



Preparation and Quality Control of ^{113}Sn , ^{125}Sb and ^{60}Co Adsorbed in ZrSiW: Volume and Planar Reference Source for γ -Detector Calibration

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NEUTRON activation of tin metal and cobalt chloride targets for production of ^{113}Sn and ^{125}Sb and ^{60}Co was carried out in Egypt's Second Research Reactor (ETRR-2). The ^{113}Sn , ^{125}Sb and ^{60}Co were loaded onto zirconium silicotungstate gel matrix from aqueous solution at pH 1 by the batch technique. The results obtained during the adsorption process of radionuclides were explained through the Visual MINTEQ program version 3. Then 1 and 0.1g of ^{113}Sn , ^{125}Sb and ^{60}Co -ZrSiW were packed into the volume and planar sealed sources, made of Chinese Artelon, respectively. The radioactivity levels of ^{113}Sn , ^{125}Sb and ^{60}Co were measured and found to be $(2.96 \pm 0.26, 2.22 \pm 0.26, 0.74 \pm 0.074 \text{ kBq})$ and $(0.3 \pm 0.03, 0.26 \pm 0.03, 0.11 \pm 0.01 \text{ kBq})$ for the volume and planar sealed source, respectively, on 27 June 2018. The efficiency curves for the two sealed sources were determined in the energy range 36-1332.5keV, which were measured at 5cm from the HPGe-detector to determine the radioactivity levels of unknown samples for the neutron activation method.

Keywords: Sealed Source; ^{125}Sb ; ^{113}Sn ; ^{60}Co ; Efficiency calibration.

Introduction

Radionuclides have been extensively used for more than 50 years in medicine and industry. The use of unsealed sources of radionuclides is largely applied in the diagnostic and therapeutic purposes as well as the use of sealed sources in many fields (Enomoto et al., 1975; Pappalardo et al., 1996; Sahoo et al., 1996; Hino et al., 2000; Krazaniak & Coppel., 2002; Saniot et al., 2004; IAEA-TECDOC-1512, 2006). "A Sealed Source" means a radioactive material that is permanently bonded or fixed in a capsule or matrix designed to prevent the release and dispersal of the radioactive material under the most severe conditions, which are likely to be encountered in normal use and handling (Radiation Safety Policy Manual, 2005). Several radionuclides such as of the well-known sealed sources: ^{32}P , ^{60}Co , ^{63}Ni , ^{65}Zn , ^{90}Sr , ^{90}Y ,

^{103}Pd , ^{106}Ru , ^{125}I , ^{133}Xe , ^{137}Cs , ^{144}Ce , ^{147}Pm , ^{166}Ho , ^{169}Yb , ^{192}Ir , ^{210}Po , and ^{241}Am are generally used (Enomoto et al., 1975; Sahoo et al., 1996; Hino et al., 2000; Krazaniak & Coppel, 2002; Saniot et al., 2004; Radiation Safety Policy Manual, 2005; IAEA-TECDOC-1512, 2006). Sealed sources are categorized by the IAEA according to their radioactivity in relation to a minimum dangerous source (where a dangerous source is the one that could cause a significant injury to humans). The ratio used is A/D, where A is the radioactivity of the source and D is the minimum dangerous activity. The radioactivity of the sealed sources is ranged from micro to thousand Curies depending on their use, where the latter type is used in industry and nuclear medicine (Mathew, 2002; IAEA-TECDOC-1512, 2006) while the former is commonly used in γ -detectors calibration such as ^{226}Ra (IAEA-TECDOC-1512, 2006).

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Different methods such as ion exchange and electro-deposition have been used for the preparation of sealed sources (Mostafa & El-Amir, 2007; El-Azony et al., 2009). In medicine ^{32}P , ^{60}Co , ^{90}Y , ^{103}Pd , ^{125}I , ^{133}X , ^{144}Ce , and ^{166}Ho are used in radiation therapy (teletherapy and brachytherapy) for the treatment of malignant diseases, bone density measurements and treatment of brain and prostate cancer (Enomoto et al., 1975; Sahoo et al., 1996; Hino et al., 2000; Krazaniak & Coppel, 2002; Mathew, 2002; Saniot et al., 2004; IAEA-TECDOC-1512, 2006; El-Azony et al., 2009).

Often, the samples are collected and measured in a granular form in order to determine the natural radioactivities. In neutron activation analysis, samples are bulky, granular, or may have no uniform distribution of radioactive isotopes. Evaluation of the radioactivity requires an absolute reference source, which has geometrical shape and mass attenuation coefficient similar to the sample measured (Gustafsson, 2014) for adjusting the efficiency calibration. The gamma energy lines emitted from the absolute reference source must cover a wide range of energy (< 100 up to more than 1000keV) for adjusting the energy calibration.

Many sealed sources have been prepared using an ion exchange technique based on the adsorption of an element or more on a granular ion exchanger material such as ^{65}Zn -permutitand ^{134}Cs -12-molybdocerate (IV) (El-Azony, 2009) ^{60}Co and ^{137}Cs -6-tungstocerate(IV) (Mostafa & El-amir, 2007).

In this study, the Visual MINTEQ (Gustafsson, 2014) as chemical speciation software packages has been used to explain and interpret the empirical results that obtained based on the distribution coefficients of ions between aqueous and solid phases because it can be extrapolated to systems of different ionic strengths, pH values, and component compositions (Koretsky, 2000; Bethke, 2008; Elmaghraby, 2016).

Antimony-125 ($T_{1/2} = 2.758$ y) is produced as a radioimpurity during the neutron activation of natural tin (^{nat}Sn (n, γ) $^{125}\text{Sn} \xrightarrow{\beta^-} ^{125}\text{Sb}$) to produce the $^{113}\text{Sn}/^{113\text{m}}\text{In}$ generator (El-Said et al., 2018). Due to the suitability of ^{125}Sb , ^{113}Sn and ^{60}Co physical properties (half-lives and gamma energies) as shown in Table 1 (Aydia et al., 2018), the authors

planned to use ^{125}Sb , ^{113}Sn and ^{60}Co in a sealed source together.

In a previous study (Aydia et al., 2018), zirconium silicotungstate (ZrSiW) was prepared by an inexpensive method and it is chemically stable gel matrix, and ^{113}Sn and ^{125}Sb were completely adsorbed on the ZrSiW matrix (El-Said et al., 2018).

The present study focuses on the findings of the previous study on the uptake of ^{113}Sn and ^{125}Sb on the ZrSiW gel matrix to prepare the sealed source of ^{113}Sn and ^{125}Sb -ZrSiW. Unfortunately, the γ -energy lines emitted from the ^{113}Sn and ^{125}Sb -ZrSiW are located in the range 36-671keV, which is insufficient to calibrate the γ -detector, so the sealed source of ^{60}Co , ^{113}Sn and ^{125}Sb -ZrSiW, was prepared with a wide energy range (36-1332.5keV).

Experimental

Reagents

All reagents used were of analytical grade. Double distilled water (DDW) was used throughout this work. $\text{ZrOCl}_2 \cdot 2\text{H}_2\text{O}$ from Sigma-Aldrich, $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$, and Na_2SiO_3 from Fluka WO_3 , ZrO_2 , and SiO_2 were purchased from Merck.

Preparation of zirconium silicotungstate (ZrWSi)

ZrSiW gel was prepared by mixing 50ml of 0.04M sodium tungstate ($\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$) with 50ml of 0.28M sodium silicate (Na_2SiO_3), then the tungstate-silicate solution was added dropwise to 50 ml of 0.04M warmed zirconium oxychloride solution ($\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$, in 0.2M HCl) within 30min at pH 8. Then the precipitate was washed several times using bi-distilled water till a constant pH is reached .

Neutron irradiation and targets dissolution

Tin-113, ^{125}Sb and ^{60}Co radionuclides were prepared by thermal neutron irradiation of the corresponding target materials, tin metal and cobalt chloride, respectively, in the 22MW ETRR-2 Egypt's Research Reactor for 4h at a neutron flux of 1×10^{14} n.cm⁻² s⁻¹. The irradiated tin target was dissolved using 6M HCl, gently heated and a few drops of 30% H_2O_2 solution were gradually added until complete dissolution and oxidation of Sn(II) to Sn(IV). Fresh solutions of ^{113}Sn were prepared by dilution.

TABLE 1. Decay mode of the radionuclides that used in the preparation of gamma standard sealed sources.

Radionuclide	Half-life	Decay mode					
		X-rays (keV)	I (%)	E (keV)	I (%)	E _β (keV)	I _β (%)
¹¹³ Sn	115.09 d	24.0	28.2				
		24.2	52.8				
		27.2	4.6	255.05	1.82	644.3*	98.3
		27.3	8.9	391.69	64		
		27.9	2.4				
¹²⁵ Sb	2.7582 y			35.49	4.3		
						95.25	13.5
				176.31	6.8		
						124.5	5.8
		27.21	13.5	427.87	30		
		27.47	25.2	463.36	10.5	130.6	18.1
		30.99	4.4	600.6	17.9	303.3	40.3
				606.72	5.0	445.6	7.2
⁶⁰ Co	5.2714 y			635.95	11.3	621.9	13.6
				1173.23	99.97	318.1	99.9
				1332.50	99.98		

A certain amount of the irradiated cobalt chloride was dissolved in hydrochloric acid then re-dissolved in double distilled water to obtain a clear solution.

Determination of the distribution coefficient (K_d) values

The distribution coefficients for [¹¹³Sn(IV) and ¹²⁵Sb] and ⁶⁰Co(II)(1X10⁻⁴ M) ions in aqueous HCl acid solutions of zirconium silicotungstate gel matrix were individually determined by the batch equilibrium technique. The reaction mixtures consisted of 0.1g gel material and 10ml of different concentrations of HCl (0.002-1M) in 25ml glass vials. The samples were shaken 24h using shaker thermostat. The distribution coefficient values (ml/g) were calculated using Equation (1):

$$K_d = \frac{A_o - A_e}{A_e} \times \frac{V}{m} \text{ mL/g} \tag{1}$$

where, A_o and A_e are the counting rates of the aqueous phase before and after equilibration with the gel matrix, respectively. V is the total volume of the aqueous phase (ml), m is the weight of the

gel matrix (g).

Loading of ¹¹³Sn, ¹²⁵Sb and ⁶⁰Co radionuclides on ZrSiW gel matrix

The dissolved tin (¹¹³Sn, ¹²⁵Sb) and cobalt (⁶⁰Co) were loaded using 1.2g ZrSiW matrix and equilibrated using 30ml of 10⁻²M ¹¹³Sn(VI) solution and 10ml of 10⁻³M ⁶⁰Co(II) solution at pH 1, and shaken overnight using the shaker thermostat. The supernatant was decanted, drying the loaded gel (¹¹³Sn, ¹²⁵Sb, ⁶⁰Co-ZrSiW) at 50°C then, washed with distilled water. It was dried once again at 50°C.

Preparation of ¹¹³Sn, ¹²⁵Sb, ⁶⁰Co-ZrSiW in different shapes (volume and planar sealed source) for the - detector calibration

The preparation technique was applied earlier using chromo-nickel stainless steel 12X18H10T in the ¹⁹²Ir sealed source preparation (IAEA-TECDOC-1512, 2006). The holder discs were made of Chinese artelon for preparation the volume and planar sealed sources. The Chinese artelon has some advantages such as its lightness that leads to reducing the attenuation of γ-ray

(self-absorption of γ -ray), its resistance to acids and alkaline solutions, organic solvents, hard shocks and and the temperature up to 140°C. The sketch of capsules constituents is shown in Fig. 1. Calibrated amounts of ZrSiW matrix loaded with ^{113}Sn , ^{125}Sb and ^{60}Co were packed in the cavity of the capsule base on the volume and planar discs. The space above the packed ZrSiW matrix was filled with thermal silicon to the top of the capsule to fix the core material. Then, the capsule lid was compacted into the capsule base (Platonov et al., 2002; Shtrombakh et al., 2005). After 24h, the sealed sources were subjected to the quality control tests.

Quality control tests

Sealed sources were tested to adjust the radioactivity measurement using gamma analysis equipment such as high-puritygermanium detector connected with Multi-Channel Analyzer (MCA). Several tests were carried out such as leachability in water (Tompkins, 2003), immersion test (leak test) (ISO, 1999), wipe tests (surface contamination testing), impact test (Martinez, 2002) and accurate measurement of activity.

Radioactivity measurements

All radioactivity measurements were carried out using a gamma-spectrometer, which has a p-type coaxial HPGe detector (GX2518 model), Canberra, USA, with 29.4% efficiency

and 1.66keV FWHM at 1332.5keV (^{60}Co). The detector is connected with a multichannel analyzer (MCA), power supply and amplifier that are contained in one unit (Inspector 2000 model, Canberra Series, made in USA). The relative efficiency curve was obtained using $^{152,154}\text{Eu}$ point source at 5 cm distance from the detector and this distance was used to determine the radioactivity of the sealed sources (^{113}Sn , ^{125}Sb , and ^{60}Co -ZrSiW), measured under the same conditions.

Methodology of energy and efficiency calibration of the gamma detector

The energy calibration is periodically carried out using the standard sources for the HPGe detector reported by Vucanac et al. (2008). After the identification of the energy using point source ($^{152,154}\text{Eu}$), the efficiency value was calculated taking into account the probability of disintegration for each energy line. These data are necessary for the efficiency calibration as illustrated by Equation (2):

$$\varepsilon_x = \frac{N}{t \times A \times p_x} \quad (2)$$

ε_x is the efficiency at a specific energy line, N is the peak area (number of counts at that energy), A is the absolute activity of the standard [Bq], P_x is the probability of disintegration of the radionuclide at that energy.

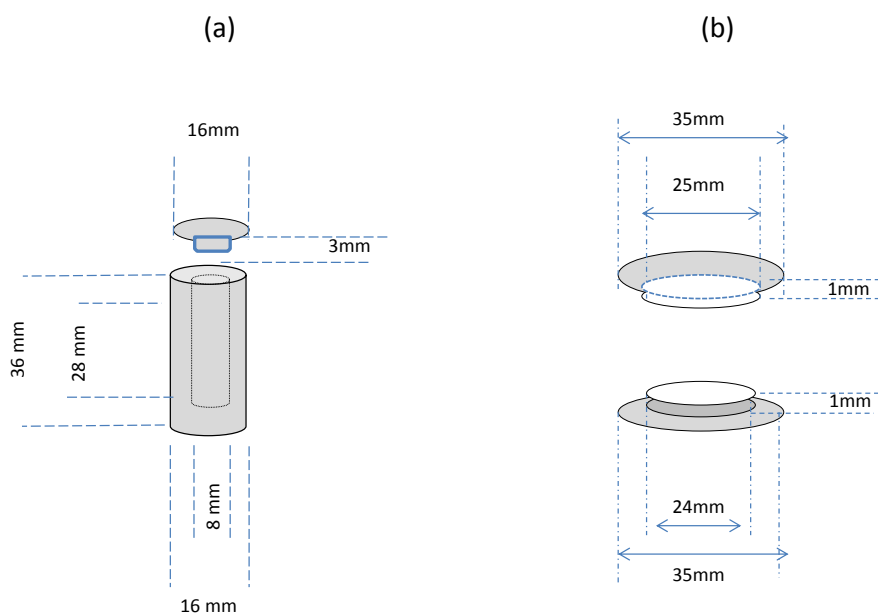


Fig. 1. A sketch of the volume (a) and planar (b) sealed source capsules used for preparation of ^{113}Sn , ^{125}Sb and ^{60}Co .

The efficiency percentage values for each gamma energy line of ^{152,154}Eu were drawn to obtain a curve, which was used to determine the polynomial coefficients in an equation of the sixth degree. Then, the radioactivity levels of ¹¹³Sn, ¹²⁵Sb and ⁶⁰Co in sealed sources (volume and planar sources) were determined using the same previous formula after calculating the efficiency for each gamma energy line and rearranging the previous formula to obtain Equation (3):

$$A = \frac{N}{t \times \epsilon_x \times p_x} \quad (3)$$

Result and Discussion

Figure 2 shows the gamma-ray spectrum of the metal tin target, which was measured one year after its irradiation for decay out the short-lived radionuclides by using High-pure Germanium detector connected to Multi-Channel Analyzer (MCA). The gamma-ray energies of ¹¹³Sn and ¹²⁵Sb were detected by the following nuclear reactions:

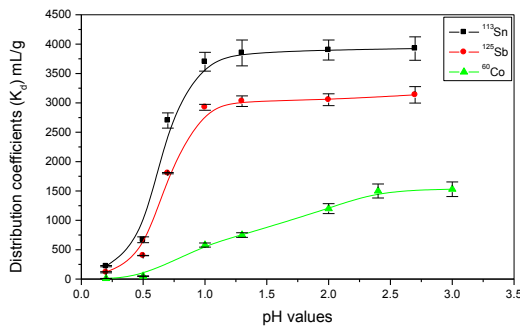
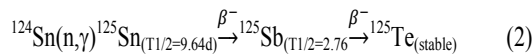
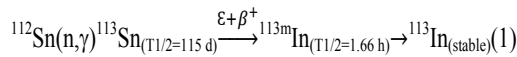
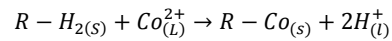
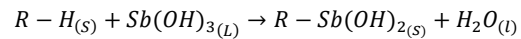
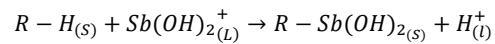
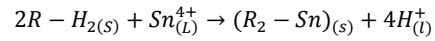


Fig. 2. Variation of Kd of ¹¹³Sn(IV), ¹²⁵Sb(III), ⁶⁰Co(II) in an aqueous medium on the ZrSiW gel matrix as a function of pH.

The high distribution coefficients of ¹¹³Sn (IV), ¹²⁵Sb (III) and ⁶⁰Co (II) were experimentally obtained (3700, 2925 and 580ml/g, respectively) by the batch technique at pH 1 as shown in Fig. 2. The results obtained have been interpreted based on Visual MINTEQ 3.0 program that determines the studied elements concentrations of different aqueous species as a function of the pH as shown in Fig. 3 (a, b and c), which indicate the aqueous concentration of the

dissolved species of tin, antimony and cobalt in cationic forms, Sn⁴⁺, Sb(OH)₂⁺, Sb(OH)₃ and Co⁺² with the concentrations 100, 78,22 and 100%, respectively at pH 1. The mechanism was carried out depending on the hydrolytic sorption or the cation exchange.



On the other hand, the ionic charge and radius of Sn⁴⁺, Sb³⁺, and Co⁺² play an important role in the absorption mechanism onto the ZrSiW as a cation exchanger. The results confirm that the absorption of Sn⁴⁺, Sb³⁺, and Co⁺² are based on their ionic charges +4 > +3 > +2, and also their ionic radius 83, 90 and 88.5pm, respectively.

Radioactivity of Sealed sources (volume and planar)

In a previous work (El-Amir et al., 2013), a point source based on irradiated natural tin target was prepared. Most of the time, the samples of natural radioactive materials are collected and measured in a granular form. In neutron activation analysis, samples are granular, and they may not be uniformly distributed from radioisotopes. The assessment of radioactivity requires an absolute reference source. The γ-energy peaks emitted from the absolute reference source must cover a wide range of energy for adjusting the energy calibration.

The radionuclides of ¹¹³Sn, ¹²⁵Sb and ⁶⁰Co were loaded onto zirconium silicotungstate (ZrSiW) by the batch technique and the volume and planar sealed sources were prepared by the packing of 1 and 0.1g of ¹¹³Sn, ¹²⁵Sb and ⁶⁰Co-ZrSiW into the holders made of the Chinese Artelon as shown in Figs. 1-a and 1-b, respectively, then they were measured in 27 June, 2018. Figures 4 and 5 show typical gamma-spectra of ¹¹³Sn, ¹²⁵Sb and ⁶⁰Co for the volume and planar sealed source, respectively. The spectra show only the peaks attributed to these radioisotopes. The absence of any radionuclide as an impurity confirms the success of the separation and preparation method of the sealed source. In addition to the importance of the radionuclidic purity for the gamma energy calibration, there

are several factors play an important role in the estimation of the radioactivity with a high accuracy of the unknown sample using the neutron activation method such as the geometric shape of the sealed source holder and the unknown sample holder, and accuracy of the efficiency curve of the sealed source, which was used to calculate the radioactivity values of the unknown sample. Figures 5 (a and b) shows the efficiency calibration curves, which were determined at 5 cm from the detector and processed by the polynomial fitting to obtain Equation (4) of the sixth degree:

$$y = A + Bx + B_2X^2 + B_3X^3 + B_4X^4 + B_5X^5 + B_6X^6 \quad (4)$$

where y is the efficiency (ϵ_x) at a any gamma-energy, B values are the polynomial coefficients, X is the gamma energy line (keV)

The efficiencies calibration for gamma energy lines of ^{113}Sn , ^{125}Sb , and ^{60}Co were determined by measuring the source on the HPGe-detector and using Equation (2) to calculate the efficiency for each gamma-line, then by compensation

in Equation (3), the radioactivity levels were set for these radionuclides, which were found to be $2.96_{\pm 0.26}$ kBq ($0.08_{\pm 0.007}$ Ci), $2.22_{\pm 0.26}$ kBq ($0.06_{\pm 0.007}$ Ci), and $0.74_{\pm 0.074}$ kBq ($0.02_{\pm 0.002}$ Ci), respectively as shown in Table 2. The results show that the radioactivity values of the planar source were less by about the tenth (1/10) of the volume sealed source values as shown in the Table 2 that compiles data of ^{123}Sn , ^{125}Sb and ^{60}Co radioactivity of the planar source, which was found to be $0.3_{\pm 0.03}$, $0.26_{\pm 0.03}$ and $0.11_{\pm 0.01}$ kBq, respectively.

Figure 6 (a and b) illustrates the two efficiency curves using the volume and planar sealed sources, respectively, and the results show that there is a slight difference between the values of the two curves. This may be due to the difference in the geometric shape of the two sealed sources, in addition to the inverse proportion relationship between the radioactivity and the efficiency. Therefore, the efficiency values of the volume source were less than the values that were obtained using the planar source at the same measured position (5cm)

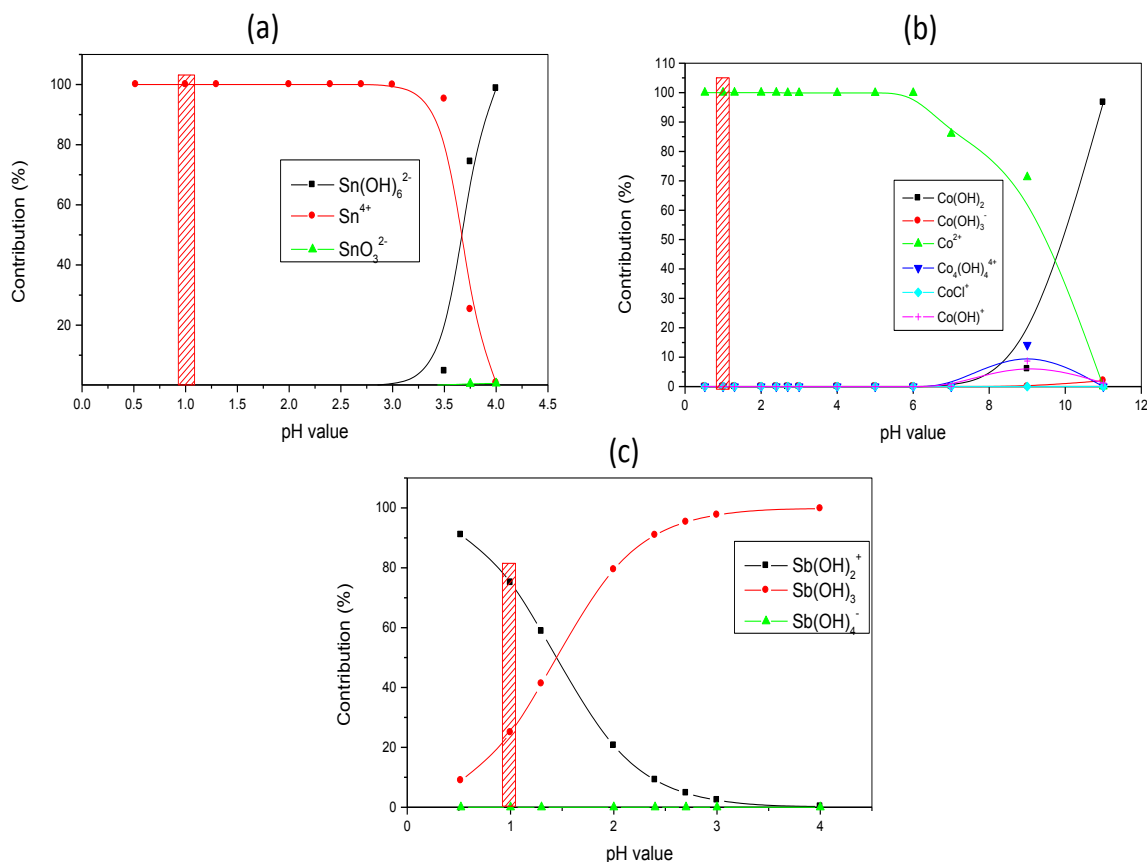


Fig. 3 (a, b and c). Variation concentration (%) of different species of Sn, Sb and Co, respectively as a function of pH in an aqueous solution (chloride medium) by using VISUAL MINTEQ Version 3.0, beta software.

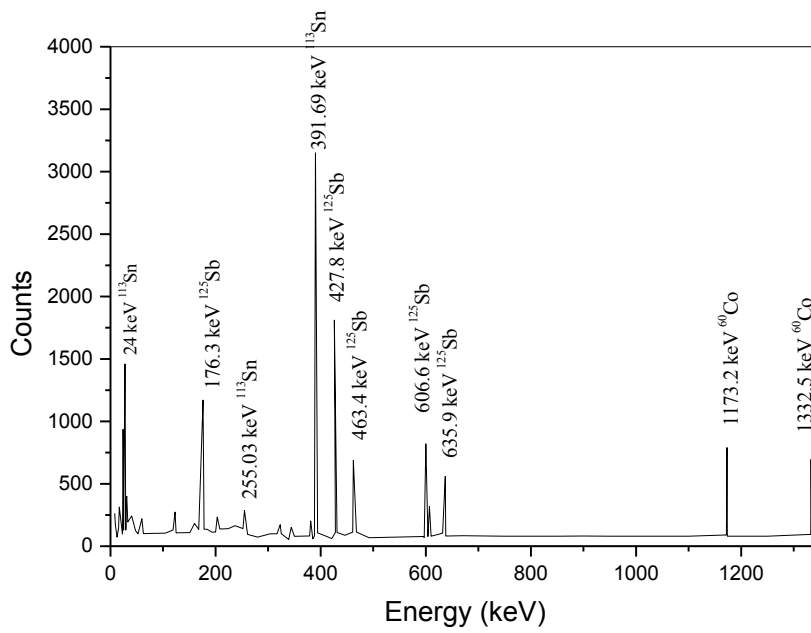


Fig. 4. γ -ray spectrum of the ^{113}Sn , ^{125}Sb and ^{60}Co -ZrSiW emitted from the volume sealed source that measured at 5cm from the detector.

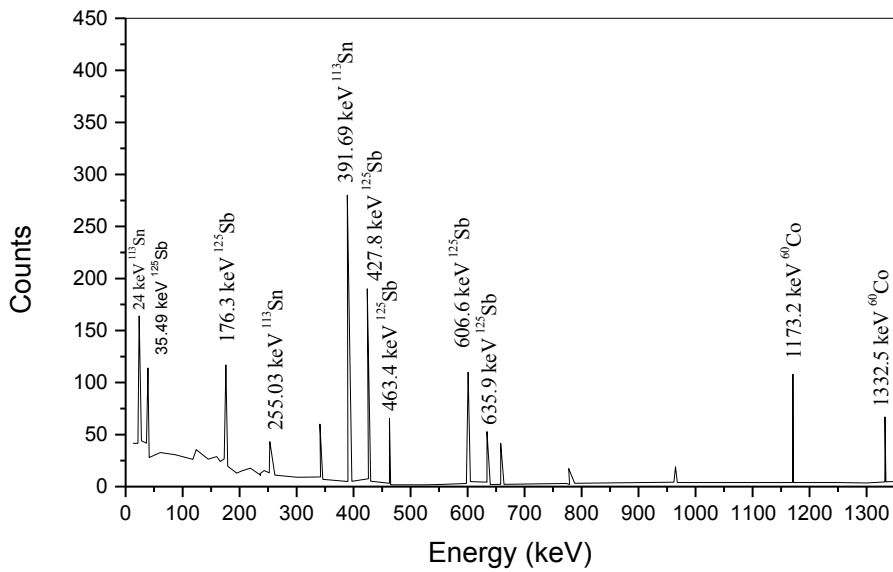


Fig. 5. γ -ray spectrum of the ^{113}Sn , ^{125}Sb and ^{60}Co -ZrSiW emitted from the planar sealed source that measured at 5cm from the detector.

TABLE 2. The determination of radioactivity of ^{113}Sn , ^{125}Sb , ^{60}Co -ZrSiW in the volume and planar sealed sources.

Sample	Matrix materia	Weight of radioactive ZrSiW	Radioactivity(μCi)		
			Sb-125	Sn-113	Co-60
Plannar source	ZrSiW	0.1	0.007 \pm 0.0008	0.008 \pm 0.00080	0.003 \pm 0.0003
Volume source		1	0.06 \pm 0.007	0.08 \pm 0.007	0.02 \pm 0.002

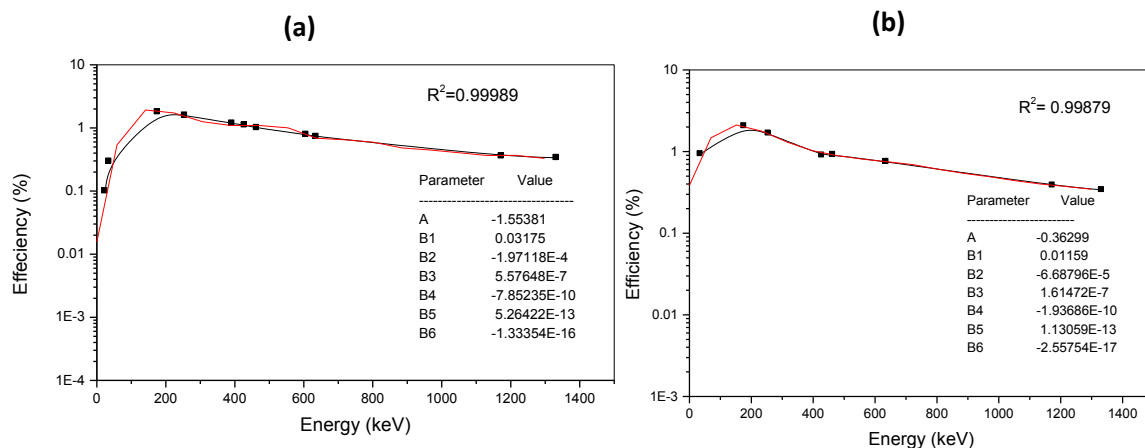


Fig. 6. Efficiency curve as a function of γ -energy at 5cm using (a) a volume sealed source (b) a planar sealed source of ^{113}Sn , ^{125}Sb , ^{60}Co -ZrSiW.

Conclusion

Sealed sources of ^{113}Sn , ^{125}Sb and, ^{60}Co -ZrSiW were prepared using the neutron activation technique. The prepared sealed sources could be used to measure an unknown sample, and this requires several demands such as the accuracy of the radioactivity values of the prepared sealed sources (^{113}Sn , ^{125}Sb and, ^{60}Co) that depend on using a known standard source ($^{152,154}\text{Eu}$), so as to determine the radioactivity values of the prepared sealed sources of ^{113}Sn , ^{125}Sb and, ^{60}Co . The prepared sealed sources have wide g-energy range (36-1332.5keV). In this study, the holder in the unknown sample and the standard source is made of Chinese Artelon and have the same geometric shape, which is a suitable material due to its low absorption of the emitted g-ray. The quality control tests were carried out on the prepared sealed sources (volume or planar) to examine its suitability for the gamma-energy calibration of the HPGe detector and for the determination the radioactivity of the unknown sample via estimating the efficiency calibration at 5cm that confirms its relevance for application. Calculating the efficiency of two different types in the geometric shape of this holder enables us to assign the samples, which have high radioactivity and small-weight using neutron activation method.

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تحضير و مراقبة الجودة للمصادر المرجعية الحجمي و المسطح للقصدير-113 والأنتيمون-125 و الكوبالت-60 المدمص على سليكو تنجستات الزركونيوم

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تم التنشيط النيوتروني لأهداف من القصدير وكلوريد الكوبالت في مفاعل مصر البحثي الثاني (ETRR-2) لتحضير نظائر القصدير-113 والأنتيمون-125 والكوبالت-60 على التوالي. وقد تم تحميل نظائر القصدير-113 والأنتيمون-125 والكوبالت-60 على سليكو تنجستات الزركونيوم من المحاليل المائية عند أس هيدروجيني 1 بطريقة إيزان الطريجة. وقد تم تفسير النتائج التي تم الحصول خلال عملية الإدمصاص باستخدام برنامج Visual MINTEQ الإصدار الثالث. وقد تم تصنيع المنابع المغلقة بتعبئة 1 و 0.1 جم سليكو تنجستات الزركونيوم المحملة بنظائر القصدير-113 والأنتيمون-125 والكوبالت-60 على التوالي داخل كبسولات حجمية و مسطحة مصنوعة من مادة الأرتيلون الصيني. وقد تم قياس إشعاعية نظائر القصدير-113 والأنتيمون-125 والكوبالت-60 ووجد أنها 2.96 ± 0.26 , 2.22 ± 0.26 , 0.74 ± 0.074 و 0.3 ± 0.03 , 0.26 ± 0.03 , 0.11 ± 0.01 كيلو بيكريل على التوالي. وقد تم تعيين منحنيات الكفاءة للمصدرين في نطاق الطاقة 36-1332.5 كيلو إلكترون فولت وذلك بالقياس على بعد 5 سم من كاشف الجرمانيوم عالي النقاوة لتقدير الإشعاعية للعينات المجهولة بطريقة التنشيط النيوتروني.