

Targetry and radiochemistry for no-carrier-added production of $^{117,118m,119,120m,122}\text{Sb}$

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Abstract. Targetry of $^{\text{nat}}\text{Sn}$ -target on Cu substrate was investigated for the production of $^{117,118m,119,120m,122}\text{Sb}$. The electrodeposition experiments were carried out using potassium stannate trihydrate and potassium hydroxide. The optimum conditions of the electrodeposition of tin were as follows: 40 g/L $^{\text{nat}}\text{Sn}$, temperature 75°C and current density (dc) used throughout of 50 mA/cm². The deposited target was irradiated at 160 μA current and 16 MeV proton beam (10 min). Separation of no-carrier-added (nca) $^{117,118m,119,120m,122}\text{Sb}$ from the irradiated $^{\text{nat}}\text{Sn}$ target hydrochloric solution was investigated using silica-gel column chromatography.

Key words: electrodeposition • targetry • silica-gel column • $^{\text{nat}}\text{Sn}$ • $^{117,118m,119,120m,122}\text{Sb}$

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Introduction

Antimony is a widely distributed toxic trace element in soils, and it is likely to be a pollutant in industrial environments [15, 16]. The mobility of antimony in agricultural soils was studied with the help of ^{124}Sb radiotracer [21]. The radionuclide ^{122}Sb was used for the studies of environmental contamination or food crops [4, 10, 11]. Only very few radionuclides exist that decay exclusively by electron capture (EC) mode without accompanying radiation, ^{119}Sb is one of them [2, 7, 8, 23]. Auger emitter ^{119}Sb ($T_{1/2} = 38.9$ h, $I_{\text{EC}} = 100\%$) are a potent nuclide for targeted radionuclide therapy based on theoretical dosimetry calculations at a subcellular scale [28–31]. ^{117}Sb ($T_{1/2} = 2.8$ h) decays mainly by EC (only 1.7% β^+) with the emission of nearly single X-ray of 158.56 keV suitable for imaging. In fact, the energies and intensities of the emitted photons in the ^{117}Sb decay are very similar to the photons emitted by the widely used single photon emission computed tomography (SPECT) isotope ^{123}I [22, 28, 29].

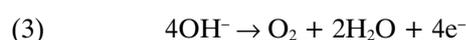
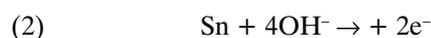
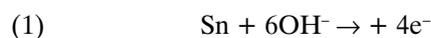
Cross section for the production of nca (no-carrier-added) $^{117,118m,120m,122,124}\text{Sb}$ radionuclides was measured by Khandaker *et al.* [11], Hermanne *et al.* [4], Johnson *et al.* [6], Kormali *et al.* [14], and Dmitriev *et al.* [3]. Cyclotron and reactor produced radioantimony were separated from the target matrix by liquid-liquid extraction (LLX) [1], ion-exchange chromatography [19, 20, 28, 29] and silica-gel column chromatography [9]. The aim of this work was to targetry of $^{\text{nat}}\text{Sn}$ by electrodeposition via alkaline solution on a pure copper substrate that has

sufficient stability at high-power beam bombardment, and separation of antimony radionuclides from natural tin using silica-gel column chromatography. Also several methods of targetry (alkaline and acid solution and sedimentation) have been studied.

Experimental

Target preparation

Tin targets are prepared by DC constant current electrolysis (CCE) of the metal from alkaline plating solutions. ^{nat}Sn (40 g/L, Merck 99.9%) was added in a 1000 ml beaker, followed by the addition of KOH (20 g/L), $\text{K}_2\text{Sn}(\text{OH})_6$ (115 g/L, Aldrich 99.9%) and deionized water (250 mL). After mixing and stirring at a temperature of 65–90°C, the homogenized solution was filtered through a fine glass filter (0.45 μm) to remove any residual particles if necessary to be ready for electroplating. Non-reactive plating vessels are hollow perspex cylinders (diameter 6 cm, height 20 cm) fitted with a tin anode foil. Four symmetrical windows (22.36 or 11.69 cm^2) on the vertical side wall allow up to four copper targets backing positioning. Each slot is sealed by an O-ring fitted-window. The slot geometrical shape determines the actual target electrodeposition area. Windows liquid-tight sealing is realized by stainless steel mechanical pestles mounted on a polyvinyl chloride (PVC) ring surrounding plating vessel and by pressing the copper backing against O-ring seal. An external PVC ring is fitted with four supporting pins to hold a motor-stirrer combination in position. The stirrer is a hollow perforated polyoxymethylene (POM) cylinder mounted on the axis of dc. The stirrer rotation speed is set at 1000 rpm; during the process, its rotating direction is reversed after 8 s improving deposit homogeneity [25, 27]. The electroplating process was carried out with a bath temperature of approximately 70–90°C with a plating current density of 30–80 mA/cm^2 for 3–5 h. Factors such as the operating temperature, solution constituent concentration and operating current density all affect the efficiency and plating rate of the system and must be property balanced and controlled. Three reactions are possible at tin anodes in alkaline solutions:



Equation (1) represents the overall process occurring at the anodes when the foil is intact and the tin is dissolving as stannate ion, with tin in the Sn^{+4} . Equation (2) is the process occurring if there is no foil and the tin is dissolving as stannite ion, with tin in the Sn^{+2} state. The addition of H_2O_2 to the electrolyte oxidizes the Sn^{+2} to Sn^{+4} , returning it to a usable condition. Equation (3) shows the decomposition of hydroxyl ion with the formation of oxygen. While this is a normal reaction at the anode, it should not be permitted to become the dominant reaction, as occurs when the anode current density is too high [12].

According to SRIM code [32, 33] the thickness has to be 168 μm for 90° geometry. To minimize the thickness of the ^{nat}Sn layer and to increase heat transfer, 6° geometry is preferred, in which case a 16.56 μm tin layer is recommended.

In method of sedimentation, ethyl cellulose was added to ^{nat}Sn . Then, suspensions of this mixed in acetone were obtained by mixing and stirring. To prepare the target a particular home-made system was constructed. It consists of two plates (19 \times 10 \times 3 cm^3) made of teflon. To achieve the desired thickness (16.56 μm), 169 mg ^{nat}Sn is required. But in order to avoid proton beam incidence with Cu backing and ^{65}Zn production, ^{nat}Sn deposit should be 30 μm . The ^{nat}Sn suspension solution was stirred for 5–10 min and loaded into a cylinder of the upper disk immediately. The window was covered by a plate made of teflon that includes a small hole. The solution evaporated slowly through the hole at room temperature after about 12–24 h [26].

For other electroplating processes, three types of electroplating baths were tested. At first, the plating was done with an acidic bath containing SnSO_4 dissolved in 4% H_2SO_4 . This bath was chosen due to the advantage that the plating can be carried out at room temperature (RT). The second type of plating solution tested, was made by dissolving Sn metal powder in hot 2–3 M KOH containing H_2O_2 (50–100 mL) to ensure the formation of $\text{Sn}(\text{IV})$ [29]. The obtained solution was diluted to 0.20–0.5 M KOH with distilled water and the electroplating was then carried out with a bath temperature of 60–100°C on a silver coated copper substrate. The third type of plating was done with an acidic bath containing ^{nat}Sn , 800 mg of Sn metal powder in 50 mL conc. HCl with H_2O_2 added. Then, 15 mL of 10 M KOH was added and when the freshly formed precipitate had disappeared, the solution was diluted to 400 ml with distilled water. The resulting solution was heated to approximately 75°C and transferred to the electroplating cell. The electroplating process was carried out for 8 h at 70–80°C with a current density of 3–5 mA/cm^2 [28, 31].

Irradiation

The coated natural tin was introduced into a target holder and bombarded with 16 MeV protons at a current of 160 μA for 10 min. The AMIRS (Agricultural, Medical and Industrial Research School, Iran) employs a Cyclone-30 (IBA, Belgium). To protect the target material from reaching excessively high temperatures during the irradiation a jet of cooling water flows across the back of copper substrate in direct contact with it, so heat removes efficiently from the backing. In order to improve the thermal conductivity, the copper substrate includes some grooves in the back. No direct cooling is used over the front of the deposited target.

Radiochemical separation

After irradiation, the target was dissolved in concentrated HCl containing H_2O_2 . A column size of \varnothing 1.0 \times 10 cm was used, packed with silica-gel (100–200 mesh, \geq 99%, Aldrich). According to methods of Khalid *et al.*

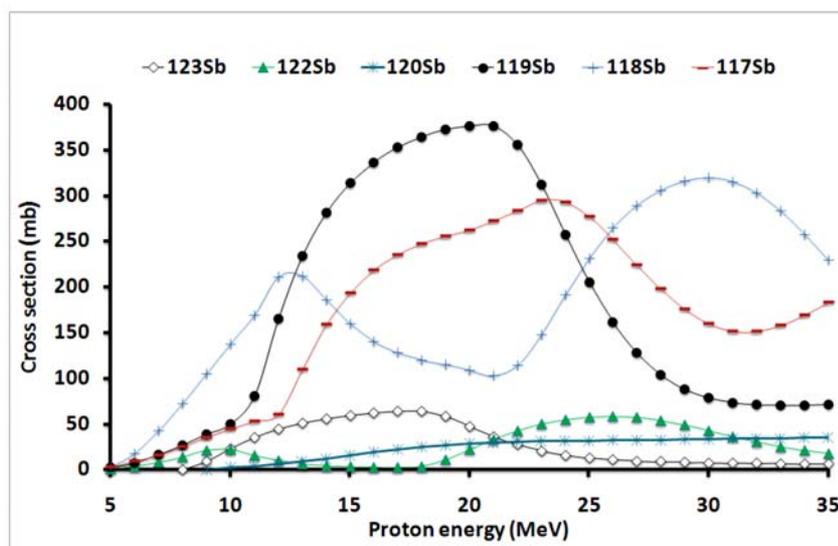


Fig. 1. Excitation function of the $^{\text{nat}}\text{Sn}(p,x)$ reaction by TALYS-1.2 code.

[9] and Thisgaard *et al.* [28, 31], it is possible to separate Sb from the bulk Sn target material by pretreating the silica-gel with 6 M HCl for 24 h prior to the separation. The pretreated silica-gel was washed and packed on the column and conditioned with 1 M HCl. The dissolved target solution (12 M HCl) diluted to 1 M HCl with deionized water and eluted through the column, followed by washing the column with 30 mL of 1 M HCl to remove any traces of Sn. Subsequently, the Sb-fraction eluted with 20 mL 6 M HCl.

Radionuclidic purity control

Identification and assay of gamma-ray emitting radionuclides was carried out using γ -ray spectroscopy with a high purity germanium (HPGe) detector (Canberra™ model GC1020-7500SL). For the $^{\text{nat}}\text{Sn}$ irradiations, the activities of the produced ^{117}Sb ($T_{1/2} = 2.80$ h), ^{118m}Sb ($T_{1/2} = 5.00$ h), ^{120m}Sb ($T_{1/2} = 5.76$ d) and ^{122}Sb ($T_{1/2} = 2.7238$ d) were measured. These isotopes were chosen instead of the ^{119}Sb due to the very low energy of the emitted γ -photon of 23.87 keV from the ^{119}Sb decay. Because of the high background from the other Sb isotopes produced simultaneously in the target it would be very difficult to get a correct measurement of the ^{119}Sb activity. Even if it had been possible to get an accurate measurement of the ^{119}Sb activity, it would not have been possible to scale this activity with increasing ^{119}Sn enrichment. This is because when using $^{\text{nat}}\text{Sn}$ as target material, ^{119}Sb will be produced from both the (p,n) and (p,2n) reactions on ^{119}Sn and ^{120}Sn , respectively [28].

Table 1. Influence of current density in alkaline solution (potassium stannate trihydrate and potassium hydroxide), the amounts used being 40 g/L tin, 20 g/L KOH and 115 g/L $\text{K}_2\text{Sn}(\text{OH})_6$ (temperature 75°C and plating time: 4 h)

| J (mA/cm ²) | Adhesion | Comments |
|-------------------------|-------------|---|
| 10 | Tolerable | Low current efficiency |
| 20 | Tolerable | Low current efficiency |
| 30 | Excellent | Reflective, smooth, low current efficiency |
| 50 | Excellent | Reflective, smooth, high current efficiency |
| 70 | Excellent | Reflective, smooth, high current efficiency |
| 100 | Tolerable | High current efficiency |
| 200 | Unfavorable | Unstable, rough, porous |

Chemical purity control

The presence of Cu^{2+} originating from the electroplated $^{\text{nat}}\text{Sn}$ on Cu backing was checked by polarography and colorimetric assays. A series of dilute copper standard solutions were checked up on our polarography apparatus limit of detection. For colorimetric assay, our limit of detection was 10 ppm. Standard copper concentrations were complexed by dithizone forming a pinkish complex [18].

Results and discussion

Excitation function of the proton-induced reaction on tin-natural was measured by TALYS-1.2 code [13] (Fig. 1). In order to optimize the $^{\text{nat}}\text{Sn}$ electrodeposition and sedimentation, the experimental conditions were investigated as follows.

Influence of current density

The electrodeposition experiments were carried out with different current density for several methods of electrodeposition (Tables 1, 2, 3 and 4). Tin ions in alkaline electrolytes have the valence of +4, whereas those in the acid electrolytes have the valence of +2. Consequently, the alkaline systems require the passage of twice as much current to deposit one gram-molecule of tin at the cathode [5, 12, 17, 24]. For electrodeposition, using potassium stannate trihydrate and potassium

Table 2. Influence of current density in alkaline solution with 2.5 M KOH containing 80 mL of H₂O₂, on a silver coated copper substrate (temperature 75°C and plating time: 7 h)

| J (mA/cm ²) | Current efficiency (%) | Adhesion |
|------------------------------|---------------------------|-------------|
| 4 | 22.1 | Unfavorable |
| 6 | 28 | Unfavorable |
| 8 | 18.6 | Unfavorable |
| 10 | 9.8 | Unfavorable |
| 45 | 3.2 | Unfavorable |
| 60 | 14 | Unfavorable |
| 85 | 47 | Unfavorable |
| 170 | – | – |

Table 3. Influence of current density in acidic solution with 70 g/L SnSO₄ dissolved in 4% H₂SO₄, on a silver coated copper substrate (temperature 30°C and plating time: 3 h)

| J (mA/cm ²) | Adhesion | Comments |
|------------------------------|-------------|-------------------------|
| 5 | Unfavorable | Low current efficiency |
| 10 | Unfavorable | Low current efficiency |
| 30 | Tolerable | Low current efficiency |
| 50 | Tolerable | Low current efficiency |
| 100 | Unfavorable | Unstable, rough, porous |
| 200 | Unfavorable | Unstable, rough, porous |

Table 4. Influence of current density in acidic solution with 15 ml of 10 M KOH and H₂O₂ (40 mL) dissolved in hot concentrated HCl (temperature 75°C and plating time: 8 h)

| J (mA/cm ²) | Adhesion | Comments |
|------------------------------|-------------|-------------------------|
| 1 | – | – |
| 3 | Tolerable | Low current efficiency |
| 5 | Tolerable | Low current efficiency |
| 7 | Tolerable | Low current efficiency |
| 10 | Unfavorable | Unstable, rough, porous |
| 50 | Unfavorable | Unstable, rough, porous |
| 100 | Unfavorable | Unstable, rough, porous |
| 200 | Unfavorable | Unstable, rough, porous |

hydroxide, the current efficiency was determined at different current densities using DC current at a temperature of 75°C. An optimum current density of about 50 mA/cm² should be applied.

Table 5. Influence of EC with 6 mL of acetone for 300 mg of ^{nat}Sn

| W (EC) (mg) | W (EC)/ W (^{nat} Sn) (%) | t^a (μ m) | Adhesion | Comments |
|------------------|---|---------------------|-------------|--------------------|
| 120 | 40 | 47.90 | Unfavorable | Porous, unstable |
| 90 | 30 | 44.48 | Unfavorable | Porous, unstable |
| 60 | 20 | 41.06 | Tolerable | Porous |
| 45 | 15 | 39.34 | Excellent | Reflective, smooth |
| 30 | 10 | 37.63 | Excellent | Reflective, smooth |

t – measured mean thickness of the deposit in micrometers.

^aThe prepared suspensions were manually introduced into the home-made system, so some suspensions were remained in the baker inevitably. Therefore, in some cases, despite of more amounts of ^{nat}Sn/EC, the obtained thickness was less than the coating condition with fewer amounts of ^{nat}Sn/EC.

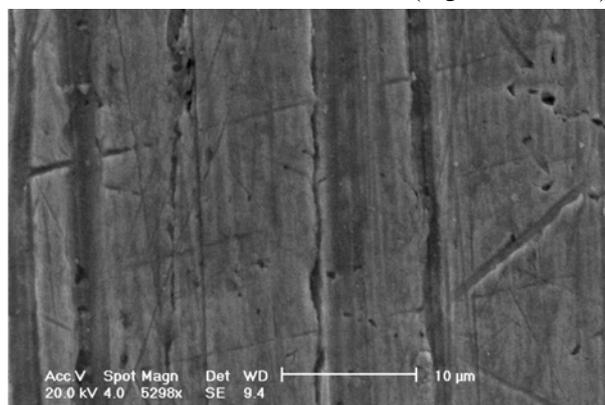
Influence of ethyl cellulose amount

Quantity of ethyl cellulose is an important factor for adhering and coating among physical properties of the samples. So, the quantity of ethyl cellulose should be at the optimum. The adhesion of samples was determined at different quantity of ethyl cellulose with respect to the ^{nat}Sn; these data are shown in Table 5 for an acetone amount of 6 mL. The optimum amount of ethyl cellulose is about 10% of ^{nat}Sn. Less amount of ethyl cellulose appears in adhesiveness of deposited ^{nat}Sn. On the other hand, more amount of it reduces the target thermal conductivity and causes tin to be porous.

Target quality control

To estimate the quality of the electroplated and sedimented tin targets some criteria had to be taken into account such as homogeneity, morphology and a thermal shock test. The homogeneity of the tin layer is important as it may seriously affect the production rate of antimony radionuclides. This was determined by measuring the thickness of several parts of the layer by a micrometer and calculation of the standard deviation of the data.

All electrodeposited tin target layers were examined for morphology by a scanning electron microscopy (SEM) technique (Figs. 2–6). The thermal shock tests involved the heating of the target up to 400°C for 1 h followed by submersion of the hot target in cold (15°C) water. SEM photomicrograph showed that the granulometry of electroplated targets with acidic baths and KOH + H₂O₂ were not as desired (Figs. 3, 4 and 5).

**Fig. 2.** SEM of a ^{nat}Sn deposit on a copper substrate (40 g/L tin, temperature 75°C, 20 g/L KOH, 115 g/L K₂Sn(OH)₆, $J = 50$ mA/cm² and plating time: 4 h).

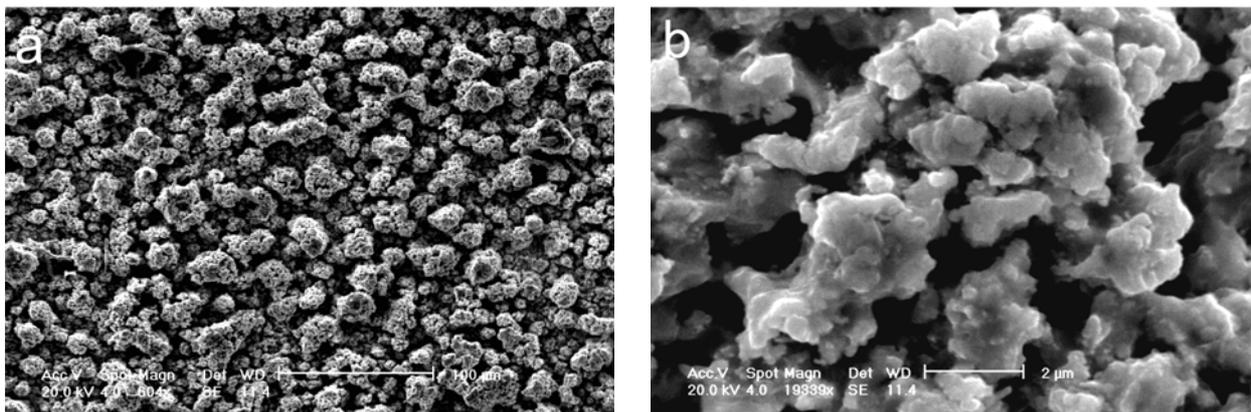


Fig. 3. SEM of a $^{117,118m,119,120m,122}\text{Sn}$ deposit on a silver coated copper substrate (a and b) (2.5 M KOH containing 80 mL H_2O_2 , temperature 75°C , $J = 5 \text{ mA/cm}^2$ and plating time: 7 h).

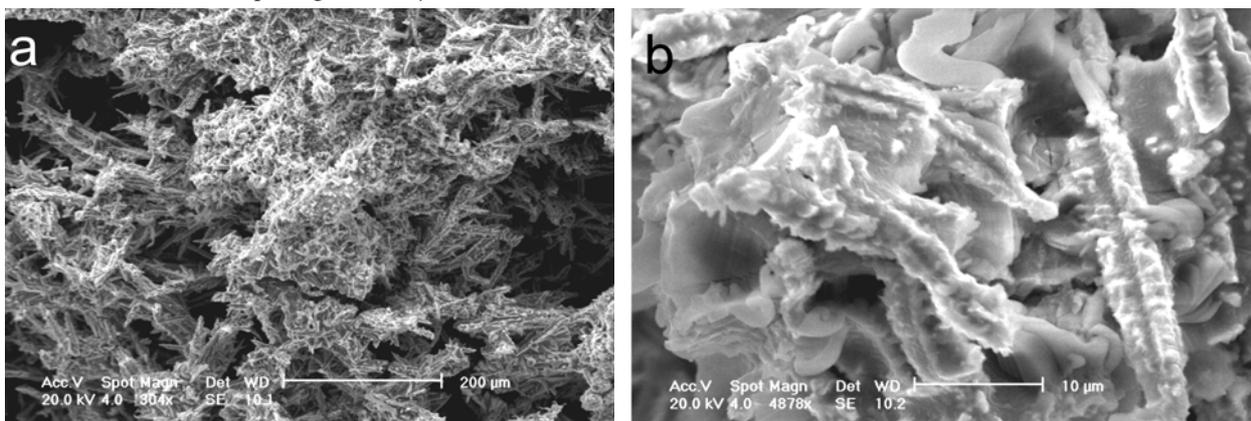


Fig. 4. SEM of a $^{117,118m,119,120m,122}\text{Sn}$ deposit on a silver coated copper substrate: (a and b) (70 g/L SnSO_4 dissolved in 4% H_2SO_4 , temperature 30°C , $J = 100 \text{ mA/cm}^2$ and plating time: 3 h).

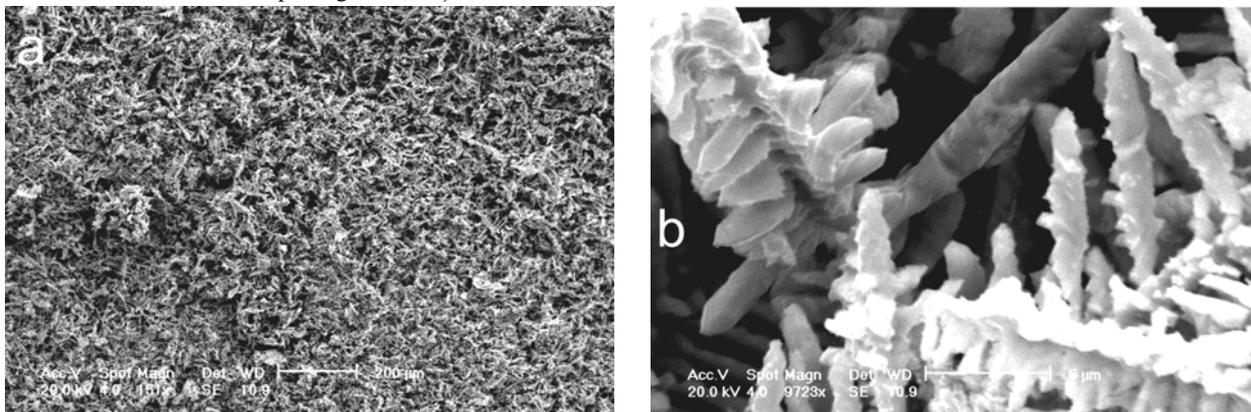


Fig. 5. SEM of a $^{117,118m,119,120m,122}\text{Sn}$ deposit on a silver coated copper substrate (a and b) (15 ml 10 M KOH and (40 mL H_2O_2) dissolved in hot concentrated HCl, temperature 75°C , $J = 5 \text{ mA/cm}^2$ and plating time: 8 h).

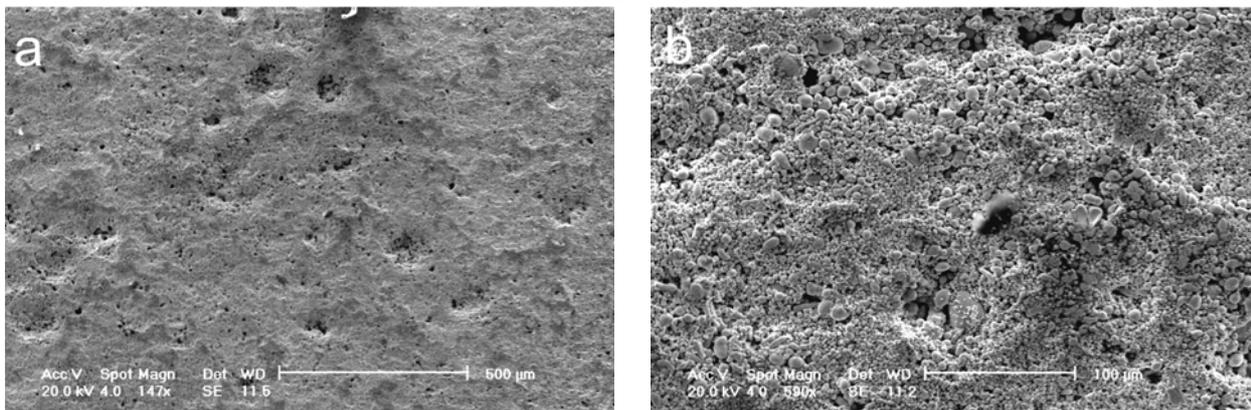


Fig. 6. SEM of a $^{117,118m,119,120m,122}\text{Sn}$ deposit on a Cu backing (a and b) (300 mg $^{117,118m,119,120m,122}\text{Sn}$, 30 mg EC, and 6 mL of acetone suspensions).

Also the sedimented tin targets in the thermal shock tests (300°C) were unstable (peeling off). Hence, electroplated targets with potassium stannate trihydrate and potassium hydroxide were chosen and the SEM photomicrograph showed suitable granulometry (Fig. 2).

After obtaining the optimum conditions (current density of 50 mA/cm² and temperature 75°C), a series of electrodeposition procedures were conducted on four target backings. Using different concentration of tin in the bath, the experiments were carried out for preparation of four targets (surface area of 11.69 cm²) with different thickness. At the end of the plating, the tin surface was shiny.

Sb/Sn radiochemical separations

After dissolution of irradiated tin in conc. HCl, the solution was evaporated three times by the addition of 12 M HCl. The dissolved target solution (12 M HCl) was diluted to 1 M HCl with deionized water and finally eluted from the column. The column was washed with 30 mL of 1 M HCl to remove the tin, while antimony chloride remained on silica gel. Then, the antimony chloride was eluted with 20 mL of 6 M HCl (eluent temperature: 70–80°C). Production yields for ¹²²Sb, ^{120m}Sb, ^{118m}Sb and ¹¹⁷Sb were 2.19 MBq/μA·h, 807 kBq/μA·h, 46.1 MBq/μA·h and 430 MBq/μA·h, respectively. As shown in Fig. 7, some impurities such

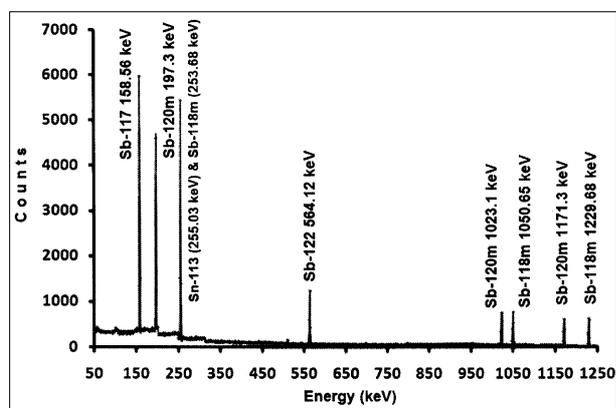


Fig. 7. Non-destructive γ -ray spectrum of proton irradiated ^{nat}Sn taken 24 h after end of bombardment (EOB).

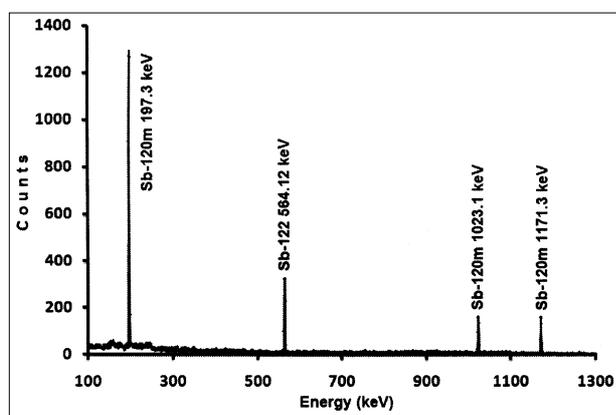


Fig. 8. HPGe spectrum of radiochemically separated antimony radionuclides taken 7 days after EOB. No other peaks have been detected in the γ -spectrum.

as ¹¹³Sn ($T_{1/2} = 115.09$ d, 255.03 keV) appeared in the dissolved sample. After separation (Fig. 8), the obtained antimony purity was better than 99%.

Conclusions

The target was prepared by electroplated tin-natural on a copper substrate using alkali solution (potassium stannate trihydrate and potassium hydroxide). The optimum conditions of the electrodeposition of tin were as follows: 40 g/L ^{nat}Sn, temperature 75°C and dc of 50 mA/cm². Also several methods of targetry (alkaline and acid solution and sedimentation) have been studied. The target was irradiated up to 160 μA current with 16 MeV protons beam and no degradation was observed. Antimony radionuclides were separated from the target with 80 ± 5% yield by using silica-gel column chromatography. Production yields of ¹²²Sb, ^{120m}Sb, ^{118m}Sb and ¹¹⁷Sb were 0.059, 0.021, 1.24 and 11.62 mCi/μA h, respectively.

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