

Isolation of Yttrium and Strontium from Soil Samples and Rapid Determination of ^{90}Sr

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A procedure of yttrium and strontium separation from calcium and other cations has been developed for rapid determination of ^{90}Sr in soil samples. The procedure involves yttrium, strontium and other cations bound on cation exchanger AMBERLITE IR-120, separation of yttrium and strontium from other cations, *e.g.* potassium, calcium, sodium, barium, on anion exchangers AMBERLITE CG-400 or DOWEX AG 1×8 with $0.25 \text{ mol l}^{-1} \text{ HNO}_3$ in alcohol mixture as eluent, separation of yttrium from strontium on cation exchanger DOWEX 50×8 with $3 \text{ mol l}^{-1} \text{ HNO}_3$, and determination of ^{90}Sr on the low-level gas-flow β -counter and by Cherenkov counting on the liquid-scintillation counter. It has been shown that this procedure might be successfully applied in rapid determination of ^{90}Sr , in a timesaving manner and without application of strong, concentrated acids. The method is appropriate for determination of the water soluble fraction of ^{90}Sr in different kinds of environmental samples (milk, water, wine, vegetables, *etc.*) with some modification of the sample preparation procedure.

Key words: radioactive strontium, rapid determination, yttrium, strontium, isolation.

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INTRODUCTION

Radioactive isotope ^{90}Sr belongs to the extremely radiotoxic group of isotopes. This isotope is deposited in bones with a high risk of development of osteosarcoma or leukaemia. Therefore, the maximum tolerated level of annual strontium uptake is extremely low and availability of very sensitive reliable determination methods is necessary.

The methodology of ^{90}Sr determination is largely determined by its radiochemical properties. Namely, ^{90}Sr is a pure β -emitter and cannot be determined without being previously chemically isolated from the samples. Its chemical isolation and determination is rather time-consuming and requires application of various chemical and other techniques.

In the environmental sample analysis, large amounts of samples are needed due to the fact that the samples usually contain very small amounts of ^{90}Sr (except for some extreme cases). Since soil samples contain large amounts of calcium, sodium, aluminium, iron and many other elements in smaller amounts, which also include radioactive isotopes of natural and artificial origin (^{40}K , ^{137}Cs , ^{232}Th etc.), it is necessary to separate in a simple and rapid way small amounts of strontium from the complex matrix of elements in most diverse concentration ratios. Several methods have been developed for the determination of ^{90}Sr in natural samples.¹⁻⁹ However, the most commonly applied one is a standard procedure in which the samples are treated with various strong acids while concentrated nitric acid is used for the separation of large amounts of calcium from small amounts of strontium. Such a procedure, however, is rather time-consuming and due to the use of aggressive chemicals, requires special precaution measures during the process.

Therefore, in order to avoid the use of strong, concentrated acids as well as to simplify and speed up the ^{90}Sr determination procedure in soil samples, a new procedure of radioactive strontium isolation was developed on the basis of our previous works, in which strong anion exchangers and an alcohol solution of nitric acid were used for the separation of calcium and strontium and for the isolation of ^{90}Sr .¹⁰⁻¹² In order to develop a simple isolation procedure, examination with model systems was applied. After that the isolation and rapid determination procedure of ^{90}Sr in soil samples using available detection techniques was created. Determination of ^{90}Sr in soil samples was carried out following both the standard procedure² and the newly developed procedure, upon which the results of such determination were compared.

EXPERIMENTAL

Chemicals

Anion exchangers DOWEX AG 1 × 8 (100 to 200 mesh) BioRad Lab., AMBERLITE CG-400 (100 to 200 mesh) Rhom Haas in Cl⁻ form, cation exchangers DOWEX 50 × 8 (100–200 mesh) Fluka Chemie and AMBERLITE IR-120 (>250 μm) BioRad Lab. in Na⁺ form were used. Calcium, strontium, barium, potassium, sodium and yttrium nitrate, Merck Darmstad, were used. All chemicals were of analytical grade. The standard solution of ⁹⁰Sr activity 17 Bq ml⁻¹ was received from Amersham, U.K. The cation solutions used were prepared from redistilled water and alcohols of analytical grade. Alcohol solutions of nitric acid were prepared from 5 mol l⁻¹ HNO₃ and absolute methanol and absolute ethanol.

Instruments

The atomic absorption spectrometer PERKIN ELMER MODEL 2110, the low-level gas-flow β-counter CANBERRA 2400 and the liquid-scintillation counter PAC-KARD TRI CARB 2770 TR/SL were applied.

Description of Model Experiments

Preparation of Model Solutions

Model solutions of cation mixtures for separation on anion exchanger columns were prepared by dissolving nitric salts of potassium, caesium, sodium, iron, yttrium, strontium and calcium in absolute methanol with the cation concentration amounting to 1 mg ml⁻¹. For separation in the cation column, a water solution of yttrium and strontium in the concentration of 1 mg ml⁻¹ was prepared.

Preparation of Exchangers and Columns

Anion exchangers AMBERLITE CG-400 and DOWEX AG 1 × 8 in Cl⁻ form were washed in redistilled water and dried at 70 °C. After that, 5 g of exchanger were put in columns of 1 cm diameter and 25 cm height. The exchanger was converted to nitric form with 5 mol l⁻¹ HNO₃ to H⁺ form with 5 mol HNO₃. Column dimensions and exchanger quantities were the same as in the case of anion exchangers.

Strontium Separation from Other Cations

Strontium and yttrium were separated from other cations on anion exchanger columns by eluting with 0.25 mol l⁻¹ HNO₃ in methanol, with 0.25 mol l⁻¹ HNO₃ in ethanol, with 0.25 mol l⁻¹ HNO₃ in methanol–ethanol mixture (140 ml methanol + 50 ml ethanol + 10 ml 5 mol l⁻¹ HNO₃) and with water. In the cation column, strontium was separated from yttrium by eluting with 3 mol l⁻¹ HNO₃. Eluting was followed by taking fractions and measuring the concentration of a particular cation in the fraction with the atomic absorption spectrometer.

Determination of Strontium in Soil Samples

Preparation of Reference Soil Samples

In ⁹⁰Sr determination, soil samples were used to which a standard solution of ⁹⁰Sr was added. Activity of ⁹⁰Sr was 17 Bq ml⁻¹. Prior to addition of an aliquot stan-

dard solution of ^{90}Sr (0.2 and 0.3 ml of ^{90}Sr standard solution), the samples were dried, ground and sieved through a 250- μm sieve. 50 g of sieved soil was taken and mixed with 200 ml of water and the standard ^{90}Sr solution under intensive stirring. Two soils were used to prepare reference samples. The prepared sample was stored for two months to dry in air. Before the analysis, the soil sample was dried at a temperature of 105 °C and 10 g of each sample was taken for the analysis. Such preparation of referent samples is appropriate for the scenario of contamination with the easily soluble form of ^{90}Sr .

New Procedure For Yttrium And Strontium Separation From Soil Samples

Cations Leaching with Ion Exchanger. – Ten grams of soil is put in 250-ml PTFE beaker and mixed with 150 ml of redistilled water. To this suspension, 1 ml of strontium carrier solution 20 mg ml $^{-1}$ and 1 ml of the yttrium carrier solution 10 mg ml $^{-1}$ are added and intensively stirred. After that, 10 g of the exchanger AMBERLITE IR-120 in H $^{+}$ form is added to the suspension. The exchanger particles should be larger than 250 μm . The exchanger is previously washed in redistilled water and, if necessary, converted to H $^{+}$ form. The exchanger and the sample are intensively stirred by introducing a nitrogen stream into the suspension. The exchanger is separated from the soil on a 250 μm sieve by washing under a stream of redistilled water and put into a glass column. Bound cations are eluted with 200 ml 5 mol l $^{-1}$ HNO $_3$ and evaporated to dryness. The obtained nitric salts of such cations are dissolved in a minimum quantity of redistilled water. Ten milliliters of absolute ethanol and 40 ml of absolute methanol are added to the water solution. This sample solution is ready for isolation in the anion exchange column.

Isolation in the Anion and Cation Exchange Column. – Alcoholic sample solution is passed through the 40 cm high column of 1 cm diameter, filled with 12 g of the exchanger AMBERLITE CG-400 (or DOWEX AG 1 \times 8) at a flow rate of 1 ml min $^{-1}$. The column is to be prepared for separation in the above-described manner. After that, calcium, iron and other elements are separated from yttrium and strontium by the flowing of 200 ml 0.25 mol l $^{-1}$ HNO $_3$ in ethanol–methanol mixture (10 ml 5 mol l $^{-1}$ HNO $_3$ + 50 ml absolute ethanol + 140 ml absolute methanol) at a flow rate of 1–3 ml min $^{-1}$. Strontium and yttrium are eluted with 350 ml 0.25 mol l $^{-1}$ HNO $_3$ in methanol. The eluted yttrium and strontium are passed through the column filled with 5 g of the cation exchanger DOWEX 50 \times 8 in H $^{+}$ form. Strontium was separated from yttrium with 125 ml 3 mol l $^{-1}$ HNO $_3$ and yttrium was eluted with 75 ml 3 mol l $^{-1}$ HNO $_3$ (note the separation time).

Detection and Determination of ^{90}Sr

Determination on the Low-level Gas-flow β -Counter. – After the separation of yttrium and strontium, ^{90}Sr was determined by means of ^{90}Y . The solid Y $_2$ (C $_2$ O $_4$) $_3$ was sub-sampled for yield determination, then counted on the low-level gas-flow β -counter immediately upon isolation. The sample activity of ^{90}Sr determined by ^{90}Y on a proportional gas counter was calculated in the following manner:

$$A_{^{90}\text{Sr}} = \frac{I \times F}{M_u \times \varepsilon \times E \times t}$$

where:

$A_{90\text{Sr}}$ – activity of ^{90}Sr /Bq kg^{-1} ,

ε – counting efficiency for ^{90}Y ,

E – yttrium or strontium recovery

t – counting time /s,

I – sample net counts,

F – correction factor ($F = e^{\lambda t}$ ^{90}Y decay correction of $F = 1/C$ equilibrium correction)

M_u – sample quantity /kg.

Determination on the Liquid Scintillation Counter (LSC). – After the separation of yttrium, strontium was deposited as SrCO_3 . The solid SrCO_3 was then dissolved in 1 ml of 2 mol l^{-1} HNO_3 with addition of 15 ml of redistilled water. This solution was put in a 20-ml Teflon vial and was stored for approximately 12 hours to be counted after that. In strontium determination, a liquid-scintillation counter was used and Cherenkov counting was applied. The counting itself was done in such a way that a spectre at the width 0–50.0 keV was taken. The activity was achieved by integration across 100 channels because the whole spectre falls in this range. Before counting of the sample, background radiation was determined by counting the blank sample (1 ml 2 mol l^{-1} HNO_3 + 15 ml H_2O) in a PTFE vial. The counting efficiency ^{90}Sr (^{90}Y) on LSC was determined by counting solutions of known activity. The isotope ^{90}Sr was previously separated from ^{90}Y . Four solutions of various activities were prepared and counted on 16 consecutive days so that disintegration of ^{90}Sr was observed through ^{90}Y . Moreover, disintegration of ^{90}Y was followed by counting the separated ^{90}Y .

The sample activity of ^{90}Sr determined by ^{90}Y on the liquid scintillation counter was calculated by the above described equation, however, taking into account the chemical recovery of strontium instead of yttrium. Cherenkov counting efficiency for LSC and the correction coefficient C for the fraction of equilibrium $^{90}\text{Sr} - ^{90}\text{Y}$. The chemical recovery of strontium and yttrium was determined by the atomic absorption spectrometer.

Detection Limit of the LSC TRI CARB 2770 TR/SL. – This instrument has a low level background (about 2 impulses per minute). This fact is important for the low-level activity determination. For 10 g of the sample, with a determined 47% of efficiency for 0.5 M HNO_3 and 60% average chemical recovery of strontium isolation and counting time of 100 minutes, the lower limit of detection calculated in the Currie manner¹³ is 4 mBq g^{-1} .

RESULTS AND DISCUSSION

Separation of Sr from Y and Other Elements in Ion-exchange Columns

The principle of calcium and strontium separation on strong base anion exchangers with nitric acid–methanol mixture as eluent may be extended to the separation of yttrium and strontium from alkaline and earth-alkaline elements and a whole series of other elements. It was shown that strontium

may be separated from the above mentioned elements in a column filled with anion exchangers DOWEX AG 1 × 8 or AMBERLITE CG-400 and with 0.25 mol l⁻¹ HNO₃ in methanol as eluent.¹⁰⁻¹² The separation will take place only if the exchanger is in NO₃⁻ form and if the eluent contains a high percentage of alcohol, as it is shown in Figures 1 and 2. The separation is almost identical in both exchange columns. However, yttrium does not separate from strontium in this manner but is simultaneously eluted from the column, as it is shown in Figure 3. Yttrium elutes in a slightly more rapid manner so that it comes to a significant overlapping with calcium, which may cause difficulties in the practical application of ⁹⁰Sr determination. Complete separation is achieved by replacing methanol with ethanol, as it is shown in Figure 4.

However, yttrium and strontium bind very strongly to the exchanger in 0.25 mol l⁻¹ HNO₃ in ethanol. This implies a rather time-consuming and protracted elution. For that reason they were eluted with water. Elution with water causes eluting of all bound cations, which includes Pu, Th and other lanthanides that otherwise remain very firmly bound to the column, as it was shown by Korkisch and Tera.¹⁴ Due to their radioactive isotopes, these elements disturb the determination of ⁹⁰Sr, so this procedure is not appropriate for practical use. Unlike the lanthanides, many other elements, such as iron and aluminium, do not bind at all onto the anion exchangers from alcohol solutions. Furthermore, since ⁹⁰Sr may be easily determined by means of ⁹⁰Y, provided that they are previously separated from each other, a

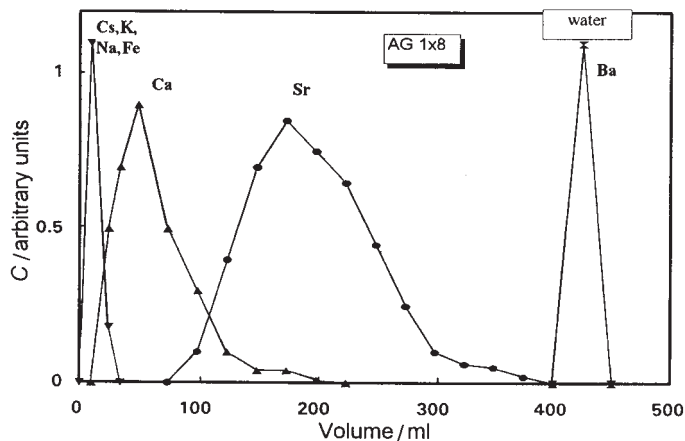


Figure 1. Separation of Sr from Fe, K, Na, Cs, Ca, and Ba in the column filled with exchanger DOWEX AG 1 × 8 and 0.25 mol l⁻¹ HNO₃ in methanol as eluent (exchanger height $h = 14$ cm, flow rate 1 ml min⁻¹, column diameter 1 cm). Ba eluted by water.

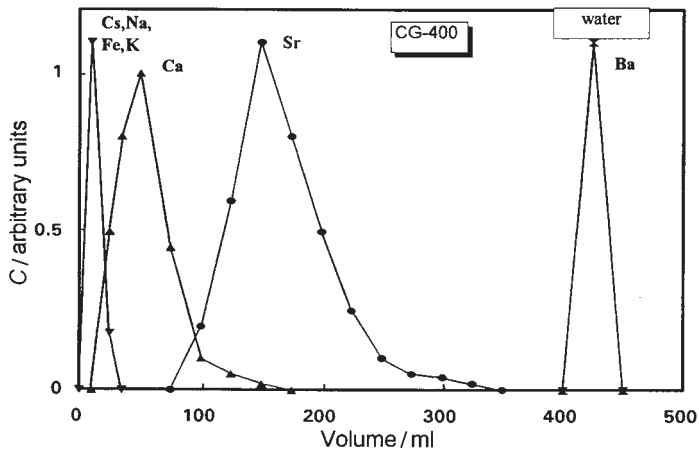


Figure 2. Separation of Sr from Fe, K, Na, Cs, Ca, and Ba in the column filled with exchanger AMBERLITE CG-400 and $0.25 \text{ mol l}^{-1} \text{ HNO}_3$ in methanol as eluent (exchanger height $h = 14 \text{ cm}$, flow rate 1 ml min^{-1} , column diameter 1 cm). Ba eluted by water.

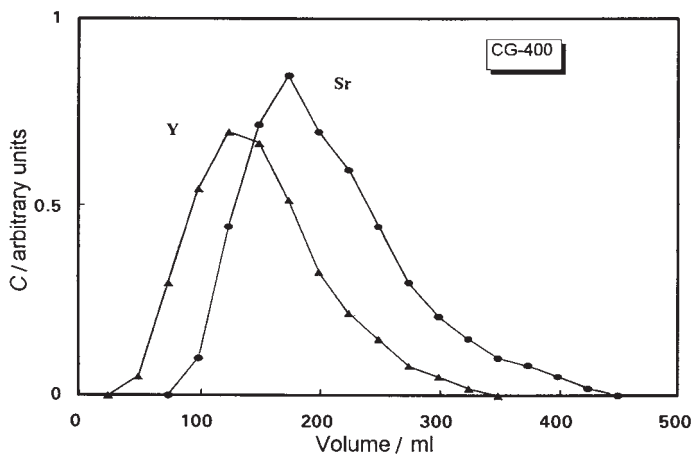


Figure 3. Elution of Sr and Y from the column filled with exchanger AMBERLITE CG-400 and $0.25 \text{ mol l}^{-1} \text{ HNO}_3$ in methanol as eluent (exchanger height $h = 14 \text{ cm}$, flow rate 1 ml min^{-1} , column diameter 1 cm).

simple procedure of yttrium and strontium separation on the cation exchanger was found, as shown in Figure 5. In addition, Figures 1–3 indicate that yttrium and strontium cannot be separated from other cations without a severe loss of chemical yield. Figure 4 indicates complete separation from cal-

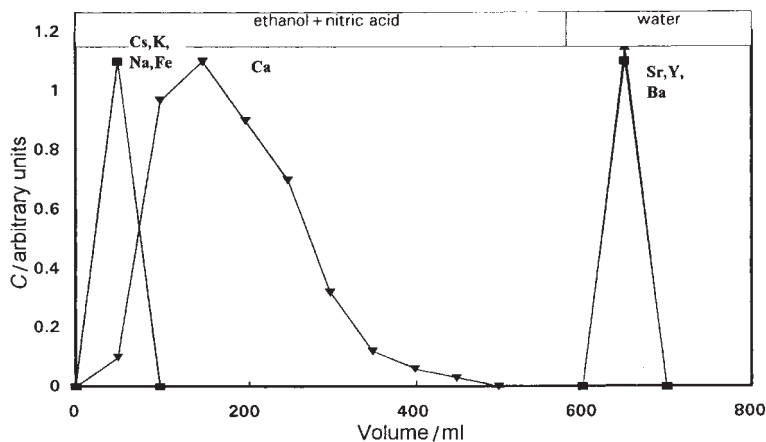


Figure 4. Separation of Sr, Y and Ba from Fe, K, Na, Cs, in the column filled with exchanger AMBERLITE CG-400 and $0.25 \text{ mol l}^{-1} \text{ HNO}_3$ in ethanol as eluent (exchanger height $h = 14 \text{ cm}$, flow rate 1 ml min^{-1} , column diameter 1 cm). Ba, Y, Sr eluted by water.

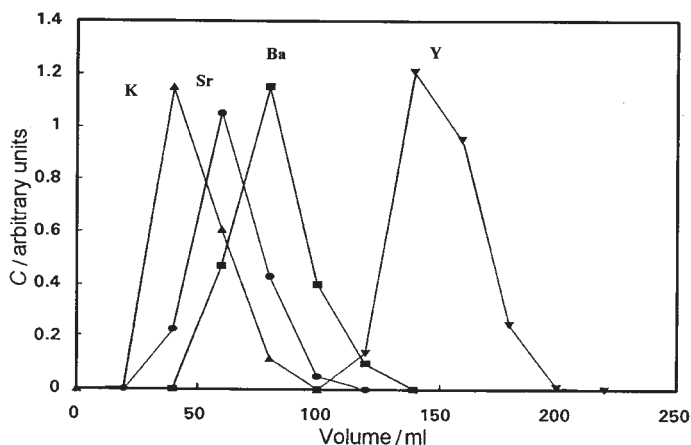


Figure 5. Separation of Y from Sr, K, Ba in the column filled with cation exchanger DOWEX 50×8 and $3 \text{ mol l}^{-1} \text{ HNO}_3$ as eluent (exchanger height $h = 18 \text{ cm}$, flow rate 1 ml min^{-1} , column diameter 1 cm).

cium and alkaline elements with ethanol, which is inadequate for isolation. Complete separation of yttrium and strontium from other cations, without larger losses of chemical yield, can be achieved by using an ethanol–methanol mixture or increasing the exchanger height, as it is shown in Figures 6 and 7.

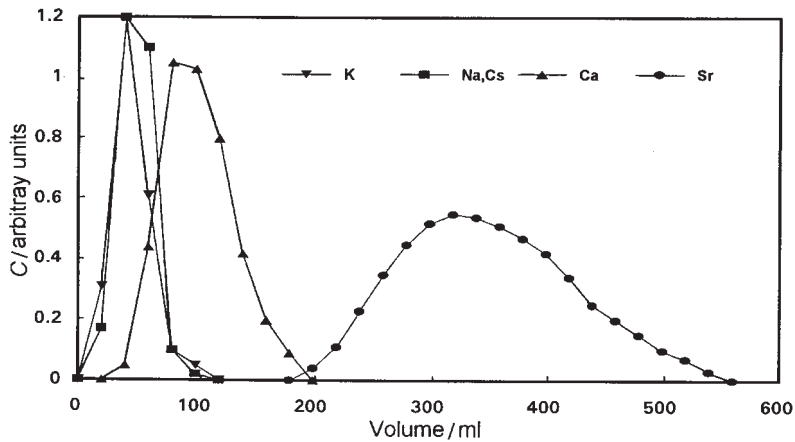


Figure 6. Separation of Sr from K, Cs, and Ca in the column filled with exchanger AMBERLITE CG-400 and $0.25 \text{ mol l}^{-1} \text{ HNO}_3$ in methanol (exchanger height $h = 26 \text{ cm}$, flow rate 3 ml min^{-1} , column diameter 1 cm).

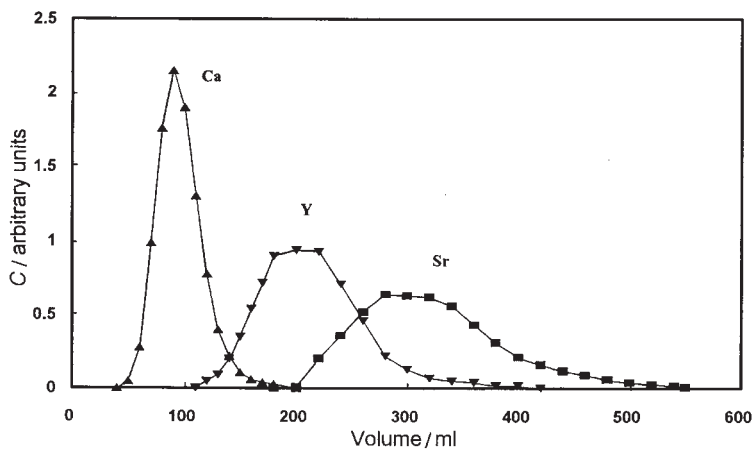


Figure 7. Separation of Sr and Y from Ca in the column filled with exchanger DO-WEX AG 1×8 and $0.25 \text{ mol l}^{-1} \text{ HNO}_3$ in methanol-ethanol mixture (methanol/ethanol, 3 : 1) (exchanger height $h = 14 \text{ cm}$, flow rate 1 ml min^{-1} , column diameter 1 cm).

Rapid Determination of ^{90}Sr in Soil

A procedure for rapid determination of ^{90}Sr was developed and tested by determination of ^{90}Sr in soil samples. Simultaneously, ^{90}Sr was determined in a standard manner by the procedure described in the IAEA report² (leach-

ing with strong concentrated acid, separation of strontium from calcium with fuming nitric acid, scavenging steps with iron hydroxide and barium chromate, strontium carbonate precipitation, strontium-yttrium separation and counting on the gas counter). Table I presents the results of ^{90}Sr determination by means of ^{90}Y on the low-level gas-flow β -counter. These results were compared to those obtained in the standard determination procedure. Table II presents the determination results obtained by the Cherenkov counting and the results obtained by the standard determination method. Determination is carried out three times in each soil sample. The mean value and standard deviations of ^{90}Sr activity of three determinations were calculated from the counting mean value with standard deviation for each determination.

The obtained results show that the procedure was developed in a correct manner and that it may be applied to the determination of ^{90}Sr . It should be mentioned that determination by means of ^{90}Y on the low-level gas-flow β -counter is feasible only if the radiochemical equilibrium has been achieved already in the sample. There is no such limitation in the determination by the Cherenkov counting. In both ways of detection one should bear in mind that ^{89}Sr may also be present. Should ^{89}Sr be present as well, one should choose one of the advanced detection techniques that enable rapid determination of both isotopes. Cherenkov counting is a technique that provides the possibility of determining ^{90}Sr by means of ^{90}Y because the efficiency of counting ^{90}Sr is below 1% for the instrument used in this work. Simultaneously, the efficiency for ^{90}Y was also measured. The characteristic of this instrument is the low level of background radiation with an average value of 2 counts per minute for Cherenkov counting. For this reason, the detection limit is rather low.

Generally, this procedure provides the possibility of a relatively rapid determination of ^{90}Sr , which takes approximately two days in the case of a routine analysis of ^{90}Sr on the low-level gas-flow β -counter (only if radiochemical equilibrium between strontium and yttrium is attained), and ap-

TABLE I

Results of ^{90}Sr determination by means of ^{90}Y on the low-level gas-flow β -counter

Sample (n) ^a	Activity ^{90}Sr /Bq kg ⁻¹		
	New procedure	Standard procedure	Known value
Soil-1 (3)	66 ± 8	65 ± 4	67 ± 3
Soil-2 (3)	94 ± 10.0	103 ± 8	101 ± 4

^a n - det. number.

TABLE II

Results of ^{90}Sr determination on the liquid-scintillation counter

Sample (n) ^a	Activity ^{90}Sr /Bq kg ⁻¹		
	New procedure	Standard procedure	Known value
Soil-1 (3)	64 ± 6	65 ± 4	67 ± 3
Soil-2 (3)	97 ± 6	103 ± 8	101 ± 4

^a n – det. number.

proximately three days on the liquid-scintillation counter in all cases). Strontium and yttrium isolation takes approximately one day. Sample measurements on the low-level gas-flow β -counter take a few hours but measurements on the liquid-scintillation counter in case of lower sample activity take two days. Such time saving, if compared with the standard procedure, was achieved due to the application of a cation exchanger for leaching strontium and yttrium from the soil samples instead of acids. This procedure can be used for determination of very low activity of ^{90}Sr . In this case, large amounts of sample and cation exchanger would be required for determination of ^{90}Sr . The required amount of exchanger is 1 g for 1 g of soil. However, in this case the whole procedure is slowed down. Unfortunately, this procedure is only adequate for determination of the soluble fraction of ^{90}Sr in soil samples. However, with some modification this procedure can be used for determination of ^{90}Sr in many kinds of environmental samples.¹⁵

Furthermore, in each separation in the anion column, an alcohol mixture was used for elution because of a more efficient separation of calcium. The mentioned alcohol mixture (methanol/ethanol, 3:1) was prepared on the basis of our previous examinations,¹² taking into account that the amount of ethanol should not cause any significant increase of the binding capacity of calcium, which would in turn slow down the entire procedure. Separation may be equally done by the use of both exchangers – DOWEX AG 1 × 8 and AMBERLITE CG-400 (the exchangers may be even mixed).

Finally, it should be mentioned that this method could be widely used if an efficient digestion step for dissolution of strontium associated with organic matter and weakly bound to different soil phase were worked up.

CONCLUSION

Strontium and yttrium may be easily and relatively rapidly isolated from soil samples by means of a strong cation exchanger and strong base anion exchangers and the alcohol solution of nitric acid. Such isolation procedure eliminates the use of strong, concentrated acids. It is timesaving and provides the possibility of a detection method leading to a rapid and accurate determination of ^{90}Sr . This method is applicable for determination of the water soluble fraction of ^{90}Sr in environmental samples.

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SAŽETAK

**Izolacija itrija i stroncija iz uzoraka tla
i brzo određivanje ^{90}Sr**

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Razvijen je postupak odjeljivanja itrija i stroncija od kalcija i drugih kationa za brzo određivanje ^{90}Sr u uzorcima tla. Postupak se sastoji od: i) vezanja itrija, stroncija i drugih kationa iz uzorka tla na kationski izmjenjivač AMBERLITE IR-120,

ii) odjeljivanja itrija i stroncija od drugih kationa (Na^+ , K^+ , Ca^{2+} , Ba^{2+}) na koloni punjenoj anionskim izmjenjivačima DOWEX AG 1×8 ili AMBERLITE CG-400, uz HNO_3 ($0,25 \text{ mol l}^{-1}$) u alkoholnoj smjesi kao eluens, iii) odjeljivanja itrija i stroncija na koloni punjenoj kationskim izmjenjivačem DOWEX 50×8 te 3 mol l^{-1} HNO_3 kao eluensom i iv) određivanja ^{90}Sr brojanjem na protočnom plinskom β -brojilu, odnosno Čerenkovljevim brojanjem na scintilacijskom brojilu. Pokazano je da se razvijeni postupak može uspješno primijeniti za brzo određivanje ^{90}Sr . Pri tome se štedi vrijeme i izbjegava upotreba jakih koncentriranih kiselina. Taj je postupak pogodan za određivanje u vodi topive frakcije ^{90}Sr u različitim vrstama prirodnih uzoraka (mlijeko, voda, vino, povrće), uz prethodnu prilagodbu postupka pripreme uzorka.