mm. However, because of some conditions of system of the electrolysis in our laboratory, 5 mm was the best choice in this work.

Fig. 2a shows the percentage deposition efficiency with varying current density keeping the distance between cathode and anode fixed at 5 mm in both two case of 50 μ l and 100 μ l of uranium solution. With the deposition time was 50 min and at a current of 10 mA, it was found that the deposition efficiency is very high and reached a nearly 100%.



Fig.2: The relationship between current density and the deposition efficiency.

Fig. 3a and fig. 3b show the relationship between deposition time and the deposition efficiency. According to fig. 3a (the first process), deposition efficiency increased rapidly at the start of the deposition process, up to 80% after 10 min and reached a nearly 100% at 40

min. While as in fig 3b (the second process), it took a long time to up to 80% (after 20 min) and reached nearly 100% at 60 min. From the results abtained in two processes, it can be noticed that after the first process, at the optimum conditions of electrodepositions as in table 1 the process shows a geometric curve behaviour, as described before. In the second process, the deposition rate was slower than in the first process, showing a different time between them. Considering that the uranium concentration of the solution in the first process is lower than in the second process, a bigger electrical conductivity of the uranium as oxide on the surface of the cathode must exist, which can explain the higher deposition rate of the uranium in first process.



Fig.3a: The relationship between deposition time and the deposition efficiency (the first process)





Table 1

Optimum conditions for the deposition of the first process

Anode	Platinum wire, 1 mm dia
Cathode (backing)	Stainless steel disc
Area deposited	7.544 cm^2
Current	10 mA
Deposition time	40 min
The distance between the two poles	5 mm
Temperature	32 °C

From the experiment and the fig. 4, it was found that satisfied samples were homogeneous, thin, fine surface, sticky on the surface and had energy resolution (FWHM) of 238 U from 36 keV to 50 keV and 234 U from 35 keV to 49 keV.



Fig.4: Spectrum of the satisfied samples.

4. Conclutions

The electrodeposition technique described in this paper is very simple, effective and expedient for preparing alpha source of ²³⁸U, ²³⁴U for alpha spectroscopy analysis. Beside the known parameters influencing the quality of layers such as current, distance between the anode and cathode and the deposition time, it is very important to consider the solute concentration. In all case the variance in the recoveries are well characterized and the deposited layer is very good no powder of uranium was erased, which concludes that the film adhered strongly to the backing material.

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