PREPARATION OF THIN SOURCES OF ^{69}Zn BY ELECTRODEPOSITION FOR USE IN NUCLEAR SPECTROSCOPY

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ABSTRACT

The aim of this study is to obtain a zinc active deposit on a copper substract in alkaline media containing zincate ions at potentiostatic conditions, using a copper rotating disk electrode. Electrochemical studies consisting of pontentiostatic and galvanostatic measurements were made in order to define the deposition conditions. An electroposition system and the respective procedure have been developed to determine the routine of preparation of thin samples of zinc The deposits adherence and their reproducibility were checked by nuclear-spectroscopy and its uniformity was checked by Scanning Electron Microscopy. Using a plastic scintillation detector, measurements have been performed of the end-point energy of the β-ray spectrum from the active deposit of ^{69}Zn

Key Words: zinc, electrodeposition, nuclear spectroscopy, electron microscopy

1.INTRODUCTION

The thickness of the radiactive source influences the experimentally obtained nuclear parameters, thus, the sources has to be prepared as a thin layer on the backing material. Basically, the method of preparing thin sources must fulfill the following requirements[1]: the active area must be thin and uniformly distributed on the backing material, which must also be thin and of low atomic number; the source preparation must be reproducible, good adhesion and have a high transfer efficiency. Based on these requirements the electrodeposition method [2] was chosen. It gives homogeneous and uniform deposits in a fairly easy reproducible way. Using this method, the sample can be prepared as a thin layer (<1μg /cm^2) on a metallic backing (~10μg /cm^2).

2.EXPERIMENTAL PROCEDURE

In the electrodeposition method is necessary to know the physical chemistry parameters associated to the radiactive material. This is carried out by potentiostatic and galvanostatic studies.

Potentiostatic Studies: In the potentiostatis studies solutions of 5mM $ZnSO_4$ was prepared using 5M NaOH as supporting electrolyte. A three electrode system was employed. The working electrode was a 2cm diameter copper rotating disk (99.9% purity) mounted in a cylindrical polytetrafluoroethylene sleeve such that only one cross section surface was exposed to the electrolyte. The counter electrode was a platinum foil and the reference electrode was a Hg/HgO/5M NaOH connected to the solution through a Luggin capillary. A conventional electrochemical

cell with a working volume of 1000ml was used. The solutions were deaerated sparging prepurefied nitrogen in the cell prior to the measurements and the gas was left over the solution during the experiments, conducted at room temperature. The potential deposition range determined in this study was around -1.50 V corresponding to a current density of 5 mA/cm^2 . In this range the electrodeposition process is controlled. The potentiostatic polarization curve of the solution containing only supporting electrolyte, $ZnSO_4$ 10mM, is presented in figure 1.

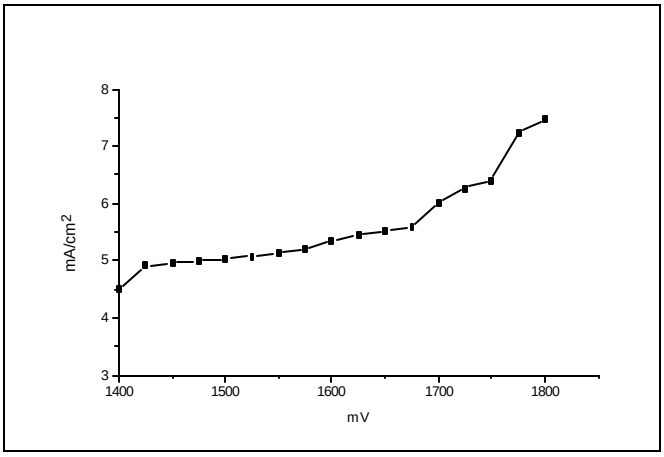

Figure 1: potentiostatic polarization curve

Galvanostatic Studies : In the galvanostatic studies about 5ml of the same electrolytic solution containing irradiated zinc was used. The radiactive zinc was prepared by neutron irradiation in the IEA-R1 research reactor at São Paulo. Approximately 7mg of $ZnSO_4$ was irradiated in a thermal neutron flux from 1.10^{13} to 2.10^{13} neutrons.cm^{-2}.s^{-1} for a period of 5 minutes. This process allows the formation of ^{69}Zn according to ^{68}Zn(n,γ)^{69}Zn reaction. Two electrode system was employed. The cathode was a copper foil (backing) and the anode was a 69mm long platinum wire with 1mm diameter and a spiral end. The cell, showed in figure 2, consist of a lucite body with conical central cavity and a brass base for electrical contact, over which the copper working electrode was mounted. The superior base diameter of the cavity was 30mm and the inferior one was 5mm. The distance between the electrodes was adjustable, normally between 1.0 and 2.0 mm, and the deposition area was about 0.2cm^2. The deposition time was estimated in 45 minutes at a constant applied current (~5 mA/cm^2).

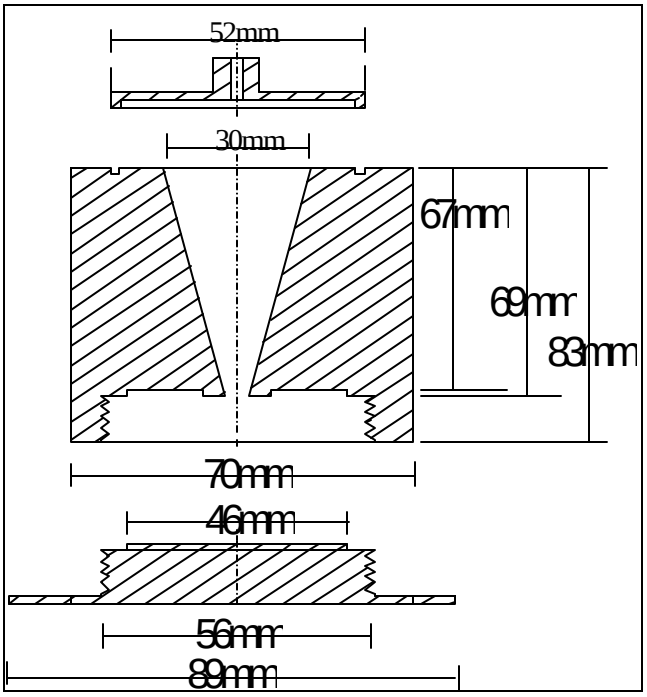

Figure 2:Diagram of the electrolytic cell. Dimensions in mm

The ^{69}Zn active deposits adherence and their reproducibility were checked by γ-spectroscopy using a HPGe detector. The data analysis consists of the integral area calculation of the select γ-transition, using the Panoramix computer code [3]. To verify the reproducibility, several active codeposits were analyzed. Each codeposit was counted for a period of one hour after which it was replaced by a freshly prepared sample containing approximately the same initial activity (~10 μCi). The area calculation of the select 438 keV γ-transition was used for this valuation. To verify the adherence of the codeposit, the same peak, at 438 keV, was used as a reference. In this test was made a comparison between the areas, in the same time interval, before and after a surface friction of the codeposits .

The data from reproducibility test shows a standard deviation of 8% and the adherence test shows a counting rate loss about 2%. The results are sastifactory considering the neutron flux variations and the deposits adhesions.

The electrochemical evidence of the deposit (presented in figure 3) and their uniformity were checked by SEM. Particularly, the uniformity was found to depend on the solution volume and the deposition time. The optimum conditions were estimate as 5ml of solution in about 45minutes of time deposition. Figure 4 shows electron micrographs of zinc deposit prepared in 5ml alkaline solution.

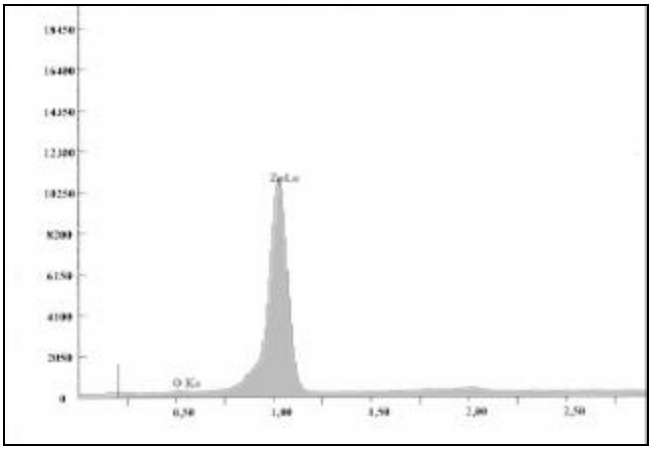

Figure 3 Electrochemical evidence of the zinc deposity

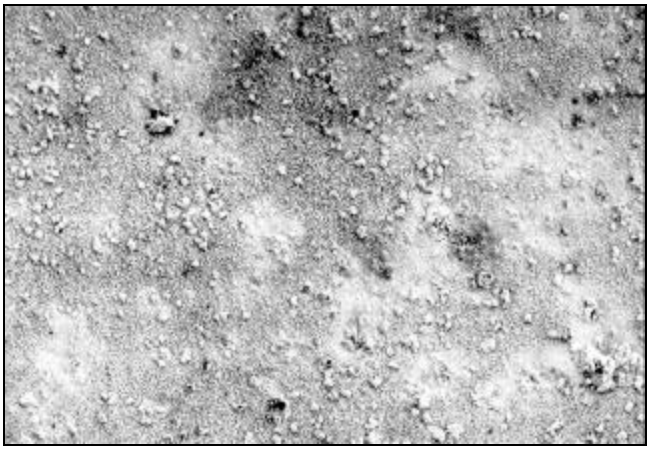

Figure 4 Electron micrograph of zinco

Beta-Spectroscopy : Basically, to obtain the β-spectrum measurements using the plastic scintillation detector, it is necessary to define the scintillation dimensions (diameter and thickness) and detection geometry.

This kind of detector was chosen based on its sensibility to beta radiation and conversion electron. It is produced at IPEN by the polymerization of styrene with organic flour. [4].

Previous studies [5,6] using these plastic scintillation to beta radiation detection, in the 100 to 2.0 keV energy range, pointed to 50 to 60mm diameter and 3 a 6mm thickness for the source-to-detector distance less than 10mm. Based on these data the scintillation dimensions and the geometry conditions were optimized. A systematic study in energy resolution terms was carried out using the ^{72}Ga, ^{137}Cs, ^{139}Ba and ^{207}Bi electrodeposited radioactive sources. The preparation of theses samples have been reported in details in reference [7,8,9]. The IDF program [10] was used to calculate the conversion electrons areas. The energy resolution data (FWHM) is comparable with the NE-102A commercially available (13% to 17%) [11].

Using the plastic scintillation detector, with 50mm diameter x 3mm thickness to 2mm source-to-detector distance measurements have been performed of the end-point energy of the β- ray spectrum from ^{69}Zn. The spectrum obtained in 1 hour acquisition time is shown in figure 5. It appears, that there is no longer any disagreement between the experimental spectrum shape and the predicted by the Fermi theory. The results is comparable to an end-point energy of the ^{69}Zn spectrum from reference [12](906±3 keV).

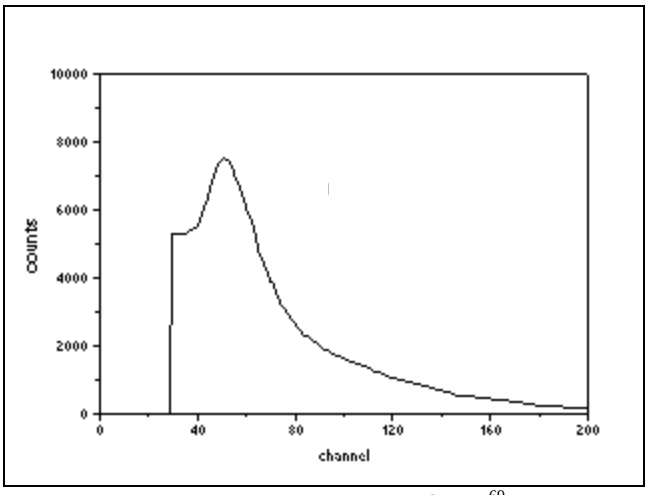

Figure 5: Beta-ray end-point energy of the ^{69}Zn spectrum (906 keV- ref.[12])

3.CONLUSIONS

Using the electrodeposition technique is possible to obtain a zinc thin uniform deposits in alkaline media with good adhesion, besides, this method offers the additional advantages of speed, simplicity, and low cost.

This electrochemical system as well as the apparatus described have been in continuos use and have proved satisfactory.

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