

Rapid determination of radiostrontium isotopes in samples of NPP origin

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A relatively simple chemical separation procedure has been developed for the simultaneous determination of ^{89}Sr and ^{90}Sr activities in water samples and on aerosol-filters of the Nuclear Power Plant (NPP) Paks origin. The procedure combines the cation-exchange chromatographic (Dowex 50 WX 8 resin) and solid phase extraction (EiChroM Sr.SpecTM, DC18C6 crown ether) steps. The beta-radiation of radionuclides can be measured directly after the chemical separation by LSC. The activities of ^{89}Sr , ^{90}Sr and ^{90}Y are calculated from an over determined set of equations using a method of constrained optimization technique. The equations are based on LSC measurements performed in three counting windows plus the ^{90}Sr – ^{90}Y decay law. The chemical yield of strontium is determined by ICP-AES. The lowest limits of detectable activity, for the measurement time of 600 minutes, are 30 mBq/sample and 18 mBq/sample for ^{89}Sr and ^{90}Sr , respectively.

Introduction

The ^{235}U fission process results in great cumulative yield of strontium isotopes among which is, due to its long half-life, the ^{90}Sr is of special interest. Concerning its biological hazard, the determination of ^{90}Sr in environmental samples is inevitable from both radiation protection and nuclear environmental protection points of view. The biological feature of ^{89}Sr is similar than ^{90}Sr , but due to its shorter half-life ($T_{1/2}=50.5$ d) ^{89}Sr has shorter term environmental impact. The importance of ^{89}Sr grows up direct after nuclear accidents, or incidents like the one that happened on the 10th of April 2003, Unit 2 at NPP Paks, Hungary. In such cases the presence of ^{89}Sr in technological waters, in aerosol filters of NPP origin and in environmental samples may indicate the failure of nuclear fuel elements.

Many methods have been described for the determination of ^{90}Sr in environmental and biological samples.^{1–17} Numerous methods are based on the secular radiochemical equilibrium and chemical separation of ^{90}Sr from his daughter element ^{90}Y and measurement of beta-activity of ^{90}Y .^{18–23} These methods are not applicable if we would like to determine the activity of ^{89}Sr as well, for which the chemical separation of the strontium isotopes is necessary. The selective separation of strontium using EiChroM Sr.SpecTM chromatographic column is nowadays a widespread efficient technology. The column is filled with Amberlit XAD 7 neutral resin coated with active molecule of dicitclohexano-18-Crown-6 (DC18C6) crown ether.^{24–33} Prior to separation by EiChroM Sr.SpecTM technology, pre-concentration of strontium in

liquid samples is needed, which can, for example, be performed by column filled acidic cation-exchange resin. For the simultaneous determination of $^{89}\text{Sr}/^{90}\text{Sr}$ in urine, a method has been developed based on the application of cation-exchange column and EiChroM Sr.SpecTM column, thus eliminating precipitation steps.³⁴

The LSC technique is a widely used method for the beta-counting of $^{89}\text{Sr}/^{90}\text{Sr}$.^{35–41} Since the energy-distribution of beta-spectra measured by LSC is well defined and the difference between the maximal beta-energies (^{89}Sr : 1460 keV, ^{90}Sr : 550 keV, ^{90}Y : 2250 keV) is appropriate, the activity of ^{89}Sr and ^{90}Sr can be determined accurately and simultaneously using, for instance, spectrum-unfolding method,⁴² some of the digital overlay techniques⁴³ or other full spectrum methods.⁴⁷

In this paper chemical separation processes of strontium adapted to aqueous samples and aerosol-filters are presented. The determination of the efficiency-calibration of the LSC instrument and the activity calculation method for $^{90}\text{Sr}/^{89}\text{Sr}/^{90}\text{Y}$ will be described in detail. The importance of the quenching effect of nitric acid and inactive strontium in the sample matrices will also be illustrated. The incident in April 10, 2003 at NPP Paks due to the purification of nuclear fuels resulted in the damage of fuel elements. As a consequence, ^{89}Sr appeared in technological water and aerosol filter samples. Thus, besides spiked test samples, an opportunity occurred to determine the activity of ^{89}Sr and ^{90}Sr applying our new procedure in real samples. The result of these measurements shall also be presented as well.

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Experimental

Preconcentration of strontium

The following carriers were added to 1–5 liters of water sample: 10 mg Ag⁺, 5 mg Sr²⁺, 10 mg Co²⁺, 10 mg Cs⁺, 10 mg Mn²⁺, 10 mg Nb²⁺, 10 mg Zr²⁺, 40 mg Y³⁺ and 100 mg Ca²⁺. The solution was heated to 60–80 °C for 1–2 hours and stirred. Then 200 cm³ 1.5M Na₂CO₃ was added to the solution. After cooling of the solution, a precipitate was formed which was filtered through 0.45 μm membrane filter. The precipitate was then solved in 10 cm³ 3M HNO₃ then diluted to 100 cm³ and pH 1 (Solution A). The carriers mentioned above, except Ca²⁺, were added onto the aerosol filters by injection. After this, the filter was reduced to ashes at 600 °C. The residue was dissolved in 10 cm³ 8M HNO₃, filtered and diluted to 100 cm³ and pH 1 (Solution B). From these solutions 1 cm³ were taken for ICP-AES measurement of strontium.

The strontium content of solution A or B was preconcentrated with cation-exchange column containing 10 cm³ Dowex 50WX-8 (100–200 mesh) resin. The column was first pretreated with 50 cm³ 0.1M HNO₃ solution then 10 mg Cu²⁺ (solved in 0.1M HNO₃) was introduced onto the column, thus a blue ring was arisen on the top of column. The sample solution was then passed through the column with 1–2 cm³·min⁻¹ flow rate. The moving of the copper-ring assures that strontium stays on the column because the strontium is more strongly bonded than the copper. After this the column was rinsed with 50 cm³ 0.1M HNO₃. It is important not to let the blue ring go lower than 2/3 of the column length. The passed sample solution and rinsing solutions were discarded. The strontium (also other cations) was eluted with 20 cm³ 8M HNO₃ from the column.

Purification and LSC analysis of ⁸⁹Sr/⁹⁰Sr

First the EIChroM Sr.SpecTM column was conditioned with 10 cm³ 8M HNO₃. After this the solution containing strontium was passed through the column then rinsed with 20 cm³ 3M HNO₃. The eluted sample solution and rinsing solutions were discarded. Finally the strontium was eluted with 30 cm³ double distilled water. The time of separation was registered (*t*₀). From this solution 1 cm³ was taken to ICP-AES to determine the chemical yield of total strontium. The remaining solution was evaporated almost to dryness and dissolved in 2 cm³ 1M HNO₃ and rinsed into LSC-vial then diluted with 18 cm³ Ultima Gold AB acidic scintillator. The LSC measurements were performed in a Tricarb 2275 type instrument.

Quench-effect measurements

The composition of the matrices prepared for LSC counting influences the shape of the measured beta-spectra. The shift of beta-spectra was measured by means of spiked series containing different concentrations of inactive strontium and nitric acid. The beta-counts of individual samples were measured for 60 minutes in two different windows, A: 20–1200 channels, and B: 1200–4000 channels. The A_{Sr-90}(=A_{Y-90}) activity of 4.835 Bq was used. The *t*_{SIE} values, which quantitatively characterize the quenching-effect, were calculated from the shift of the beta-spectra.

The results presented in Figs 1–3 show that the shift of beta-spectra depends on the nitric acid content of samples only, and the influence of the strontium content is negligible. Our results are in good agreement with those presented in the literature.⁴⁴ It is apparent from Fig. 3, that the measured net intensity in the region of lower energy (NA) saturates above 0.1M of acid content, while that in the region of higher energy (NB) practically constant. With accurate adjustment of nitric acid content of the LSC samples, therefore, the shift of the beta-spectra can be correctly regulated and the spectra will not be influenced by the different chemical yield of strontium of the individual samples.

Calibration procedure

Since the maximal beta-energies of the nuclides of interest are significantly different (⁹⁰Sr: 0.544 MeV, ⁹⁰Y: 2.270 MeV and ⁸⁹Sr: 1.462 MeV), the simultaneous determination of the activity of nuclides is viable, provided that the counting windows are selected properly. To prove this point, the beta distributions of individual nuclides are given in Fig. 4, and the gross beta distribution curve of nuclides of equal activity are given in Fig. 5, which were calculated according to the method given in the literature.⁴⁵ It can be seen in Fig. 5, that the border of the counting regions corresponding to the different nuclides are clearly indicated by break points of the collective beta distribution curve and, therefore, the individual measuring windows can accurately be determined.

This provides three equations relating measured net intensities (*I*_A, *I*_B and *I*_C) to sample activities of the individual nuclides (⁹⁰Sr = A, ⁸⁹Sr = B, ⁹⁰Y = C), which is given in matrix notation as:

$$\begin{bmatrix} \eta_{AA} & \eta_{BA} & \eta_{CA} \\ \eta_{AB} & \eta_{BB} & \eta_{CB} \\ \eta_{AC} & \eta_{BC} & \eta_{CC} \end{bmatrix} \cdot \begin{bmatrix} A \\ B \\ C \end{bmatrix} = \begin{bmatrix} I_A \\ I_B \\ I_C \end{bmatrix} \equiv \mathbf{M} \cdot \mathbf{A} = \mathbf{I} \Rightarrow \mathbf{A} = \mathbf{M}^{-1} \cdot \mathbf{I} \quad (1)$$

where η_{XY} is the counting efficiency of isotope Y in counting the region of X . Equation (1) was solved by constrained optimization technique with the constraint of the kinetic law of ^{90}Y ingrowths in the sample:

$$\begin{aligned} A &\geq 0 \\ B &\geq 0 \\ C &= A \cdot \left[1 - e^{-\frac{\ln(2)}{64.1}(t-t_0)} \right] \end{aligned} \quad (2)$$

where t is the time of the measurement and t_0 is as indicated above.

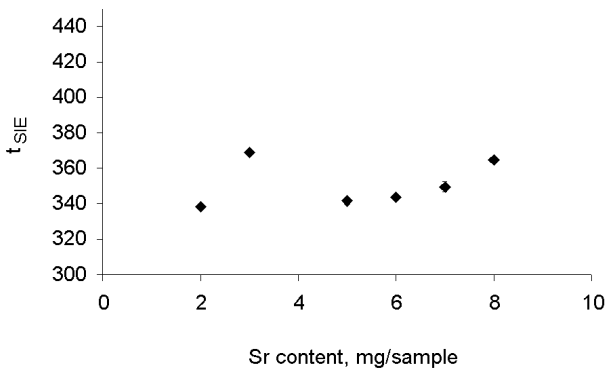


Fig. 1. The t_{SIE} values as a function of inactive strontium content, at constant (0.6M) HNO_3

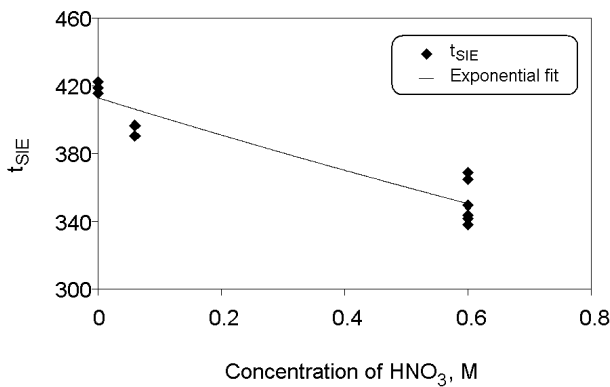


Fig. 2. The t_{SIE} values as a function of nitric acid concentration, at constant (5 mg) content of Sr

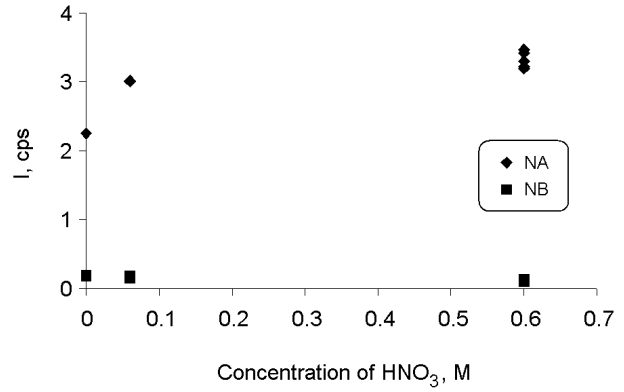


Fig. 3. Net intensity of ^{90}Sr in window A (NA, channels: 20–1200) and window B (NB, channels: 1200–4000) as a function of nitric acid content, at constant (5 mg) content of Sr

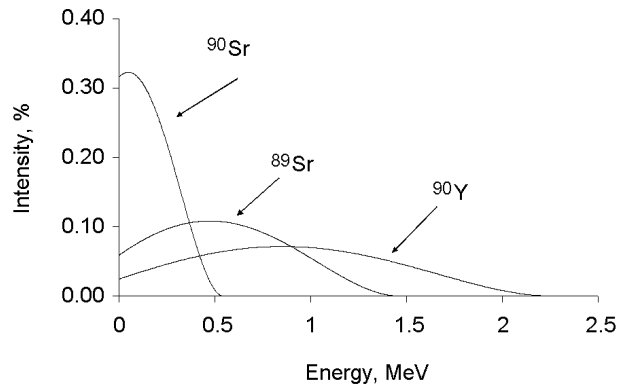


Fig. 4. Theoretical beta-distribution curves of ^{90}Sr , ^{89}Sr and ^{90}Y isotopes

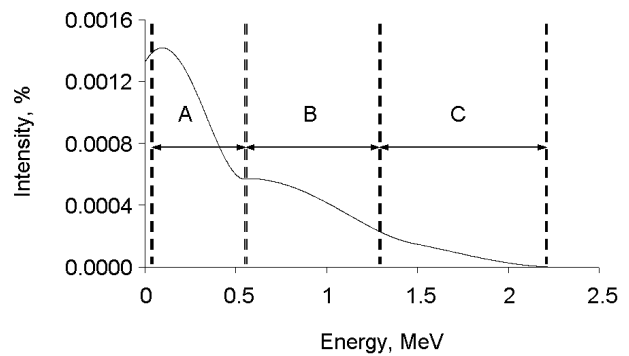


Fig. 5. Collective theoretical beta-spectrum of ^{90}Sr , ^{89}Sr and ^{90}Y isotopes of equal activities

The object function used for the constrained optimization is:

$$f(\mathbf{A}) = \sqrt{\text{norm}(\mathbf{I} - \mathbf{M} \cdot \mathbf{A}) + \left\{ C - A \cdot \left[1 - \exp\left(-\frac{\ln(2) \cdot (t - t_0)}{64.1} \right) \right] \right\}^2}$$

For the solution of the constrained optimization problem the *fmincon* function of MATLAB® (Release 13) was used.

For the determination of the measuring regions and of the calibration matrix, a series of etalon samples were prepared with the following composition:

N1 5 mg Sr²⁺ carrier + 9.667 Bq (⁹⁰Sr+⁹⁰Y);

N2 5 mg Sr²⁺ + 14.61 Bq ⁸⁹Sr;

N3 5 mg Sr²⁺ + 9.667 Bq (⁹⁰Sr+⁹⁰Y) + 14.61 Bq ⁸⁹Sr;

N4 5 mg Sr²⁺ + 4.833 Bq ⁹⁰Sr;

N5 5 mg Sr²⁺.

Each sample was solved in 2 cm³ 1M HNO₃ and 18 cm³ Ultima Gold AB scintillator was added. In the preparation of sample *N4*, ⁹⁰Sr was separated from ⁹⁰Y on Sr-Spec. column using the standard method provided by EIChrom Industries, Darien, IL.

For the sake of comparison to the theoretical energy distribution shown in Fig. 5, the beta-spectra of two parallel samples of type *N3* are given in Fig. 6. It is evident that the experimental distribution curves are practically equivalent indicating that the procedure used for the etalon preparation is reliable. Also these curves are qualitatively similar to the theoretically expected distribution curve and the border of the counting regions can easily be determined. The beginning of the first counting region A was determined based on the optimization of the signal to noise ratio as described in Reference 44.

From these measurements the counting regions and the calibration matrix were determined. The results are given in Tables 1 and 2, respectively.

Results

Our method was first tested with spiked distilled water (DW) and spiked technological water samples (TM) of NPP origin which had no gross-beta activity. The DW samples were contaminated with 0.14 Bq ⁹⁰Sr and 0.16 Bq ⁸⁹Sr, the TM samples were contaminated with 0.174 Bq ⁹⁰Sr. The results of the test measurements are given in Table 3. It can be seen that the calculated and added activities are within 5% error, which is impressive for such a low activities. The results for

⁹⁰Sr/⁹⁰Y indicate the calculated activities differ more from the theoretical values, the error range (10–20%) is acceptable at the presence of ⁸⁹Sr and mBq activities. The chemical yield varied around 60%, which is practically acceptable.

Due to the incident happened on the 10th of April 2003, Unit 2 at NPP Paks, the appearance of ⁸⁹Sr and that of ⁹⁰Sr in certain technological water samples and on aerosol filters were expected. Therefore, samples were taken after the incident and analyzed with our procedure.

The LSC measurements of the real samples were continued for a longer period of time for the analysis of the radiochemical composition, which should change according to the decay laws of the isotopes presented. This way the radiochemical purity of the samples can also be proved. The results are given in Table 4 and the decay curves are shown in Fig. 7. It is apparent from Fig. 7, that for both matrices the measured activity of the radioisotopes followed the theoretical kinetic curves within the experimental error with the exception of the ⁸⁹Sr activity of the water sample. In this case, the calculated activity values at longer time scatter around the theoretical curve without clear trend but follow the decay on the average. This is probably due to the greater uncertainty of the ⁸⁹Sr determination in excess of ⁹⁰Sr/⁹⁰Y isotopes, which is caused by its decay and the ingrowths of ⁹⁰Y. It can be stated, however, that the radiochemical purity of the real, high activity samples were on overall acceptable.

Table 1. The limits of the individual counting windows given in channels and in energies according to energy calibration of LSC instrument (Tricarb 2275)

Window	Lower limit		Upper limit	
	Channel	Energy, keV	Channel	Energy, keV
A	23	12	200	100
B	201	101	800	400
C	801	401	4000	2000

Table 2. Determined efficiency matrix for the evaluation of LSC measurements for matrices with $t_{S/E} = 360 \pm 30$

Isotope	Window A 23–200	Window B 201–800	Window C 801–4000
⁹⁰ Sr	0.5308	0.1478	0
⁸⁹ Sr	0.1258	0.2881	0.0594
⁹⁰ Y	0.0157	0.2336	0.1414

Table 3. Results of measurements of ⁸⁹Sr/⁹⁰Sr activities in spiked water samples

Sample code	Chemical yield	Added activity (AA ⁹⁰ Sr and AA ⁸⁹ Sr)	Mean intensities in the measuring regions corrected by chemical yield			Calculated activity of ⁹⁰ Sr, Bq (CA ⁹⁰ Sr)	Calculated activity of ⁸⁹ Sr, Bq (CA ⁸⁹ Sr)	CA ⁸⁹ Sr/AA ⁸⁹ Sr, %	CA ⁹⁰ Sr/AA ⁹⁰ Sr, %
			cps A	cps B	cps C				
DW1	0.696	0.14 Bq ⁹⁰ Sr + + 0.16 Bq ⁸⁹ Sr	0.08431	0.06431	0.02125	0.161 ± 0.020	0.119 ± 0.025	100.63	85.00
DW2	0.684	0.14 Bq ⁹⁰ Sr + + 0.16 Bq ⁸⁹ Sr	0.07931	0.06417	0.01903	0.163 ± 0.019	0.11 ± 0.028	101.88	78.57
DW3	0.690	0.14 Bq ⁹⁰ Sr + + 0.16 Bq ⁸⁹ Sr	0.07667	0.06028	0.02319	0.152 ± 0.022	0.107 ± 0.030	95.00	76.43
01TM51	0.590	0.174 Bq ⁹⁰ Sr	0.09356	0.03633	0.00819	-	0.138 ± 0.016	-	79.31
01TM52	0.632	0.174 Bq ⁹⁰ Sr	0.09744	0.04411	0.00986	-	0.173 ± 0.012	-	99.43
01TM53	0.589	0.174 Bq ⁹⁰ Sr	0.08022	0.03244	0.00236	-	0.161 ± 0.015	-	92.53
01TM50	0.618	-	0.02234	0.0296	0.00225	0.045 ± 0.022	0.021 ± 0.012	-	-

Measuring time: 600 minutes.

Table 4. Results of analysis of ⁸⁹Sr/⁹⁰Sr activities in water and aerosol samples taken after the 10-04-2003 incident at NPP Paks Unit 2

Sample type	Chemical yield	Time elapsed since separation, hours	Mean intensities in the measuring regions corrected by chemical yield			Calculated activity of ⁹⁰ Sr, Bq (CA ⁹⁰ Sr)	Calculated activity of ⁸⁹ Sr, Bq (CA ⁸⁹ Sr)	Calculated activity of of ⁹⁰ Y, Bq (CA ⁹⁰ Y)
			cps A	cps B	cps C			
Water	0.593	12	18.17	10.54	2.46	29.53 ± 1.50	19.10 ± 0.96	3.66 ± 0.56
		393	17.39	13.46	4.86	30.26 ± 1.65	7.12 ± 0.75	29.83 ± 1.52
		6000	15.84	11.54	3.90	28.37 ± 1.58	2.37 ± 0.59	28.37 ± 1.48
Aerosol	0.754	7	6.32	9.65	1.59	4.66 ± 0.48	30.60 ± 1.55	0.34 ± 0.15
		20	7.00	12.67	2.45	3.29 ± 0.35	41.69 ± 1.58	0.64 ± 0.48
		41	6.35	10.27	1.84	4.34 ± 0.45	31.96 ± 1.60	1.56 ± 0.39
		68	6.14	9.66	1.67	4.61 ± 0.51	28.96 ± 1.45	2.40 ± 0.36
		174	6.14	9.74	1.79	4.72 ± 0.52	27.91 ± 1.39	4.00 ± 0.60
210	6.29	10.02	1.77	4.82 ± 0.52	28.52 ± 1.45	4.33 ± 0.65		

Measuring time: 600 minutes.

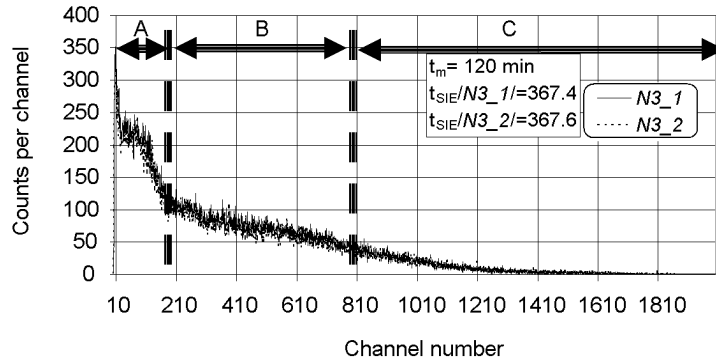


Fig. 6. Experimental beta-spectrum of calibration etalon samples of type N3 (see text for details) with measurement time of 120 minutes

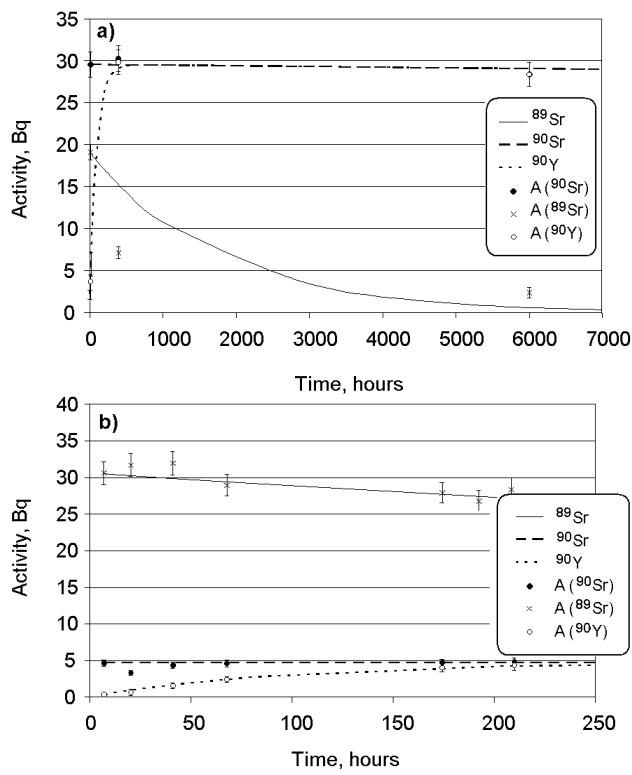


Fig. 7. $^{89}\text{Sr}/^{90}\text{Sr}/^{90}\text{Y}$ theoretical decay curves (lines) and measured activities (markers) of a water (a) and an aerosol (b) samples taken after the 10-04-2003 incident at NPP Paks Unit 2

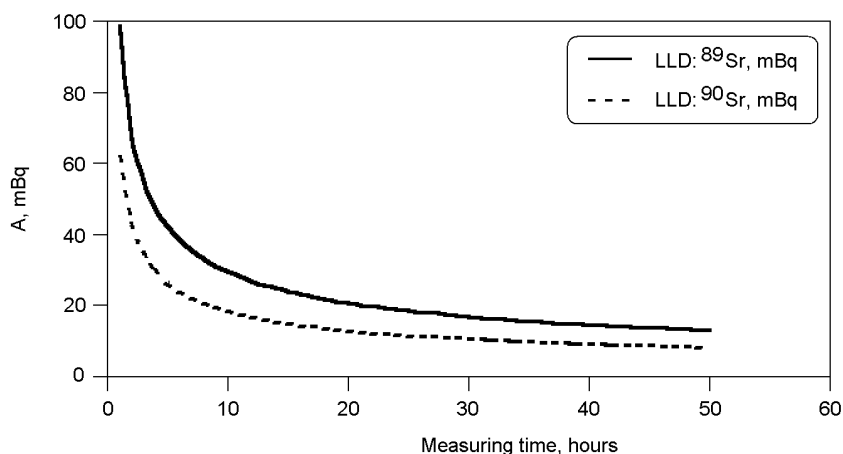


Fig. 8. Lower limit of detection (LLD) for ⁸⁹Sr and ⁹⁰Sr isotopes as a function of the measuring time

Determination of lower limit of detection

The lower limit of detection (LLD) of the process was determined using the Currie criterion. The calculation for individual isotopes was performed by:

$$LLD = \frac{C_1 + C_2 \cdot \sqrt{2 \cdot I_B \cdot t_M}}{t_M \cdot \mu \cdot \eta}$$

where C_1 and C_2 are the Currie constants, I_B is the background intensity in appropriate counting window, t_M is the measuring time, μ is the chemical yield of strontium and η is the counting efficiency of individual isotopes in appropriate counting window. The Currie constants are $C_1 = 5.5$ and $C_2 = 3.3$ values representing 99.5% significance level. The results for individual isotopes are given in Fig. 8. For the measuring time of $t_M = 600$ minutes, 30 mBq/sample for ⁸⁹Sr and 18 mBq/sample for ⁹⁰Sr can be obtained.

Conclusions

A new chemical separation is described, part of which has been adapted from literature for technological water and aerosol filter samples. The separation procedure combines the precipitation and two chromatographic column separation steps, which resulted in an average chemical yield around 60%. Work is in progress to investigate the source of loss, which could enhance the sensitivity of the method. The beta-activity of the separated samples is counted by LSC technique and the activity calculation is similar to that of the most-probable-value-theory.⁴⁸ We have found that the introduction of the ⁹⁰Y decay law as an additional constrain enhance the performance of the method as far as full spectrum evaluation is concerned.

The application of our method to test and real samples proved the radiochemical purity of the prepared samples, thus the reliability of the analytical process.

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