



APPLICATION OF ADSORBENTS IN RADIONUCLIDE SEPARATION FOR RADIO-CHRONOMETRY PURPOSES

Mykola Strilchuk¹, Igor Maliuk¹, Ivan Mironyuk², Hanna Vasylyeva^{3*},
Volodymyr Tryshyn¹, Maryna Hryhorenko¹, Oleksandr Zhukov¹, Khrystyna Savka²

¹NAS of Ukraine Institute for Nuclear Research, Laboratory of Nuclear Forensics, Kyiv, Ukraine
²Department of Chemistry, Vasyl Stefanyk Precarpathian National University, Ivano-Frankivsk, Ukraine
³Department of Theoretical Physics, Uzhgorod National University, Uzhgorod, Ukraine

Abstract. The aim of this work is the application of well-known adsorbents for the separation of ⁹⁰Sr, ⁹⁰Y, and ⁹⁰Zr radionuclides. Three basic types of adsorbents have been studied: Dowex HCR S/S cation exchange resin, Dowex 1x8 anion exchange resin, and titanium dioxide with a chemically modified surface. The most effective adsorbent for the separation of strontium, yttrium, and zirconium ions was titanium dioxide with a chemically modified surface. This adsorbent selectively absorbs zirconium cations against the background of excess strontium and yttrium ions. The separation takes place in 2% HNO₃ at initial concentrations of the studied cations 10 ng/ml and 100 ng/ml. Analysis of the initial mixture and the mixture after separation was conducted using ICP-MS “Element-2” with argon plasma. Age of ⁹⁰Sr-⁹⁰Y β⁻ -source (approximately 30.2 years old) was measured using the method of the chemical separation of ⁹⁰Sr and ⁹⁰Zr by the titanium dioxide and following calculation of the ⁹⁰Zr/⁹⁰Sr ratio. The age of ⁹⁰Sr-⁹⁰Y β⁻ -source was calculated as 31.9 ± 1 year. The combination of liquid scintillation counting of ⁹⁰Sr and ICP-MS analysis was proposed as an alternative method of determination of the ⁹⁰Zr/⁹⁰Sr ratio. It was shown, that both methods provide similar results in radio chronometry of ⁹⁰Sr-contained compound, i.e. age-dating of liquid ⁹⁰Sr-⁹⁰Y β⁻ -source, and could validate each other.

Keywords: Strontium, yttrium, zirconium, separation, ICP-MS, liquid scintillation counting

1. INTRODUCTION

Accurate radionuclide separation is necessary for archaeological dating or in nuclear forensics to determine the age of unknown ⁹⁰Sr-containing devices [1, 2]. The isotopic ratio of ⁹⁰Sr(β⁻)-⁹⁰Y(β⁻)-⁹⁰Zr (stable), measured using ICP-MS, serves as a radio chronometer and can be used to determine the date of fabrication of a ⁹⁰Sr -⁹⁰Y radioactive source. The half-life of ⁹⁰Y is much shorter (64 hours) than that of ⁹⁰Sr (28,8 years). On elapse of time equal to several half-lives of yttrium, the amount of ⁹⁰Y becomes constant [3-6]. Therefore, the number of ⁹⁰Sr and its “granddaughter” ⁹⁰Zr nuclei, i. e., the ⁹⁰Zr/⁹⁰Sr ratio is an important parameter for nuclear forensics, thus methods of chemical separation of these elements become the main direction of scientific research in this field. The most effective method of separating the elements is the adsorption method [3, 4] at present time.

Liquid Scintillation Counting (LSC) offers unmatched convenience and sensitivity for pure beta-emitters, such as ⁹⁰Sr or ⁹⁰Y [6-8]. While LSC has disadvantages as quenching, requiring cocktail, and little selectivity, it has an advantage as 4π geometry and high efficiency. LSC detects radioactivity via the same type of light emission events that are used in the solid scintillator. The key difference is that the scintillation takes place in a solution of a scintillator. This allows

close contact between the radioactive isotopes and the scintillator substance.

From another hand, inductively coupled plasma mass spectrometry (ICP-MS) is an analytical technique that can be used to measure elements at trace levels in a liquid's medium. From a laboratory perspective, the most significant advantage of ICP-MS is its multi-element capability, which allows multiple elements to be measured simultaneously in a single analysis. The high sensitivity of ICP-MS provides the use of this analytical method in nuclear forensics.

Many different adsorbents are proposed for the adsorption of heavy metal cations and radionuclides, for example, zeolites [9], salts of polyvalent metals [10], mesoporous TiO₂ [11-17]. Separation of Sr, Y, and Zr elements is often performed using ion exchange resin [18-22]. As usual, ion exchange resin is cross-linked polystyrene with different types of active groups, for example, sulfonic groups -SO₂OH giving strongly acid cation exchangers [18]; carboxylic groups -COOH giving weakly acid cation exchangers [22]; quaternary ammonium groups -NR₃⁺ giving strongly basic cation exchangers [20] as well as chelators agent onto neutral polymeric matrix [21].

The aim of this work is the application of well-known adsorbents for the separation of ⁹⁰Sr, ⁹⁰Y, and ⁹⁰Zr radionuclides and calculate the age of ⁹⁰Sr-⁹⁰Y β⁻ - source using the method of the chemical separation of

* h.v.vasylyeva@hotmail.com

^{90}Sr and ^{90}Zr by the best adsorbent in this field. The aim of this work is also to compare the obtained results with the combination of LSC of ^{90}Sr and ICP-MS analysis of peak with 90 amu as an alternative method of determination of the $^{90}\text{Zr}/^{90}\text{Sr}$ ratio.

2. EXPERIMENTAL TECHNIQUES

2.1. Adsorbents

Three basic types of adsorbents have been studied: Dowex HCR S/S cation exchange resin with active sulfonate groups, Dowex 1x8 anion exchange resin having quaternary amines in its structure [17], and mesoporous anatase modification titanium dioxide with a chemically modified surface. Dowex HCR S/S and Dowex 1x8 are commercially available adsorbents. Titanium dioxide of anatase modification was synthesized in the Chemical Department of V. Stefanyk Precarpatian National University (Ivano-Frankivsk, Ukraine) [17].

Active adsorption sites of adsorbents are shown in Fig.1.

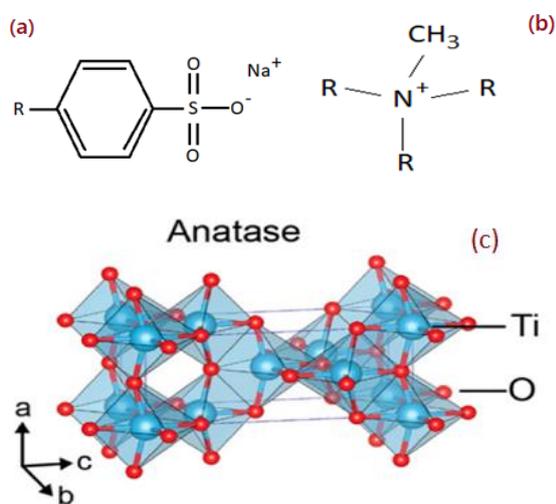


Figure 1. The sulfonic groups $-\text{SO}_2\text{OH}$ active site of Dowex HCR-S/S (a) and quaternary ammonium groups $-\text{NR}_3^+$ (Dowex 1x8) (b). Titanium dioxide of anatase modification (c). The structure of titanium dioxide was adapted from publication [11].

2.2. Chemical separation of stable strontium, yttrium, and zirconium ions from their mixture

The chemical separation of stable strontium, yttrium, and zirconium ions from their mixture was performed using stable isotopes ^{88}Sr , ^{89}Y , ^{90}Zr . Separation of these elements by cation exchange resin and titanium dioxide was performed from a 2% HNO_3 medium with HF micro impurities. The separation of strontium and zirconium ions by Dowex 1x8 occurs in a 5% HCl medium and for subsequent analysis using ICP-MS a solution of 5% HCl is evaporated for 5 hours, and then the dry residue is dissolved in 2% HNO_3 (Optima).

Several series of investigations were performed for the effective analysis of the chemical separation of the elements. In the first series of the studies, the initial

mixture of stable isotopes with concentrations of 10 ng/ml was used. The weight of the adsorbent was 100 mg, the volume of the mixture was 10 ml. Duration of interaction 60 min. The initial concentration of elements (stable isotopes) in the mixture was increased to 100 ng/ml in the second series of studies, other conditions of the experiment remained the same.

In the third series of studies, a mixture of 100 ng/ml of each element was used, however, the volume of the solution was 1 ml + 1 ml of pure nitric acid. Accordingly, the mass of TiO_2 decreased by 0.02 g. High-purity standards were used to prepare a mixture of strontium, yttrium, and zirconium. Analysis of the initial mixture and the mixture after separation was conducted using ICP-MS “Element-2” with argon plasma.

2.3. Chemical separation of ^{90}Sr and ^{90}Zr in liquid ^{90}Sr - ^{90}Y β^- -source using titanium dioxide

1 ml of ^{90}Sr liquid ^{90}Sr - ^{90}Y β^- -source, was diluted with 1 ml of pure 2% HNO_3 (Optima) and 20 mg of TiO_2 was added to this solution. The separation process was performed for 120 min with weak stirring. Then the solution was filtrated and diluted 5 times. It was the “sample after separation”. The initial solution of ^{90}Sr source was diluted 10 times, and was the “unseparated sample”. Samples were analyzed by ICP-MS.

2.4. ICP-MS analysis

The mixture of investigated elements was ionized in argon plasma and after transiting through the ion optics system, ions arrive at the mass analyzer. The signal measured by the ICP-MS detector is in units of counts per second CPS. External calibration was provided using calibration Standard A solution. The inductively coupled plasma mass spectrometry was performed using mass- spectrometer “Element-2”. It has the following characteristics: dual-mode secondary electron multiplier (SEM); low dark noise: <0.2 Cps; sample times of down to 100 μs ; large SEM plateau range ~ 300 V; dynamic range 0.2 Cps - $5 \cdot 10^8$ Cps. Formation of polyatomic ions and tailing interference were controlled by measuring $^{88}\text{Sr}^{16}\text{O}$, $^{89}\text{Y}^{16}\text{O}$, and $^{90}\text{Zr}^{16}\text{O}$ amounts, as well as the formation of $^{89}\text{Y}^1\text{H}$. The efficiency of detecting investigated elements was measured twice: for initial concentration of the elements on the 10-ppb level, and initial concentrations of the elements on the 100-ppb level. The ICP-MS spectrum of the ^{90}Sr - ^{90}Y radioactive source is shown in Fig.2.

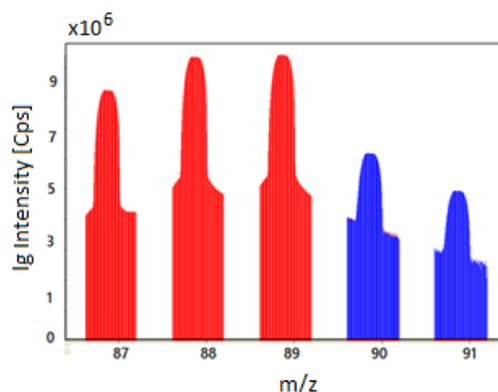


Figure 2. The ICP-MS spectrum of the ^{90}Sr - ^{90}Y radioactive source.

2.5. Liquid scintillation counting of ^{90}Sr - ^{90}Y source

The quantity of ^{90}Sr was controlled by liquid scintillation counting using a low-background liquid scintillation spectrometer-radiometer Quantulus-1220. Each of the measured samples was diluted with an organic scintillator Optiphase "HISAFE" 3 up to a volume of 20 ml and elements spectra were obtained. (During 10-30 min). The simulations of spectra ^{14}C , ^{90}Sr , and ^{90}Y were conducted, using the IAEA Library database to determine the detection conditions of β^- -spectra of the Quantulus-1220 installed in KINR NAS of Ukraine. The results are shown in Fig. 3 and Fig. 4 (a), (b).

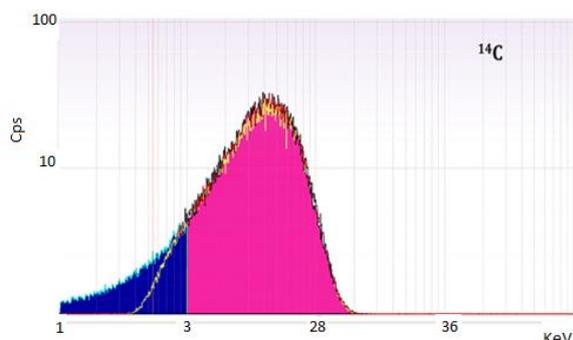


Figure 3. Simulation of the experimental ^{14}C spectrum.

The spectrum of ^{14}C , ^{90}Sr i ^{90}Y from the IAEA Library was simulated by the Monte-Carlo method (deposited spectrum). The efficiency of radionuclide detecting was calculated using the GEANT 4 [23].

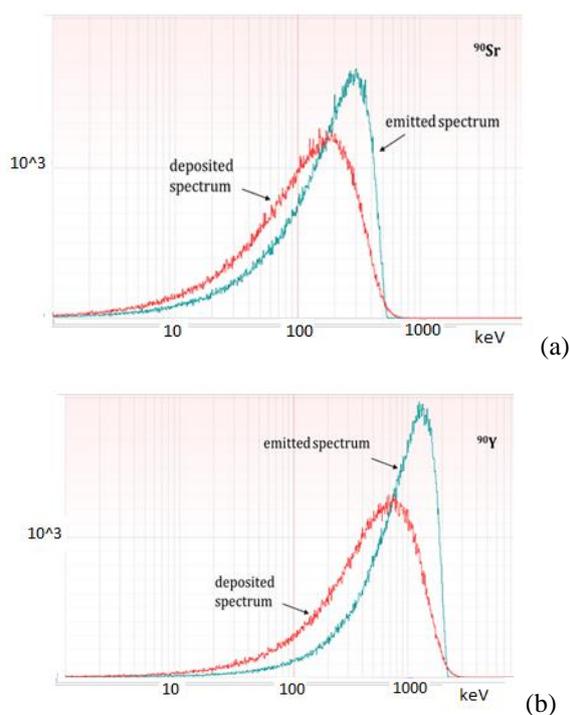


Figure 4. Comparison of the deposited spectrum and emitted spectrum: (a) for ^{90}Sr ; (b) for ^{90}Y .

The low energy region (less than 8-10 keV) of the deposited model spectrum does not correspond to the experimental one (Fig. 3). However, for the other

(higher) energy there is good correspondence between the processed spectrum of ^{14}C and emitted spectrum of ^{14}C . According to publication [8], various physical, chemical, and optical processes in the source and detector decrease the detection efficiency of low-energy particles, i.e. shifting and quenching the obtained experimentally spectrum, as can be seen in Figures 4 (a) and (b).

As can be seen, there is a little less correspondence between emitted and deposited β^- -spectra for ^{90}Sr and ^{90}Y than for ^{14}C spectra. However, this analysis allows us to consider the errors caused by quenching and shifting of the spectrum.

3. RESULTS AND DISCUSSION

3.1. Chemical separation of the stable strontium, yttrium, and zirconium ions

Dowex HCR S/S resin showed increased selectivity for zirconium cations at an initial concentration of elements of the order of 10 ng/ml. However, at higher concentrations, this adsorbent does not show selectivity for zirconium cations and adsorbs all three elements in approximately equal amounts. In general, this ion exchanger intensively absorbs yttrium cations. The Dowex 1x8 adsorbent separates strontium and zirconium due to the complete absence of adsorption of strontium cations. However, this resin together with zirconium ions adsorbs a small number of yttrium ions.

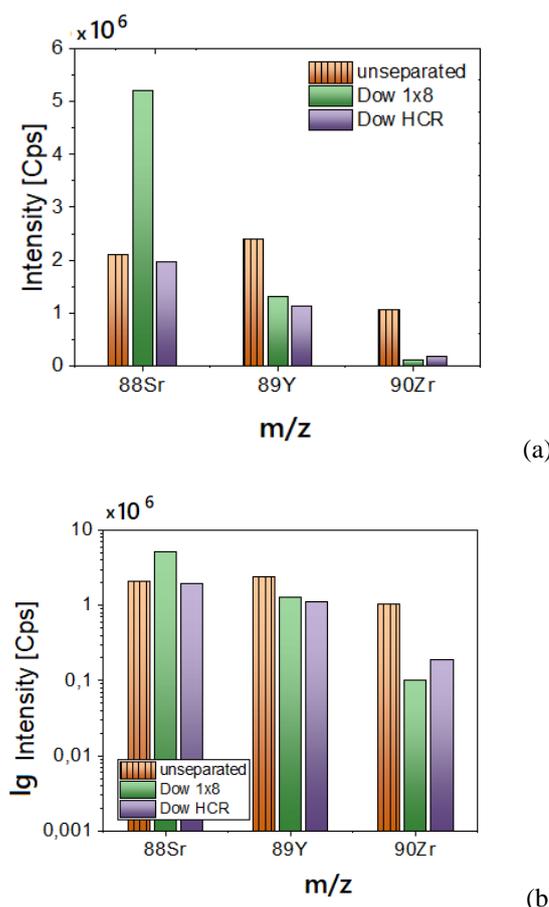
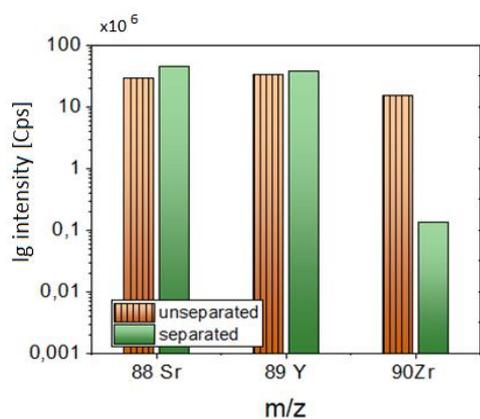


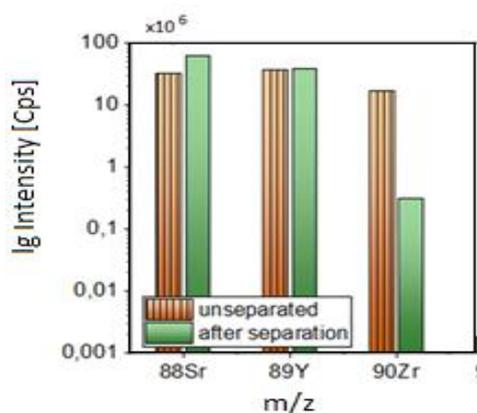
Figure 5. Results of separation by Dowex 1x8 and Dowex HCR s/s (initial concentration 10 ng/g).

The effective separation of strontium and zirconium ions by Dowex 1x8 occurs in a 5% HCl medium and for subsequent analysis using ICP-MS a solution of 5% HCl is evaporated for 5 hours, and then the dry residue is dissolved in 2% HNO₃ (Optima). The most effective adsorbent for the separation of strontium, yttrium, and zirconium ions was mesoporous titanium dioxide. This adsorbent selectively absorbs zirconium cations against the background of excess strontium and yttrium ions from 2% HNO₃ at initial concentrations of the studied cations 10 ng/ml and 100 ng/ml.

Selective zirconium ions adsorption by TiO₂ can be explained by a combination of electrostatic and steric effects. It is concluded that TiO₂ with the chemically modified surface is the most optimal material for the separation of Sr²⁺, Y³⁺, Zr⁴⁺ concentrations of 10-100 ng/ml among studied adsorbents.



(a)



(b)

Figure 6. ICP-MS spectrum of initial mixture and mixture of the elements after chemical separation. (initial concentration 100 ng/g): (a) solution volume is 7 ml, adsorbent mass is 70 mg; (b) solution volume is 2 ml, adsorbent mass is 20 mg.

3.2. Age dating of the ⁹⁰Sr-containing compound using chemical separation of strontium and zirconium ions onto titanium dioxide

Previous studies on the separation of strontium, yttrium, and zirconium using various adsorbents have shown that titanium dioxide is the most effective adsorbent tested in this work for these elements' separation. Therefore, for age dating of ⁹⁰Sr-⁹⁰Y β⁻ source the titanium dioxide was chosen. The investigated source was fabricated in 1991, so the age of investigated source was approximately 30.2 years. It should be noted, that the "age" of any radioactive device

or compound is the date of its last chemical separation [1]. The results on separation of ⁹⁰Sr and ⁹⁰Zr are shown in Figures 7 (a) and (b).

As can be seen, in the liquid ⁹⁰Sr-⁹⁰Y β⁻ source there are a lot of stable isotopes ⁸⁸Sr and ⁸⁹Y (almost 3·10⁻³ mg/ml). Despite this, the adsorbent selectively reduced the peak with a mass of 90 amu. The fact that zirconium was adsorbed can be checked by reducing the peak of ⁹¹Zr, which was present in the source, as an admixture.

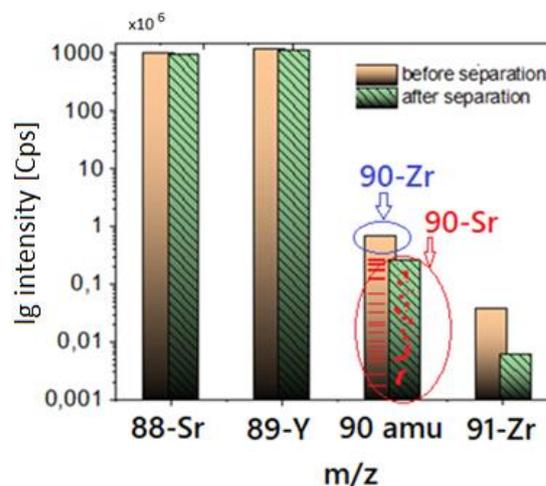


Figure 7. ICP-MS spectrum of ⁹⁰Sr-⁹⁰Y β⁻ source before and after selective adsorption of ⁹⁰Zr by the titanium dioxide.

According to Formula (1), based on Bateman equations, the age of the radioactive source can be calculated.

$$T = \frac{1}{\lambda} \ln \left(1 + \frac{N_D}{N_P} \right) \quad (1)$$

In this formula λ is ⁹⁰Sr decay constant; N_D and N_P – number of nuclei of daughter and parent nuclides, respectively.

The formation of poly ions and tailoring effects were considered during the ICP-MS analysis. The effectiveness of strontium, yttrium, or zirconium ion registration by ICP-MS was calculated using the spectra of the mixture with a concentration of 10 ppb of each ion.

Age of ⁹⁰Sr-⁹⁰Y β⁻ source, calculated using the chemical separation of ⁹⁰Sr and ⁹⁰Zr by titanium dioxide is 31.9 ± 1 year.

3.3. Age dating using a combination of liquid scintillation counting of ⁹⁰Sr and ICP-MS analysis

The β⁻ spectrum of the ⁹⁰Sr-⁹⁰Y β⁻ source was measured using the liquid scintillation spectrometer-radiometer Quantulus 1220 according to the procedure described in paragraph 2.5. The effectiveness of the registration of ⁹⁰Sr was determined. The errors caused by ⁹⁰Y, quenching, and shifting of the spectrum were considered. The mass of the sample was 0.4 g and then it was diluted 10-times, and 0.1005 g was chosen for measurement. The activity of the sample was 398 ± 2 Bq. Specific activity is 3960 ± 20 Bq/g.

In addition, the spectrum of ⁹⁰Sr-⁹⁰Y β⁻ source was measured using ICP-MS (Fig. 8). The number of ⁹⁰Zr isotopes was calculated by the difference between the

peak of 90 amu and the amount of ^{90}Sr determined by the LSC method.

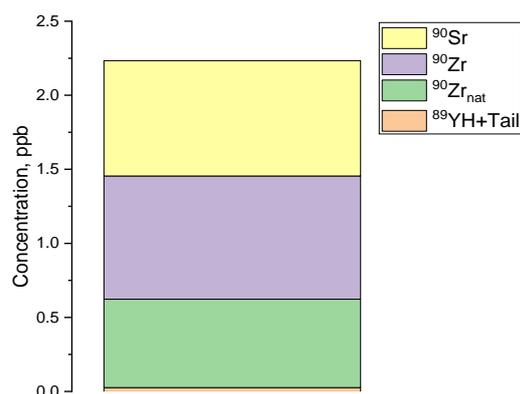


Figure 8. The concentration of ^{90}Sr , ^{90}Zr , and $^{90}\text{Zr}_{\text{nat}}$ and tailoring effects in ICP-MS peak with mass 90 amu.

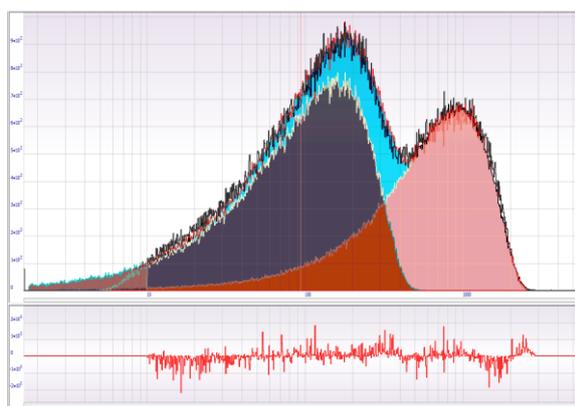


Figure 9. Liquid scintillation spectrum of ^{90}Sr - ^{90}Y liquid β^- -source.

Thus, according to the ratio of $^{90}\text{Zr} / ^{90}\text{Sr}$ (calculated using the Bateman equation) the date of production of the source was calculated. Age of ^{90}Sr - ^{90}Y β^- -source, calculated using a combination of liquid scintillation counting of ^{90}Sr and ICP-MS analysis is 31.7 ± 1.5 years. (Real ^{90}Sr - ^{90}Y source number: 488/51 was fabricated 23.04.1991; Date of age determination 24. 06. 2021). Both methods: (a) the method with chemical separation of ^{90}Zr by TiO_2 and further ICP-MS analysis of residual concentrations of ^{90}Sr , and (b) combination of LSC of ^{90}Sr and ICP-MS analysis of peak with mass 90 amu provide similar results in radio chronometry of ^{90}Sr -containing compound, i.e. age-dating of liquid ^{90}Sr - ^{90}Y β^- -source, and could validate each other. However, the method with chemical separation (method (a)) will be preferable for the age dating of the younger ^{90}Sr radioactive source. A very large amount of ^{90}Sr (natural ^{90}Zr and $^{89}\text{Y}^1\text{H}$) compared with a small amount of the radiogenic ^{90}Zr will increase the uncertainty, and add some limitations in the application of method (b).

4. CONCLUSION

The results of the present work have shown, that Dowex HCR S/S resin showed increased selectivity for zirconium cations at an initial concentration of

elements of the order of 10 ng/ml. However, at higher concentrations, this adsorbent does not show selectivity for zirconium cations and adsorbs all three elements in approximately equal amounts. In addition, this ion exchanger intensively absorbs yttrium cations. The Dowex 1x8 resin separates strontium and zirconium. However, this resin together with zirconium ions adsorbs significant amounts of yttrium ions.

It is concluded that TiO_2 is the most optimal material for the chemical separation of Sr^{2+} , Y^{3+} , Zr^{4+} from their mixture with concentrations of 10-100 ng/ml at $\text{pH}=0-1$, among studied adsorbents.

Age of ^{90}Sr - ^{90}Y β^- -source, calculated using the method of the chemical separation of ^{90}Sr and ^{90}Zr by the titanium dioxide and further ICP-MS analysis is 31.9 ± 1 year.

Age of ^{90}Sr - ^{90}Y β^- -source, calculated using a combination of liquid scintillation counting of ^{90}Sr and ICP-MS analysis 31.7 ± 1.5 years. (Real ^{90}Sr - ^{90}Y source number: 488/51 was fabricated 23.04.1991). Both methods provide similar results in radio chronometry of ^{90}Sr -containing compound, i.e. age-dating of liquid ^{90}Sr - ^{90}Y β^- -source, and could validate each other. However, the method with chemical separation (method (a)) will be preferable for the age dating of the younger ^{90}Sr radioactive source. A very large amount of ^{90}Sr (natural ^{90}Zr and $^{89}\text{Y}^1\text{H}$) compared with a small amount of the radiogenic ^{90}Zr will increase the uncertainty, and add some limitations in the application of method (b).

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