DETERMINATION OF Sr-90 IN MILK SAMPLES FROM THE STUDY OF STATISTICAL RESULTS

by

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The determination of ^{90}Sr in milk samples is the main objective of radiation monitoring laboratories because of its environmental importance. In this paper the concentration of activity of 39 milk samples was obtained through radiochemical separation based on selective retention of Sr in a cationic resin (Dowex 50WX8, 50-100 mesh) and subsequent determination by a low-level proportional gas counter. The results were checked by performing the measurement of the Sr concentration by using the flame atomic absorption spectroscopy technique, to finally obtain the mass of ^{90}Sr. From the data obtained a statistical treatment was performed using linear regressions. A reliable estimate of the mass of ^{90}Sr was obtained based on the gravimetric technique, and secondly, the counts per minute of the third measurement in the ^{90}Sr and ^{90}Y equilibrium, without having to perform the analysis. These estimates have been verified with 19 milk samples, obtaining overlapping results. The novelty of the manuscript is the possibility of determining the concentration of ^{90}Sr in milk samples, without the need to perform the third measurement in the equilibrium.

Key words: strontium, proportional counter, flame atomic absorption spectroscopy, linear regression, radiochemical separation

INTRODUCTION

Strontium is an element that has a number of anthropogenic isotopes, among which ^{89}Sr and ^{90}Sr from the radiological point of view [1]. Both are products of the fission of ^{235}U or ^{239}Pu, the production takes place in nuclear reactors and fission bombs, because their presence in the environment is related to the nuclear industry, nuclear testing and emissions of an accidental nature [2]. Strontium is one of the most dangerous pollutants in the environment because of its long half-life, its high solubility and chemical similarity between the Sr^{+2} and Ca^{+2} [3]. Because of all these factors, strontium can accumulate in organisms through the food chain, being present in biological samples where there is calcium, such as cow's milk [4, 5]. Therefore, it is essential to develop techniques to determine the activity of the strontium concentration in milk samples [6-9], as is the case in the present study. The Commission Recommendation of 8 June 2000 (2000/473/Euratom) sets a warning level for ^{90}Sr of $2 \cdot 10^{-1}$ BqL^{-1} in milk. The Nuclear Safety Council estimated 5.5·10-2 BqL^{-1} as the lower detection limit (2008) [10-11].

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The aim of this study is to create an estimate that allows the calculation of the activity concentration of Sr-90 without measuring the radioisotope in the equilibrium, as an alternative to that measure.

MATERIALS AND METHODS

This study was conducted with 39 cow's milk samples collected from a number of farms in the province of A Coruna (Spain). The radiochemical separation thereof is performed using a cationic resin which selectively retains Sr [12-14], and the determination was performed in a low-level proportional gas counter [15, 16]. There are two ways to determine the activity of ^{90}Sr. The first involves the implementation of two measurements out of equilibrium (5 days and 12 days after separation of ^{90}Sr), and the other consists of performing a measurement at equilibrium (26 days after separation of ^{90}Sr). In the Laboratory of Environmental Radioactivity of the University of a Coruna the latter method was chosen, although in each determination of ^{90}Sr in milk samples the three measurements are made (two out of equilibrium and one in equilibrium). The results were tested using the technique of flame

atomic absorption spectrometry (FAAS) [17]. With the results obtained a statistical treatment was carried out, obtaining reliable estimates of the counts per minute (cpm) of the third measurement (at equilibrium) of each sample in the low-level proportional gas counter, as well as the mass of ^{90}Sr using the FAAS technique without performing the analysis. In this way it is possible to reduce the time and cost of determining the ^{90}Sr in cow milk samples.

MATERIALS

Ion exchange column of glass (50 cm in length and internal diameter 3 cm), analytical balance (Sartorius B 120S), pH meter (Crison Basic 20), vacuum filtration device (Millipore) filters, cellulose nitrate acetate + (Millipore, 47 mm diameter, 0.45 microns), centrifuge (Heraeus Primo Biofuge), drying oven (Memmert UE500), muffle furnace (Horn of Valles Benjamin 2S), epirradiator quartz (Silica quartz & RC-2), with magnetic stirring hotplate (Selecta Agimatic-N), magnetic stirrers and materials commonly used in the laboratory.

REAGENTS

All reagents used were of analytical grade and belong to the commercial Panreac. For the preparation of solutions milli-Q water was used. A carrier is used as an aqueous solution of Sr 40 $mgmL^{-1}$, prepared from the necessary amount of $Sr(NO_3)_2$ anhydrous. The attack of the milk sample once dried and calcined at 650 °C is carried out with 6N HCl and 6N HNO_3. At different stages of the production process ethylenediaminetetraacetic acid ($Na_2EDTA{\cdot}2H_2O$) was used and directly added and dissolved in water at a concentration of 2 % and pH 5.1, a buffer solution acetic acid / sodium acetate pH 2M 4.75, 4M NaCl and 1M sodium carbonate solution prepared from anhydrous Na_2CO_3. For adjusting the pH of the solutions NH_4OH and 3M HCl at 30 % were used. The resin used was a Dowex 50WX8 house (50-100 mesh) [18].

Determination of the prepared carrier concentration and the initial content of strontium in the milk samples and the radiochemical yield were carried out using the FAAS [19]. For the preparation of calibration standards we started from a standard solution of 1.000 0.002 gL^{-1} of $Sr(NO_3)_{-2}$ in HNO_3 0.5 M.

INSTRUMENTATION

The equipment used for measuring ^{90}Sr is a low-level proportional gas counter with a continuous flow of gas (PR-10: 90 % argon and 10 % methane), 10 channels with a low background model Berthold LB-770, a preamplifier unit and anticoincidence discriminators with LB 2025, and a high voltage source. The detector block is connected to a PC via an interface LB-530-PC, obtaining information for the monitor and printer. This information is processed through the UMS program. For measurements of the samples and a protocol we used white count 5 cycles of 285 minutes, as well as for blanks.

For the calculation of the detection efficiency, a standard solution of ^{90}Sr + ^{90}Y chloride, in a hydrochloric solution, of activity concentration 0.10099 0.00026 (0.26 %) $Bqmg^{-1}$, with reference MRC 2001-080, was used. This solution was prepared by CIEMAT (Center for Energy, Environmental and Technological Research). An aliquot of this standard solution is taken and the radiochemical separation of Sr^{+2} and Y^{+2} in a ^{90}Sr pattern in equilibrium with ^{90}Y is performed. Sr^{+2} is isolated by chemical processes and precipitates, with the aid of a Sr^{+2} stable carrier, as $SrCO_3$ which is measured in the proportional counter calibrated with a ^{90}Sr standard, which in turn was prepared with the same procedure. It is also necessary to know the counting efficiency for ^{90}Y, since at the same instant that ^{90}Sr is separated from ^{90}Y, it begins to grow at the expense of its father. The measurement of the sources obtained allows us to calculate the associated efficiencies and uncertainties of the proportional counter for each of these radioisotopes.

To determine the radiochemical yield the equipment used was an Atomic Absorption Spectrometry Perkin-Elmer (Norwalk, CT) employing a reducing flame of acetylene-air, a wavelength of 460.7 nm and a slit width 0.2 nm.

EXPERIMENTAL

The experimental value of the activity concentration of ^{90}Sr in milk samples has been compiled from historical analysis of the Environmental Radioactivity Laboratory of the University of A Coruna, for the period 2009-2012, yielding a total of 39 results. All these samples correspond to the same point, a set of farms located in the municipalities of San Sadurniño, Narahío and A Capela (A Coruna). The values of the concentration of ^{90}Sr activity are shown in tab. 1.

Table 1. ^{90}Sr activity concentration in cow milk samples

Number of samples	Activity ^{90}Sr [Bqm^{-3}] Maximum	Activity ^{90}Sr [Bqm^{-3}] Minimum	Activity ^{90}Sr [Bqm^{-3}] Average	Standard deviation
39	165.8	60.8	103.7	8.8

Two liters of collected cow milk samples are dried and calcined to an attack performed subsequently with concentrated acids (HNO_3 and HCl, 1:1). The resulting solution is treated with disodium EDTA. The difference of the value of the stability constants of the complexes formed at the working pH (4.8) makes the Sr (II) is retained in an ion exchange resin, resulting in elution of other radionuclides (Ca (II) mainly), which ensures passing milli-Q water. By passing disodium EDTA at pH 5.1, Y (III) is retained in the resin, creating a very stable complex that is eluted. Finally Sr is eluted by passing NaCl 4M saturated, a process that also reinstates the resin to its original sodium form, allowing its reuse. Then Sr is precipitated as $SrCO_3$ [20], which is purified. Finally the analysis of 90Sr is made on the low-level proportional gas counter in three measurements. The first two are outside the equilibrium between ^{90}Sr and ^{90}Y and the activity is calculated using a procedure based on the following considerations.

– In the milk sample the ^{90}Sr is in a secular equilibrium with its child ^{90}Y (half-life of 64.00 hours), from which it separates in the analytical procedure ($t = 0$), and from that moment ^{90}Y begins to grow again until reaching again the equilibrium in approximately 21 days (7 half lives of the son), according to the laws of radioactivity. On the other hand, the ^{89}Sr decays with its half-life of 50-53 days, while the activity of ^{90}Sr can be considered constant since its half-life is 28-79 years.
– The beta particles of both strontiums and ^{90}Y are indistinguishable from each other, the method of calculation is based on two measurements and two equations, taking into account that the total activity recorded in each measurement is the sum of the activities of the three isotopes, of which approximately ^{90}Sr remains constant, ^{90}Y increases, and ^{89}Sr decreases, depending on the time elapsed between the two measurements. These first two measurements are performed one and two weeks after radiochemical separation of ^{90}Sr and ^{90}Y.
– The third measurement is performed at least three weeks after radiochemical separation, to allow time for ^{90}Sr and ^{90}Y to reach secular equilibrium (the half-life of ^{90}Y is 64.00 hours and it is assumed that the secular equilibrium is reached after seven half-lives of the son isotope), so that the activity of the sample will decay according to the half-life of ^{90}Sr (^{89}Sr activity is considered undetectable because it occurs only by fission and has a very low half-life of 50-53 days).

The chemical yield of the process is obtained by the addition of a Sr stable carrier at the beginning of the radiochemical separation, and subsequent measurement by FAAS. The chemical yields obtained are over 50-95 %.

CALCULATION

During the period 2009-2012, 39 samples of cow's milk have been analyzed. The cpm values were obtained in the first and second measures out of the equilibrium between ^{90}Y and ^{90}Sr, and in the third measure in the equilibrium between ^{90}Y and ^{90}Sr. The values of Sr mass of each sample are also known, which have been determined by two analytical techniques, such as gravimetry and FAAS. With all this data, two statistical estimates have been developed. The first one, allows us to obtain the cpm of the third measure in the equilibrium between ^{90}Y and ^{90}Sr, from the cpm obtained with the first and second measures out of the equilibrium. The second estimate allows us to obtain the mass of Sr obtained by FAAS, from the determination of the Sr mass by gravimetry. Thus, it would not be necessary to perform the third measure in the equilibrium between ^{90}Y and ^{90}Sr, nor the determination of Sr mass by gravimetry.

The statistical model starts collecting the number of accounts in the channel beta of the proportional counter in the 3 measurements for the determination of ^{90}Sr. The first measurement is performed 1 week after the radiochemical separation, out of equilibrium between ^{90}Sr and ^{90}Y. The second measurement is performed 2 weeks after the radiochemical separation, out of equilibrium. Finally, the third measurement is performed at least 3 weeks after the radiochemical separation, in the equilibrium. This data will be used to create a curvilinear estimate, based on various assumptions. In this way the following curve estimation is obtained

$$\begin{matrix} [c(t_3)\beta]/[Ef\beta(\text{cpm})] \\ a\ [c(t_i)\beta]/[Ef\beta(\text{cpm})] \quad b \end{matrix} \tag{1}$$

where $c(t_3)\beta$ is counts per minute in the beta channel in the third measurement of the cow milk sample, $Ef\beta$ – the efficiency in beta, $c(t_i)\beta$ – the counts per minute in the sample channel beta cow's milk in the first or the second measurement, depending on which is the best fit of the curve estimation. a is the slope of the curve estimation, and b – the intercept of the curve estimation.

For the adjustment of the estimation data from 39 samples of cow's milk were used. With this first estimate, the cpm of ^{90}Sr in the beta channel are determined, without performing the third measure in the equilibrium.

The second part of this design consists in estimating the mass of Sr obtained by FAAS from actual Sr mass determined by the gravimetric technique. As in the previous estimation of the study samples of milk for the last 4 years (2007-2011), which was determined by calculating the concentration of Sr activity by both techniques. With this data, another curve estimation is built, which is set with the adjustment data Sr

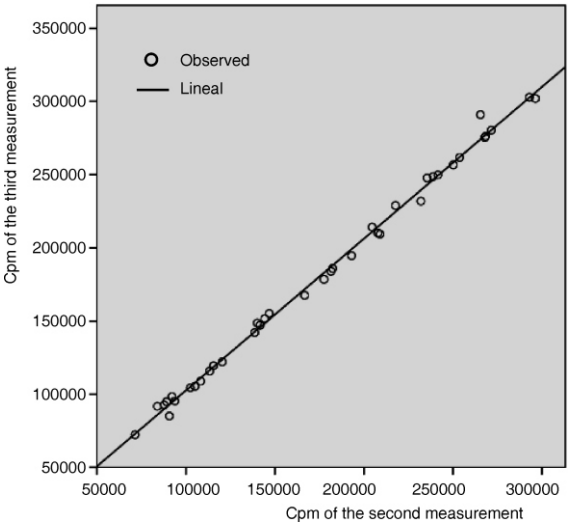

Figure 1. Cpm curve estimation of the β channel in the third measure β *vs.* cpm in the beta channel in the second measurement

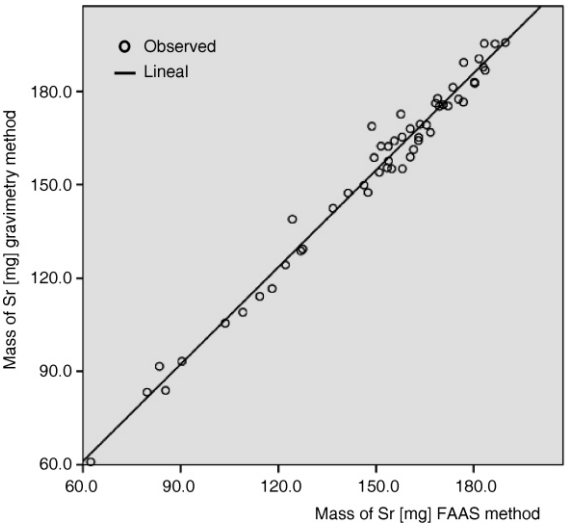

Figure 2. Curve estimation Sr mass [mg] obtained by the gravimetric technique *vs.* Sr mass [mg] obtained by the technique of FAAS

separations conducted on samples of cow's milk. In this way the following curvilinear estimate is obtained

$$\text{mSr(mg)FAAS} = a[\text{mSr(mg)grav}] + \text{b} \qquad (2)$$

With this correlation estimate the mass of Sr on the plane is obtained by applying FAAS from Sr mass determined by the gravimetric technique. To simplify calculations, it can adjust an equation analogous to the preceding, but with the mass of $SrCO_3$ instead of Sr gravimetric mass as shown in fig. 2.

RESULTS AND DISCUSSION

The results for the estimation of the counts per minute in the beta channel for ^{90}Sr in equilibrium, are shown in fig. 1.

The curve obtained is as follows

$$[c(t_3)\beta]/[Ef\beta(\text{cpm})] = (1.038 \quad 0.011)\ c(t_i)\beta]/[Ef\beta(\text{cpm})] + (-0.129 \quad 0.199)$$

$$R = 0.998,\ R^2 = 0.996,\ R^2_{\text{corrected}} = 0.996$$

The results for the estimation of the mass of Sr by FAAS, are shown in fig. 2.

The estimate was obtained as follows:

$$[\text{mSr(mg) FAAS}] = (0.9413 \quad 0.021)\cdot [\text{mSr(mg) grav.}] + (4.373 \quad 1.308)$$

$$R = 0.989,\ R^2 = 0.978,\ R^2_{\text{corrected}} = 0.977$$

The two curvilinear estimates are verified with 19 cow milk samples collected between July 2012 and January 2014, with the following results, shown in tabs. 2 and 3.

In these two tables we can observe the similarity of the 19 results obtained between the real values obtained (the measures of cpm in the third measure in the equilibrium in the proportional counter and the measure of strontium by FAAS and the estimated, calculated from the linear regressions designed. With the third measure at equilibrium the concentration of ^{90}Sr activity is obtained, and with the FAAS measure the chemical yield of the process is obtained. For the use of these estimates, it is necessary to take into account that ^{89}Sr activity is not significant, the time between measurements is not significantly different between samples and the difference of decay correction between samples is insignificant. These estimates are valid for cow milk samples of the study area (farms located in the municipalities of San Sadurnino, A Capela and Narahio (A Coruna). Other sampling points with similar characteristics would require a study similar in character to that described in this article in order to properly use curvilinear estimates.

The real and estimated cpm values in the third measure at equilibrium obtained are overlapping. These values are used to determine ^{90}Sr activity, applying the following formula

$$A_{\text{Sr-90}} \quad \frac{x}{60E_{\text{Sr-90}}PR_q e^{-\lambda_{\text{Sr-90}}t}} \qquad (3)$$

where

$A_{\text{Sr-90}}$ is the activity concentration of ^{90}Sr [Bqm^{-3}], x – the counts per minute of ^{90}Sr (cpm), $E_{\text{Sr-90}}$ – the counting efficiency ^{90}S, P – the weight of the dry sample, R_q – the chemical yield, and $e^{\lambda_{\text{Sr }90}t}$ – the decay correction factor of ^{90}Sr radioactive decay in time t.

Table 4 shows the ^{90}Sr activities obtained with the real and estimated cpm values of the third measure at equilibrium.

These estimates are valid for cow milk samples of the study area (farms located in the municipalities of San Sadurnino, A Capela and Narahío, A Coruna). Other sampling points with similar characteristics

Table 2. Comparison between the actual result and the estimate of the number of counts per minute measured in samples of cow milk

Sample	3rd measurement in the equilibrium estimated [cpm]	*u* (2) 3rd measurement in the equilibrium estimated [cpm]	3rd measurement in the equilibrium real [cpm]	*u* (2) 3rd measurement in the equilibrium real [cpm]	Recovery [%]
1007123-lv	7.8515	0.1942	7.8425	0.1722	75.31
0708123-lv	10.7780	0.2221	10.587	0.1951	95.56
0409123-lv	4.4968	0.3967	4.0625	0.1342	89.35
0910123-lv	2.8346	0.3991	2.9221	0.1204	88.74
0711123-lv	3.0968	0.2435	3.1930	0.1238	88.13
1112123-lv	4.9073	0.1981	4.8400	0.1428	91.20
1501133-lv	2.6573	0.3520	2.7579	0.1183	94.35
0502133-lv	3.3761	0.3552	3.1375	0.1231	87.80
0503133-lv	5.7708	0.3172	5.7502	0.1523	89.65
1004133-lv	3.4234	0.3402	3.4309	0.1267	74.22
0705133-lv	5.2942	0.2055	5.3754	0.1485	94.86
0506133-lv	4.2242	0.2064	4.1796	0.1355	53.95
0207133-lv	3.9651	0.2110	4.0168	0.1337	86.55
0708133-lv	3.3729	0.2470	3.3958	0.1263	88.59
1809133-lv	5.9692	0.2066	6.0702	0.1555	82.03
0910133-lv	1.9813	0.2663	2.0751	0.0980	73.35
0611133-lv	2.6081	0.4186	2.5375	0.1154	58.72
1112133-lv	3.7477	0.1626	3.8182	0.1314	87.35
1501143-lv	5.3130	0.2067	5.3004	0.1477	97.11

Table 3. Comparison between the actual result and the estimate of the mass of Sr in milk samples obtained from cows

Sample	Mass Sr FAAS estimated [mg]	*u* (mass Sr FAAS estimated) [mg]	Mass Sr FAAS real [mg]	*u* (mass Sr FAAS real) [mg]
1007123-lv	154.0	3.3	150.6	1.2
0708123-lv	193.5	4.4	191.1	2.7
0409123-lv	175.5	4.0	178.7	1.2
0910123-lv	180.1	4.1	177.5	1.7
0711123-lv	178.8	4.1	176.3	1.0
1112123-lv	187.6	4.3	182.4	1.2
1501133-lv	187.6	4.3	188.7	1.2
0502133-lv	169.7	3.9	159.8	1.9
0503133-lv	182.6	4.2	179.3	2.2
1004133-lv	145.8	3.4	148.4	2.4
0705133-lv	191.4	4.4	189.8	2.2
0506133-lv	106.0	2.6	107.9	1.6
0207133-lv	171.7	4.0	173.1	2.0
0708133-lv	173.9	4.0	177.2	2.1
1809133-lv	162.6	3.8	164.1	2.0
0910133-lv	140.1	3.3	146.7	1.6
0611133-lv	111.8	2.7	117.4	1.5
1112133-lv	170.7	3.9	174.7	1.8
1501143-lv	191.2	4.4	194.2	2.0

would require a study similar in character to that described in this article in order to properly use curvilinear estimates.

CONCLUSIONS

Two linear estimates have been designed based on actual data for determining the counts per minute in the beta channel for ^{90}Sr in equilibrium and to determine the mass of Sr by FAAS. These estimates can be used as an alternative method, in case of destruction or loss of the sample before the measurement of ^{90}Sr in equilibrium. These estimates will minimize the time and cost that would result in the realization of a new radiochemical separation of ^{90}Sr, since there is no need to carry out the third step in the equilibrium for determination of cpm Sr, or subsequent determination of Sr by FAAS.

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Table 4. Comparison between the real and the estimate results of the activity of ^{90}Sr in milk samples obtained from cows

Sample	^{90}Sr activity estimated [Bqm^{-3}]	u (^{90}Sr activity estimated) [Bqm^{-3}]	^{90}Sr activity real [Bqm^{-3}]	u (^{90}Sr activity real) [Bqm^{-3}]
1007123-lv	114.2	8.8	111.7	8.6
0708123-lv	124.2	26.9	122.7	26.6
0409123-lv	43.2	6.6	44.0	6.7
0910123-lv	23.7	6.2	23.4	6.1
0711123-lv	31.1	7.6	30.7	7.5
1112123-lv	51.7	11.5	52.0	11.6
1501133-lv	23.4	6.7	23.5	6.7
0502133-lv	34.8	9.5	32.8	9.0
0503133-lv	66.4	7.9	65.2	7.8
1004133-lv	38.7	7.6	39.4	7.7
0705133-lv	62.2	6.0	61.7	6.0
0506133-lv	72.3	7.8	73.6	7.9
0207133-lv	46.1	5.4	46.5	5.4
0708133-lv	32.5	4.4	33.1	4.5
1809133-lv	73.8	7.0	74.5	7.1
0910133-lv	19.5	5.1	20.4	5.3
0611133-lv	28.4	8.9	29.8	9.3
1112133-lv	45.3	4.4	46.4	4.5
1501143-lv	54.8	5.5	55.7	5.6

AUTHORS' CONTRIBUTIONS

A. Otero-Pazos, A. Calleja-Garcia, M. Isabel Fernandez-Ibanez, B. A. Rodríguez-Gomez, A. J. Pinon-Pazos, and J. L. Calvo-Rolle conceived the study. A. Otero-Pazos, and A. Calleja-Garcia, developed the method. M. I. Fernandez-Ibanez, B. A. Rodriguez-Gomez, A. J. Pinon-Pazos, and J. L. Calvo-Rolle interpreted the results and drafted the manuscript. A. Otero-Pazos, A. Calleja-Garcia, and M. I. Fernandez-Ibanez, supervised the experimental data analysis and they also contributed to the critical revision and improvement of the paper. All of the authors have approved the final version of the manuscript.

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ОДРЕЂИВАЊЕ КОНЦЕНТРАЦИЈЕ ^{90}Sr У УЗОРЦИМА МЛЕКА НА ОСНОВУ ПРОУЧАВАЊА СТАТИСТИЧКИХ РЕЗУЛТАТА

Због утицаја ^{90}Sr на животну средину, одређивање концентрације ^{90}Sr у узорцима млека главни је задатак лабораторија за мониторинг зрачења. Концентрација активности ^{90}Sr из 39 узорака млека добијена је радиохемијским издвајањем заснованим на селективном задржавању стронцијума у катјонској смоли (Dowex 50WX8, 50-100 mesh) и каснијем одређивању пропорционалним гасним бројачем ниског нивоа. Резултати су проверени мерењем концентрације стронцијума применом пламене атомске апсорпционе спектроскопије како би се коначно добила маса ^{90}Sr. Добијени подаци статистички су обрађени методом линеарне регресије. Поуздана процена масе ^{90}Sr добијена је гравиметријским поступком као и одброј по минуту у трећем мерењу при равнотежи ^{90}Sr и ^{90}Y, без потребе да се обавља анализа. Ове процене верификоване су на 19 узорака млека са добијеним поклапањем резултата. Новост која се приказује у раду је могућност одређивања концентрације ^{90}Sr у узорцима млека без потребе за трећим мерењем при равнотежи.

Кључне речи: стронцијум, пропорционални бројач, пламена атомска апсорпциона спектроскопија, линеарна регресија, радиохемијско издвајање