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Tracer Studies on Sr Resin and Determination of ^{90}Sr in Environmental Samples

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Abstract

In this work various tracer experiments were performed to investigate the elution behaviour of some radionuclides on Sr resin, matrix effects of calcium and potassium and the resin capacity for strontium. None of the studied radionuclides interfered with the separation of strontium since all were washed out with 3M HNO_3 before the elution step with water, except barium. Barium and strontium were separated using 8M HNO_3. The maximum resin capacity for strontium was 8.1 mg of Sr/g of Sr resin. Up to 2 g of calcium and 200 mg of potassium can be loaded on the Sr column (3 g Sr.spec) without decrease in strontium recovery. The separation procedure using Sr resin was tested on soil samples spiked with ^{90}Sr standard solutions and on reference materials. All results obtained by this method gave good agreement. Counting sources were prepared by weighing SrC_2O_4 on a planchet. The ^{90}Sr content was measured with a proportional gas flow beta counter after secular equilibrium between ^{90}Sr and ^{90}Y was reached. The chemical recovery was determined gravimetrically and was always higher than 80%.

Key words: Sr resin, extraction chromatography, ^{90}Sr, beta counting

Introduction

^{90}Sr is created in nuclear fission processes and it has been released to the environment with global fallout following atmospheric nuclear explosions, by nuclear waste discharges and by the Chernobyl nuclear power plant accident in 1986. ^{90}Sr is radiotoxic because of its relatively long physical (28.6 y) and biological (49.3 y) half-life and because of chemical similarities with calcium. It accumulates mainly in bone tissues where its daughter ^{90}Y radiates beta particles of relatively high end-point energy (E_{max} = 2.27 MeV).[1]

Both radionuclides ^{90}Sr and ^{90}Y are pure beta emitters with maximum energies of 546 keV and 2.27 MeV respectively, and they cannot be identified by direct measurements via gamma spectrometry. Procedures for determination of ^{90}Sr are complicated and involve an extensive radiochemical separation before measurement to eliminate inactive and radioactive elements present in the sample that would interfere with subsequent beta counting. Among inactive interferences calcium is the most significant since it is similar to strontium and is present in environmental samples in large amounts.[2,3] After separation and purification ^{90}Sr is quantified by beta counting of the total activity of ^{90}Sr and ^{90}Y, after secular equilibrium is reached, with a proportional gas flow counter or by liquid scintillation.

Different methods have been described in the literature for the determination of ^{90}Sr. The oldest is the fuming nitric acid method, which relies on the better solubility of calcium nitrate in strong nitric acid compared to strontium nitrate. However, it involves numerous precipitation and filtration steps and is thus time consuming. Many other methods have been proposed that offer less aggressive conditions.[4] These include liquid-liquid extraction[3,5,6] and ion exchange.[7–9] Recently, solvent extraction based on the macro-cyclic polyether 18-crown-6 for the selective extraction of strontium has been proposed, with different derivative compounds.[10–12] The resolution of strontium from other elements, particularly barium and calcium, was not satisfactory using liquid-liquid extraction as the only step for separation because of the low stability of crown ether complexes. Horwitz et al.[13,14] overcame this problem by development of a Sr resin (Sr.spec resin, Eichrom® Industries). The Sr resin consists of the crown ether 4,4´(5´)-bis(tert-butylcyclohexano)-18-crown-6 dissolved in 1-octanol and sorbed on an inert polymeric support. It possesses the selectivity of solvent extraction and the ease of column chromatography. Many authors have reported methods using Sr resin for strontium separation in various environmental samples.[1,2,15–20] Procedures using Sr resin are simpler, shorter, result in higher recoveries and more reproducible results.

The aim of this study was to study the behaviour of some radionuclides on Sr resin and to test the suitability of Sr resin for separation of strontium from solutions containing different amounts of potassium and calcium. Further, ^{90}Sr in some reference materials was determined by the procedure developed by Vajda et al.[20] which we slightly modified. Before the elution of strontium with deionised water a wash with 8M HNO_3 was added in order to separate strontium from barium completely and for the measurement a proportional gas flow beta counter was used instead of liquid scintillation.

Experimental

a) Materials and instrumentation

Reagents: All the reagents used were of analytical grade.

Tracers: ^{85}Sr 435.9 kBq g^{-1}, (reference date: 30.11.2001), ^{133}Ba 124.9 kBq g^{-1}, (reference date: 30.4.1999) obtained from the Czech Metrological Institute, Czech Republic, ^{88}Y 59.1 Bq g^{-1} (reference date: 9.12 2002) and ^{137}Cs 58.7 Bq g^{-1} (reference date: 9.12.2002) obtained from LEA, France. ^{212}Pb tracer was prepared from $Th(NO_3)_4 \cdot 4H_2O$ solution by extraction with dithizone in chloroform. A ^{90}Sr/^{90}Y standard solution of 48.2 Bq g^{-1} from LEA, France was used for spiking experiments.

Samples: The reference materials IAEA 152 (milk powder), IAEA 154 (whey powder), IAEA 156 (clover), IAEA 375 (radionuclides in soil) and Soil-6 were analysed.

Detectors: For gamma measurements an HP Ge Low Energy Photon Detector connected to a Canberra MCA by a Genie-2000 Software multi-channel analyser system was used. For beta counting of ^{90}Sr and ^{90}Y a Berthold MULTI-LOGGER LB 5310 proportional gas flow beta counter was used.

b) Sr column preparation

The Sr columns were prepared by soaking approximately 3 g of Sr.spec (100–150 μm) chromatographic material in deionised water for 1 h and then packing it into a 20 cm long and 1 cm in diameter glass chromatographic column (Bio-Rad Laboratories). The column was washed with 100 mL of deionised water and pre-conditioned with 70 mL of 3M HNO_3. For column regeneration 100 mL of deionised water and then 60 mL of 0.1M EDTA solution were passed through the column.

c) Elution behaviour of Sr, Cs, Y, Ba and Pb

^{85}Sr, ^{137}Cs, ^{88}Y, ^{133}Ba and ^{212}Pb tracers were added to a solution of 3M HNO_3. The solution was first measured on a gamma detector to determine the initial activity of each tracer and then loaded onto the top of the Sr column. The column was washed with nitric acid of different concentrations and with deionised water. Fractions were collected and measured on the gamma detector. The percentage of tracer in each fraction was determined relatively by comparison with the initial activity of each tracer.

d) Calcium and potassium matrix effects

Solutions of 3M HNO_3 containing ^{85}Sr tracer, 10 mg of Sr^{2+} carrier as $Sr(NO_3)_2$ and different amounts of Ca^{2+} or K^+ were prepared. The initial activity of ^{85}Sr was measured on the gamma detector at 514 keV. The solution was loaded onto the Sr column, washed with 3M HNO_3 and the strontium was eluted with deionised water. Fractions were collected and measured on the gamma detector. The percentage of ^{85}Sr in each fraction was determined relatively by comparison with the initial activity.

e) Column capacity for Sr

Solutions containing ^{85}Sr tracer and 100 mg of Sr^{2+} carrier (in excess) in 20 mL of 3M HNO_3 were prepared. The solution was loaded onto the column (3 g of Sr.spec) and washed with 3M HNO_3. Fractions were collected and measured on the gamma detector. The percentage of ^{85}Sr found in the water fraction was used for the determination of the column capacity for strontium. In each experiment the same Sr column was used.

f) The stoichiometry of SrC_2O_4

Solutions containing ^{85}Sr tracer, 10 mg of Sr^{2+} carrier and 10 mg of Ca^{2+} carrier in 3M HNO_3 were passed through the Sr column. The strontium in the water fraction was precipitated as strontium oxalate (see section h) below), filtered on a weighed filter paper and dried.

g) Sample preparation

Soil and biological samples were dried at 105 ºC and then ashed in a furnace at 550 °C. About 1–3 g of ash was then used for the analysis. To each sample, 10 mg of Sr^{2+} carrier as strontium nitrate was added for chemical recovery determination. Biological samples (1–3 g) were leached with up to 100 mL of 8M HNO_3 in a covered beaker on a hot plate with magnetic stirring. The leachant and the residue were separated by filtration through a 0.45 μm filter. For soil samples (1 g) total dissolution was used. The soil samples in Teflon® beakers were treated by repeated addition of 63% HNO_3, 40% HF and 37% HCl. First 5 mL of 63% HNO_3 were added and the solution was heated and evaporated to dryness. Next 5 mL of 63% HNO_3 and 5 mL of 40% HF were added. Again the solution was heated and evaporated to dryness. In the last step 5 mL of 63% HNO_3, 5 mL of 40% HF and 5 mL of

37% HCl were added. After evaporation of the final solution to near dryness, 0.3 g of H_3BO_3 was added and evaporated to dryness. The heating and the evaporation of the solutions were performed in an aluminium block at 220 °C. The final residue was converted to nitrate form by evaporating it twice with 3 mL of 63% HNO_3, dissolved in 100 mL of 1M HNO_3 and filtered through a 0.45 μm filter.

h) Separation and determination of strontium[20]

The filtrate was then heated to boiling and approximately 5 g of oxalic acid were added. The pH of the solution was adjusted to 5.5–6 with addition of sodium hydroxide. Depending on the matrix composition, additional Ca^{2+} was sometimes added (up to 300 mg) in order to obtain the oxalate precipitate. The suspension was centrifuged in a 100 mL centrifuge tube for 10 min at 3000 rpm (Tehtnica Železniki). The oxalate precipitate was washed three times with 70 mL of deionised water. The oxalate was then destroyed by evaporating it twice with 5 mL of 63% HNO_3. The residue was dissolved in 30 mL of 3M HNO_3 and loaded onto the Sr resin. After washing the column with 100 mL of 3M HNO_3 and 60 mL of 8M HNO_3, strontium was stripped from the column with 100 mL of deionised water and the time was noted. The water fraction was evaporated to about 20 mL and 200 mg of oxalic acid was added. Then the pH of the solution was adjusted to 9–10 with 25% NH_3. The solution was boiled for 10 minutes and SrC_2O_2 began to form. After cooling the solution, the precipitate was centrifuged on a measuring planchet (19 mm in diameter), dried, weighed for recovery determination and stored for $^{90}Sr/^{90}Y$ equilibrium for at least 14 days. The beta activity was measured with a gas flow proportional beta counter.

Results and discussion

a) Elution behaviour of Sr, Cs, Y, Ba and Pb

The determination of ^{90}Sr in environmental samples requires its chemical isolation from all interfering elements, especially alkaline and alkaline-earth elements. Among them calcium is extremely difficult to separate due to its similar chemical behaviour with strontium. These can cause three types of interference: firstly with determination of the chemical yield by gravimetry (Ca, Ba), secondly by mass absorption effects in the final precipitate during beta counting (Ca, Ba) and thirdly by radioactive interference in counting (^{40}K, $^{137,134}Cs$, ^{140}Ba). Figure 1 shows three elugrams describing the behaviour of ^{88}Y, ^{137}Cs, ^{133}Ba, ^{212}Pb and ^{85}Sr on a Sr column, filled with 3 g of Sr.spec chromatographic material. The gravity flow rate was 1–2 mL min^{-1}. Figure 1a shows the elution curves obtained when ^{88}Y, ^{137}Cs, ^{133}Ba and ^{85}Sr were loaded on the column in 3M HNO_3. Both caesium and yttrium were washed from the column in the first 60 mL of 3M HNO_3. This behaviour is due to the low distribution coefficient for caesium in 3M HNO_3 ($K_D \approx 10^{-1}$).[13] Most of the barium, however, is retained by the column during the loading and initial rinsing, and eluted as a broad peak. It started to elute at 120 mL and was washed from the column after approximately 220 mL of 3M HNO_3, but some fraction of the barium appeared in the water eluate. Strontium eluted rapidly in the first 20 mL of deionised water. We further investigated the behaviour of strontium and barium, because they could not be separated completely with

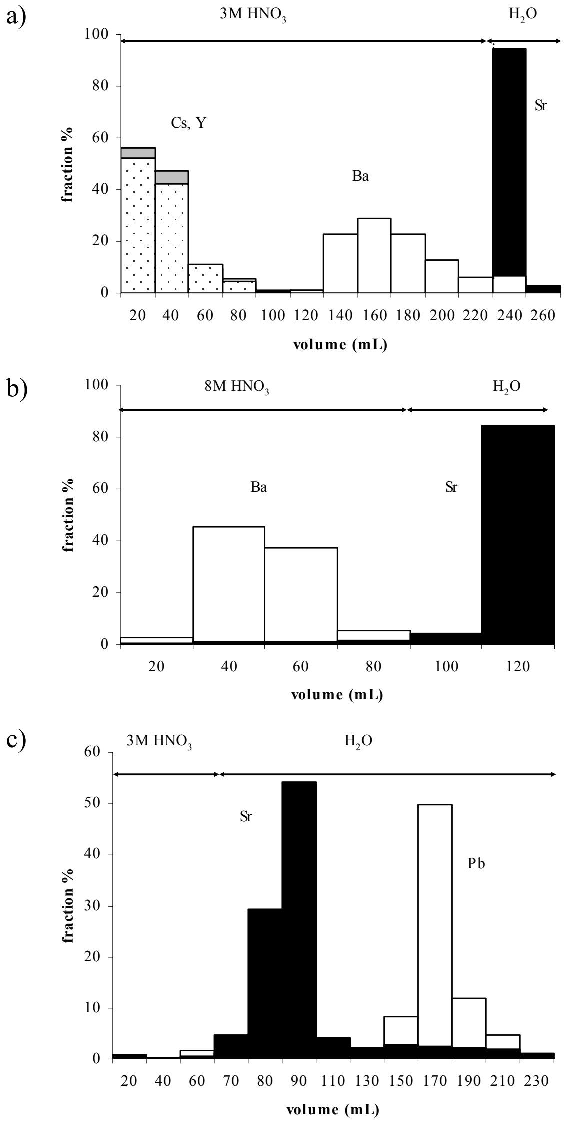

Figure 1. a) Elution curves of ^{85}Sr, ^{133}Ba, ^{88}Y and ^{137}Cs on Sr resin column in 3M HNO_3 as loading solution (3 g Sr.spec material in column Φ = 1 cm, l = 20 cm, flow rate = 1–2 mL min^{-1}). b) Elution curves of ^{85}Sr and ^{133}Ba on Sr resin column in 8M HNO_3 as loading solution (3 g Sr.spec material in column Φ = 1 cm, l = 20 cm, flow rate = 1–2 mL min^{-1}). c) Elution curves of ^{85}Sr and ^{212}Pb on Sr resin column in 3 M HNO_3 as loading solution. (3 g Sr.spec material in column Φ = 1 cm, l = 20 cm, flow rate = 1–2 mL min^{-1}).

3M HNO_3 solution. This time strontium and barium were loaded on the column in 8M HNO_3 where the distribution coefficient for barium is minimal ($K_D \approx 6$) and for strontium maximal ($K_D \approx 200$).[13] Elution curves are presented in Figure 1b. Barium started to elute quickly, after the first 20 mL of 8M HNO_3 and was completely removed after 80 mL of 8M HNO_3. The barium peak was narrower compared to the peak in the elugram in Figure 1a and no barium was detected in the strontium fraction. Finally, strontium was eluted with deionised water. Lead showed a very high retention on the Sr column, even more than strontium. It has a high distribution coefficient in the whole interval of nitric acid concentrations ($K_D \approx 10^2$–10^3).[13] Figure 1c shows the elution curves of strontium and lead in 3M HNO_3. The stripping solution was changed to deionised water and strontium and lead were sequentially eluted from the column. The Sr column has such a strong affinity for lead that it was eluted after strontium and the interference of lead on strontium was minimal.

b) Column capacity for strontium

The purpose of this experiment was first to find the maximum amount of strontium that can be loaded on the column and used for determination of the recovery of the radiochemical procedure, and to see if the column capacity changes when the column is used repeatedly. Repeated sorptions and elutions of ^{85}Sr tracer and Sr^{2+} carrier were made on the same column. The results are presented in Figure 2.

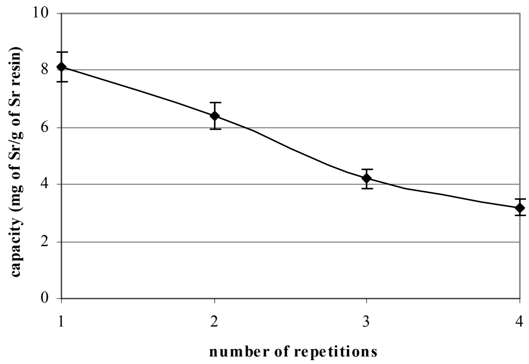

Figure 2. Dependence of the column capacity for strontium on the number of repetitions (3 g Sr.spec material in column Φ = 1 cm, l = 20 cm, flow rate = 1–2 mL min^{-1}, average uncertainty = 8%).

From the graph it can be seen that the column capacity decreased with use, but not linearly. The maximum capacity of the column for strontium was estimated to about 8.1 ± 0.5 mg of Sr per g of Sr resin. After the fourth repetition the capacity was only 3.2 ± 0.3 mg, which is 40% of the initial one. This value is three times less than the value stated by the producer. This feature could not be explained. A similar result (8.8 mg/g of Sr resin) was obtained by Torres et al.[18] Some authors believe that reason for the decrease is probably due to decomposition and removal of the stationary phase from the column, as well as possible destruction of the crown ether by the strong nitric acids used in the separation step.[21] In practice we used each column three times.

c) Matrix effect

Calcium has a small distribution coefficient in 3M HNO_3 ($K_D \approx 10^{-2}$)[13] but can be present in large amounts in some samples. To test the effect of calcium on retention and recovery of strontium, solutions with varying amounts of calcium were loaded on the column in 3M HNO_3. Figure 3 shows the elution curves obtained in this experiment. In Figure 3a the amount of calcium ranged from 100–1200 mg. After approximately 100 mL of 3M HNO_3, the column was eluted with deionised water. All the strontium was found in water fraction and none was detected in 3M HNO_3 fraction. Then a higher concentration of calcium was again loaded in 3M HNO_3. This is shown in Figure 3b. This time the washing with 3M HNO_3 was prolonged (200 mL) in order to see if any strontium appeared in that fraction. In fact about 20% of strontium was found in 3M HNO_3 when the amount of calcium was 2500 mg.

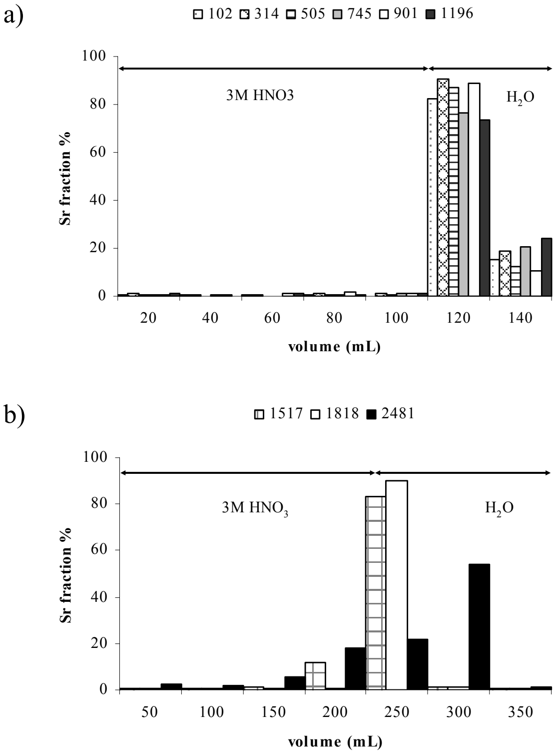

Figure 3. a) Elution curves of ^{85}Sr with 10 mg of Sr^{2+} carrier and different amounts of Ca^{2+} carrier (100–1200 mg) in 3M HNO_3 solution (3 g Sr.spec material in column Φ = 1 cm, l = 20 cm, flow rate = 1–2 mL min^{-1}, average uncertainty = 8%). b) Elution curves of ^{85}Sr with 10 mg of Sr^{2+} carrier and different amounts of Ca^{2+} carrier (1500–2500 mg) in 3M HNO_3 solution (3 g Sr.spec material in column Φ = 1 cm, l = 20 cm, flow rate = 1–2 mL min^{-1}, average uncertainty = 8%).

In Figure 4 the effect of calcium matrix on strontium recovery is presented. Practically no change in recovery up to 2000 mg is observed. The recoveries ranged from 93–100%. When the calcium content was above 2000 mg, the recovery for strontium started to decrease and dropped to 40% for 2800 mg of calcium.

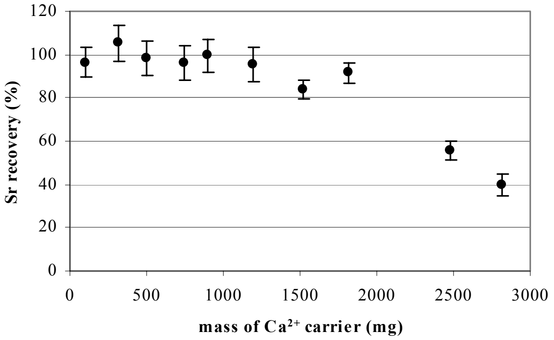

Figure 4. Calcium matrix effect on strontium chemical recovery (average uncertainty = 8%).

The same experiment was performed with potassium. Potassium, however, has a greater influence on strontium retention, as can be seen from elution curves in Figure 5. For up to 200 mg of potassium, strontium is detected only in the water fraction. Some strontium is detected in 3M HNO_3 but this is due to experimental error. The recoveries for strontium were 96–100%. When higher amounts of potassium were loaded on the column, some losses of strontium occured during the washing with 3M HNO_3. The recovery was 88% for 240 mg of potassium and 40% for 940 mg of potassium.

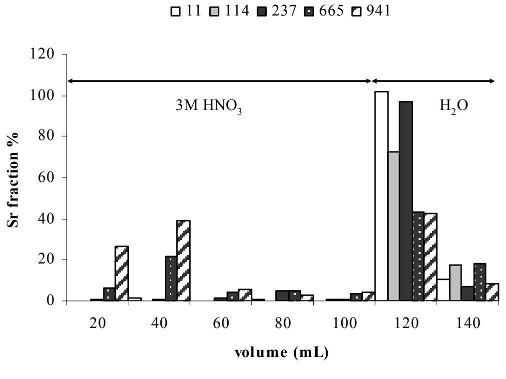

Figure 5. Elution curves of ^{85}Sr with 10 mg of Sr^{2+} carrier and different amounts of K^+ carrier (10–1000 mg) in 3M HNO_3 solution (3 g Sr.spec material in column Φ = 1 cm, l = 20 cm, flow rate = 1–2 mL min^{-1}, average uncertainty = 13%).

When determining strontium in environmental samples losses of strontium can be expected due to matrix elements present, but this is accounted for by measuring the chemical recovery at the end of the radiochemical analysis. Separation from potassium is accomplished by oxalate precipitation, where potassium remains in solution.

d) Recovery determination and the stoichiometry of SrC_2O_4

Chemical recovery in ^{90}Sr determination was determined by weighing of SrC_2O_4 precipitate. We need to be sure that the precipitate has a stoichiometric composition to avoid errors in calculating the recovery and consequently the final result. The stoichiometry of the precipitate was checked by comparison of the gravimetric and radiochemical tracer recovery. The results are shown in Table 1.

Table 1. Comparison between gravimetric and radiochemical recovery of Sr on the same Sr column.

	Gravimetric recovery (%) SrC_2O_4	Radiochemical recovery (%) ^{85}Sr
1	81.4 ± 0.9	79.9 ± 2.6
2	77.5 ± 0.8	76.3 ± 2.5
3	54.9 ± 0.6	54.1 ± 2.0
4	36.7 ± 0.4	36.3 ± 1.3

A good match between the gravimetric and radiochemical recoveries was found. The quotient of the gravimetric and radiochemical recovery was calculated and the average was found to be 1.015 ± 0.037. This result confirms the stoichiometry of the SrC_2O_4 precipitate. The decrease in recovery was due to the sequential decrease in the column capacity.

e) ^{90}Sr in some soil and biological samples

Before analysing reference materials, we spiked six soil samples by addition of a ^{90}Sr standard solution of a known activity. The solutions were left to stand overnight and analysed according to the analytical procedure described. The aim of this experiment was to study the effect of matrix elements on strontium determination. The activities of the added ^{90}Sr ranged from 1–12 Bq and the masses of soil from 2–3 g. A good linear correlation between the measured activity and the activity of the added ^{90}Sr was obtained. The quotient was 1.0189 ± 0.01147. The R^2 of linear regression analysis based on the method of least squares was 0.9988.

Table 2. Comparison of ^{90}Sr results obtained in reference materials with certified values.

Sample	This work^c (Bq kg^{-1}dry)	Chem. recovery (%)	Certified value (Bq kg^{-1}dry)
IAEA-152^a	7.4±0.5 (2)	83.9±1.0	7.7±0.6
IAEA-154^a	6.8±0.8 (3)	95.4±1.0	6.9±1.0
IAEA-156^a	14.8±0.9 (3)	79.9±0.9	14.8±1.5
IAEA-375^b	113±5 (4)	82.4±0.9	108±4
Soil-6^b	28±4 (3)	80.9±0.9	30.3±6.5

^{a} leaching. ^{b} total dissolution. ^{c} average ± 1σ standard deviation (number of determinations).

The method was then tested by analysing several reference materials. The results obtained are compared to the reference values in Table 2. The activity of ^{90}Sr in the sample was calculated by Equation (1).

$$A_{Sr} = \frac{(R - R_b)}{60 \cdot t \cdot \eta \cdot m \cdot \left[\varepsilon_{Sr} + \varepsilon_Y \cdot \left(1 - e^{-t_d \cdot \lambda}\right)\right]} , \quad (1)$$

where t is the counting time (min); η is the overall chemical recovery; m is the mass taken for the analysis (kg); ε_{Sr} and ε_Y are the counting efficiencies for strontium and yttrium, respectivelly; t_d is the time allowed for ^{90}Y ingrowth (min); λ is the decay constant of ^{90}Y (min^{-1}); R is the count rate of the sample and R_b is the count rate of the background (min^{-1}). The background of the proportional gas flow counter was about 0.25 counts min^{-1}.

The uncertainty was given by Equation (2).

$$\sigma \approx A_{Sr} \cdot \sqrt{\sigma_\eta^2 + \sigma_\varepsilon^2 + \sigma_R^2} , \quad (2)$$

where σ_η is the uncertainty of the chemical recovery; σ_ε is the uncertainty of detector efficiency and σ_R is the uncertainty of the count rate of the sample.

Good agreement was found in all cases. The recoveries were always higher than 80%.

Conclusions

Our tracer studies showed that Sr resin can be successfully used for separation of strontium from matrix elements and from interfering radionuclides. Among tested elements, only barium and lead were retained on the column. Barium was removed by washing the column with 8M HNO_3 while lead eluted after strontium due to its higher affinity. The main disadvantage is the low column capacity for strontium which is 8.1 mg/g Sr resin and decreases with use. This limits the reuse of the column. It was also shown that calcium and potassium had negative effects on strontium retention if the amounts were above 2 g for calcium and 200 mg for potassium for a column containing 3 g of Sr.spec chromatographic material. The results for ^{90}Sr in reference samples were in good agreement with their certified values. Recoveries ranged from 80–95%.

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Povzetek

Izvedli smo različne elucijske teste s sledilci, da bi raziskali obnašanje nekaterih radionuklidov na Sr koloni, določili kapaciteto Sr kolone za stroncij in matrični vpliv kalcija ter kalija na vezavo stroncija. Noben od proučevanih radionuklidov ni motil separacije stroncija, saj so se vsi razen barija sprali s kolone s 3 M HNO_3. Barij in stroncij smo ločili z 8 M HNO_3. Sr kolona prenese do 2 g kalcija in do 200 mg kalija, večje količine pa zmanjšajo izkoristek za vezavo stroncija. Metodo smo testirali z analizo prsti z dodatkom ^{90}Sr in z analizo referenčnih materialov. Vsi rezultati so se dobro ujemali s certificiranimi vrednostmi. Izvor za merjenje smo pripravili s tehtanjem SrC_2O_4 na ploščico. Vsebnost ^{90}Sr smo določili z merjenjem s pretočnim beta števcem po vzpostavitvi sekularnega ravnotežja med ^{90}Sr in ^{90}Y. Izkoristek postopka smo določili gravimetrično in je bil vedno večji od 80 %.