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Determination of ^{90}Sr in milk and milk powder in Tehran and estimation of annual effective dose

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Abstract Thirty-eight different milk and milk powder samples from Tehran-Iran were collected and analyzed for ^{90}Sr activity using a method in which the daughter product of ^{90}Sr decay (^{90}Y) was extracted by tributyl phosphate from ashed milk. ^{90}Y was then back extracted with water, and oxalate was precipitated. Following the sample analyzing, beta counting was performed with an ultralow-level liquid scintillation spectrometer. The quality control and assurance of the method were obtained by standard samples prepared with an IAEA-certified reference material. The mean determined ^{90}Sr activity concentration in the analyzed milk and milk powder (0.225 ± 0.042 and $0.216 \pm 0.024 \text{ Bq kg}^{-1}$, respectively) showed that the radioactivity concentration in our samples was too low to induce biological hazards. These data can provide useful information of the background level of contamination, which in turn can be used in the following environmental monitoring programs.

Keywords Strontium-90 · Milk · Effective dose · Activity determination

1 Introduction

The biologically hazardous radionuclide ^{90}Sr , which is present in our environment, is an artificial radionuclide, produced essentially by the ^{235}U and ^{239}Pu fission reaction, which has occurred during the previous atmospheric nuclear tests and nuclear reactor accidents (Brun et al. 2003; Stamoulis et al. 2007).

Strontium is a bone seeker element. Due to its chemical and biochemical similarities with calcium, more than 99% of strontium is efficiently incorporated into bone tissue and teeth. Characterized by a long physical and biological half-life (28.15 and ≈ 7 years, respectively), ^{90}Sr may cause damage to bone marrow and induce bone sarcoma and leukemia, because of its high-energy β -particles; $E_{\beta\text{max}}$: 546 keV; (Brun et al. 2002). ^{90}Sr decays to ^{90}Y (half-life: 64.1 h), which emits hard β -particles with maximum energy of 2,280 keV. ^{90}Y also contributes to the internal dose of ^{90}Sr (Brun et al. 2003).

^{90}Sr transfers into humans mainly via foodstuffs. Since milk is the principle source of calcium in human diet, it is a substantial contributor especially for infants (Bem et al. 1991). Moreover, because strontium transfer from soil and plant to cow milk is efficient and rapid, milk contamination level can give an indication of ^{90}Sr deposition over a wide area (Brun et al. 2002; Galle 1988). Therefore, ^{90}Sr measurement especially in milk has acquired considerable attention in environmental and personal monitoring programs (Alvarez et al. 1995; Froidevaux et al. 2006, 2004; Landstetter and Wallner 2006; Mietelski et al. 2004; Al-Masri et al. 2004). Also, there are a lot of reports, focusing on the methodology of its measurement (Chang et al. 2004; Jassin 2005; Lee et al. 2002; Baron et al. 2004; Horwitz et al. 1992; Mikulaj and Svcc 1993; Tait et al. 1997). Accordingly, this paper represents the results of

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radioactivity analysis carried out for ^{90}Sr in the milk samples consumed in Tehran-Iran followed by the estimation of its annual effective dose, in order to assess of the toxic effects of this radio isotope in the consumers.

2 Materials and methods

2.1 Reagents

All reagents used were of analytical grade and from MERCK or FLUKA companies. The radioisotopes $^{90}\text{Sr}/^{90}\text{Y}$ solution was obtained from Amersham. The method was tested and certified with reference milk (Milk-152) received by AQCS (Analytical Quality Control Services) laboratory of the International Atomic Energy Agency (IAEA), Vienna, Austria.

2.2 Equipment

Beta counting was performed with a Wallac (model Quantulus 1220) ultralow-level liquid scintillation. Other required instruments included an oven, a muffle furnace, a freeze/dryer set, an analytical balance, hot plates, and magnetic stirrers, all of which are normally available in chemical laboratories.

2.3 Sampling and sample preparation

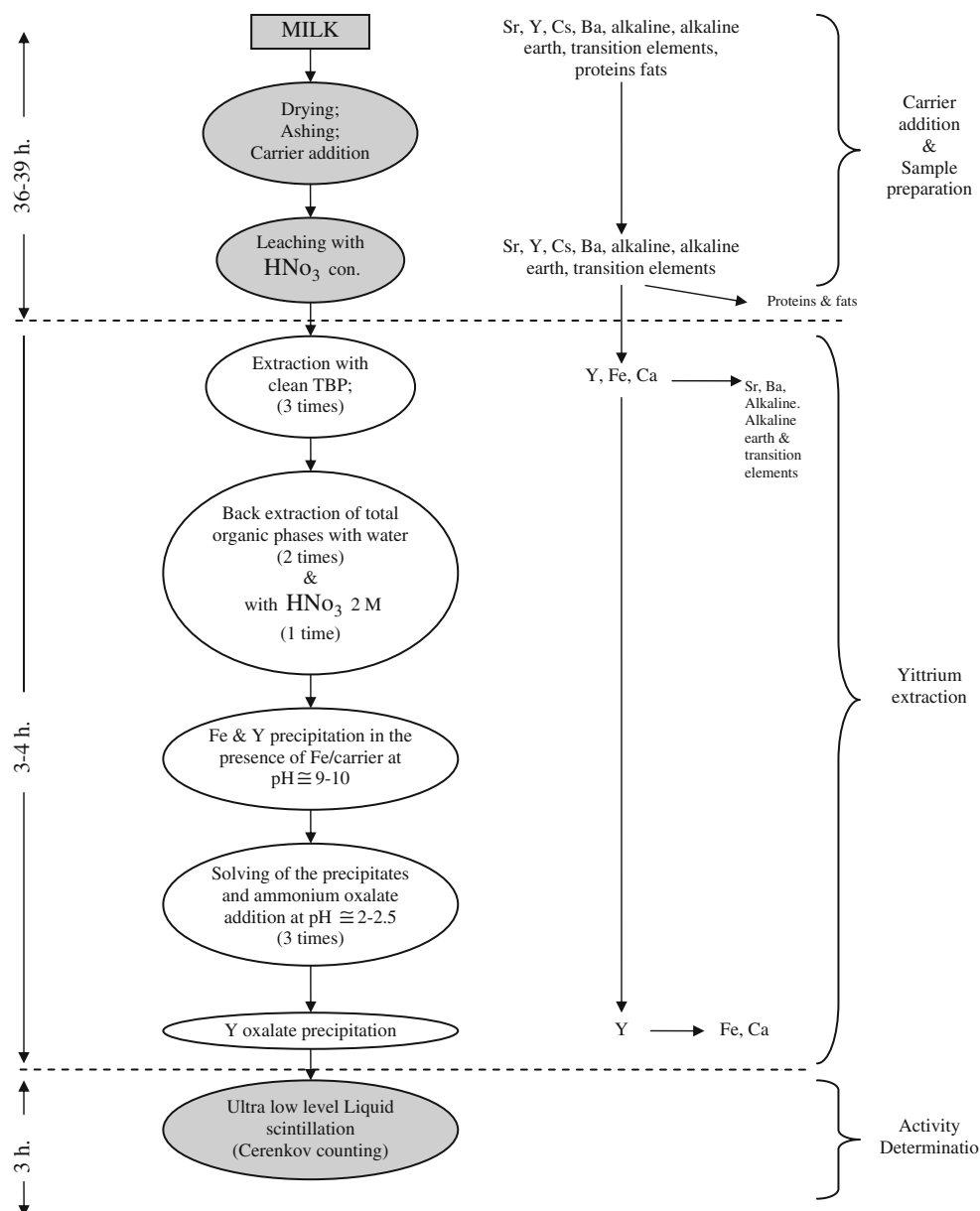
Milk samples were obtained from local markets in Tehran (the capital city of Iran). Around 12 million inhabitants live in this area, and the large population of this city was one of the main considerations for the selection of the sampling site. Twenty-eight milk and ten milk powder samples were collected during the year of the experiment. All the samples were prepared before analysis. First, about 1.5–2 l of each milk sample was dried, and each time, 100–120 g of the dried milk or milk powder was weighted in a porcelain crucible and dried in an oven at 80°C for 6 h to a constant weight. After well drying the samples, complete ashing in a muffle furnace at 700°C was carried out for about 2 h (note that the temperature should be increased gradually). As a result of this step, a large amount of organic materials such as fats and proteins will be decomposed.

2.4 Chemical procedure

The applied method; which is the combination of two common methods with some modifications, was based on the direct determination of ^{90}Y that is in secular equilibrium with ^{90}Sr , by measuring Cerenkov irradiation using liquid scintillation counter (Bem et al. 1991; IAEA 1993a,

b). The method used TBP (tributyl phosphate) extraction of ^{90}Y as follows (Fig. 1):

1. Ten–twenty grams of the ash obtained was weighed into a 250-ml beaker, and 1 ml of each Cs^{+} , Ba^{2+} , La^{3+} , and Sr^{2+} carrier solutions, with 10 ml of Y^{3+} carrier solution, was added (each containing 1 mg element. ml^{-1}).
2. Ten milliliters of conc. HNO_3 (65%) per 1 g of ash (about 100 ml) was added, and the sample was gently boiled at 200°C for about 2 h on a hot plate while the beaker was covered with a glass watch (leaching process).
3. After the leaching step, the sample was cooled enough to be filtered through a medium–fast filter paper.
4. The filtrate was transferred into a 250-ml separatory funnel and extracted for 3–5 min with 30 ml TBP (previously equilibrated with 14 M HNO_3). The time of the first extraction was recorded for the decay correction.
5. After the separation of the two phases, the organic phase was transferred into another separatory funnel and the acid phase was treated again with 30 ml of TBP. This step was repeated once more.
6. The organic phases obtained from all the three mentioned steps were combined and washed with 50 ml of 14 M HNO_3 to remove possible contamination from other radio nuclides with lower distribution coefficients between TBP and 14 M HNO_3 . The aqueous phase was discarded.
7. The organic phase was back extracted 2 times with 50 ml of water and then with 50 ml of 2 M HNO_3 to strip yttrium from TBP.
8. The aqueous phases were combined and then evaporated to less than 50 ml on a hot plate. Following that, the pH was adjusted to 9–10 with ammonia solution.
9. Two milliliters of Fe^{3+} carrier solution was then added to the sample, and after heating in a water bath, it was centrifuged for 6–8 min at 6,000 rpm.
10. The precipitate was dissolved using a minimum amount of 6 M HNO_3 by heating. Then, 30 ml of 2% ammonium oxalate solution was added and pH was adjusted up to 2–2.5 by adding ammonia again.
11. After heating in a water bath, the sample was centrifuged for 6–8 min at 6,000 rpm. The steps 10 and 11 were repeated 3 times in order to eliminate the possible presence of Fe^{3+} ions.
12. Finally, the solution containing yttrium oxalate precipitate was filtered through an accurately weighed filter paper (blue band) and washed twice with a minimum amount of distilled water and ethanol.

Fig. 1 Schematic flowchart for analytical procedure

2.5 Sample counting

As already mentioned, in this research, ^{90}Sr concentration is determined by its daughter (^{90}Y) in radioactive equilibrium with its parent. For the Cerenkov counting, yttrium oxalate together with the filter paper was dissolved with 5 ml 6 M HCl in a 20-ml Copper–Teflon scintillation vial by heating in an oven at 80°C for 3–5 min. Then, 10 ml of distilled water and 5 ml of 2 M HCl were added and mixed well by shaking.

After preparing the vials, they were counted in a Quantulus 1,220 liquid scintillation spectrometer that has

an active liquid scintillation guard counter and a 4π old lead-passive shielding that protects the spectrometer against the external and cosmic radiations. Each sample was counted once for 10,600 s in Cerenkov mode. In the beta spectrum, ^{90}Y window was selected in channel region 5–400. The detection efficiency for ^{90}Y was calibrated using some sources prepared from standard $^{90}\text{Sr}/^{90}\text{Y}$ solution after separation of ^{90}Y and equaled to 74%. Blanks were prepared in the same way as the sample using stable yttrium carrier, and the amount of the average background count rate was determined as 1.323 cpm (count per minute).

2.6 Calculations

2.6.1 Activity determination

After sample counting, the activity concentration A (Bq kg^{-1}) in the samples was calculated using the following expression:

$$A = \frac{G_b - B}{R \times W_a \times \varepsilon \times 60} \times e^{\lambda \Delta t} \times f_{a.d.} \quad (1)$$

where G_b is the gross beta count rate (cpm) for the sample, B is the count rate of the blank sample (cps), R is the chemical recovery of yttrium determined by gravimetry, and the calculation is based on the standardized yttrium carrier solution, W_a is the analyzed ash weight (kg), ε is the Cerenkov beta counting efficiency for ^{90}Y , λ is the decay constant of ^{90}Y (0.0108 h^{-1}), Δt is the decay time from the first extraction to the middle of the counting time (hours), and $f_{a.d.}$ is the ash to dry weight ratio.

2.6.2 Uncertainties

For the 2σ standard deviation, uncertainties, U (Bq kg^{-1}) for the Cerenkov method, were calculated according to the following relation (Scarpitta et al. 1999):

$$U = \frac{2\sqrt{G_b \times \text{CT}}}{\text{CT} \times W_s \times R \times \varepsilon} \quad (2)$$

where CT is the counting time (second) and W_s is the original weight of the sample (kg).

2.6.3 Detection limits

According to the Currie criteria, the minimum detectable levels, MDLs (Bq kg^{-1}), is defined so that, if an amount of a radioisotope equals to the MDL exists in the sample, it will be detected with 95% probability (Brun et al. 2003; Alvarez et al. 1995). In the condition of the present research, we used the following relation to determine the MDL values:

$$\text{MDL} = \frac{2.71 + 4.65\sqrt{B \times \text{CT}}}{\text{CT} \times W_s \times R \times \varepsilon} \quad (3)$$

2.6.4 Decay test for ^{90}Y samples

To check the interfering radionuclides such as ^{40}K in the counted samples, the decay curve should be investigated for each of the prepared sample by counting the ^{90}Y source obtained from milk for several hours, but by considering the large number of samples, it was done randomly only for some of them. The decay curves were then controlled by the half-life of ^{90}Y (64.1 h). Figure 2 presents the results for one of the attempts and can elucidate our statements.

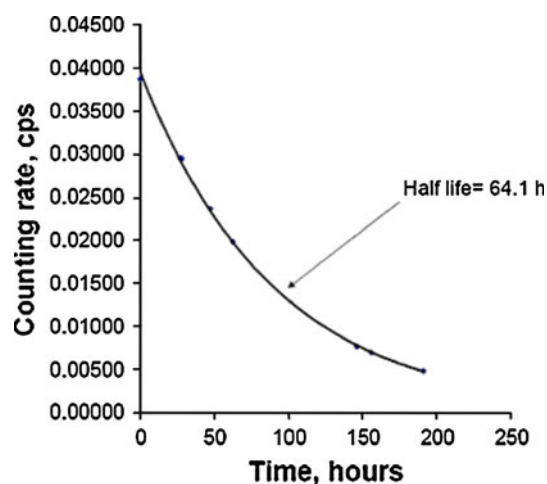


Fig. 2 ^{90}Y decay curve. This curve is fitted to the half-life of ^{90}Y ($R^2 = 0.9$) and confirms the purity of our samples

2.6.5 Assessment of effective annual dose due to ingestion

For the assessment of the effective annual dose rate D (e.g., nSv year^{-1}), following equation was applied (ICRP 1994, 1996; Till and Moore 1988):

$$D = A \times U \times g \quad (4)$$

where A is the ^{90}Sr concentration in milk samples (Bq kg^{-1}), U is annual consumption of ^{90}Sr , and g is effective dose coefficient of ^{90}Sr , which is 28 nSv Bq^{-1} for adults and 230 nSv Bq^{-1} for the children younger than 1 year old.

3 Result

Activity concentrations of ^{90}Sr (Bq kg^{-1} , dry weight) in the milk and milk powder samples and accuracy results are summarized in Tables 1 and 2, respectively. The average activity concentration of ^{90}Sr in the investigated milk samples was $0.225 \pm 0.042 \text{ Bq kg}^{-1}$. Also, the average activity concentration for the milk powder samples was determined as $0.216 \pm 0.024 \text{ Bq kg}^{-1}$. Comparison of the determined activity concentrations and the minimum detectable levels shows that the results are mostly close to or below the detection limits. The data below the MDL values were omitted in our calculations.

To estimate biological hazard from strontium-90, which can occur due to milk consumption, the effective dose is calculated using Eq. 4. Effective dose is based on the risks of radiation-induced health effects and the use of International Commission on Radiological Protection (ICRP) biokinetic model that provides relevant conservation factors to calculate effective dose from the total activity concentration of radioisotope measured in the food samples (ICRP 1994,

Table 1 Activity concentration of ^{90}Sr (Bq kg^{-1} , dry weight) in the milk samples and accuracy results

Sample ID	Gross beta count rate (cpm)	Chemical recovery of Yttrium (%)	Analyzed ash weight (kg)	Ash to dry weight ratio	The decay time (h)	Activity concentration of radio Strontium ^a (Bq kg^{-1})	Minimum detectable Level (Bq kg^{-1})
M-1	3.867	92.97	0.00373	0.06544	37.17	1.615 ± 0.126	0.178
M-2	1.650	52.90	0.00485	0.06831	49.77	0.336 ± 0.116	0.250
M-3	1.768	100.00	0.00980	0.06490	42.80	0.105 ± 0.030	0.062
M-4	1.638	89.64	0.00860	0.06143	57.37	0.105 ± 0.035	0.075
M-5	2.140	89.91	0.01069	0.06640	52.88	0.225 ± 0.034	0.065
M-6	1.847	85.14	0.00625	0.06579	16.48	0.174 ± 0.057	0.116
M-7	1.903	100.00	0.01280	0.06632	15.15	0.080 ± 0.024	0.049
M-8 ^b	1.362	90.60	0.01115	0.05000	12.08	0.005 ± 0.020	0.047
M-9	2.522	95.16	0.01302	0.06817	10.87	0.167 ± 0.030	0.052
M-10	2.055	96.63	0.01241	0.05826	18.33	0.098 ± 0.024	0.046
M-11	3.041	92.07	0.01252	0.06804	10.65	0.256 ± 0.035	0.056
M-12	2.928	95.61	0.01076	0.07126	13.57	0.290 ± 0.040	0.065
M-13	2.280	88.32	0.00971	0.06185	11.88	0.177 ± 0.037	0.068
M-14	2.055	89.13	0.01249	0.05098	2.80	0.078 ± 0.022	0.043
M-15	1.790	84.99	0.01310	0.04629	12.35	0.050 ± 0.019	0.039
M-16	1.796	91.68	0.01266	0.08275	0.80	0.077 ± 0.032	0.067
M-17	1.853	91.92	0.01334	0.06841	13.38	0.077 ± 0.026	0.052
M-18 ^b	1.368	90.00	0.01082	0.06479	14.37	0.008 ± 0.026	0.063
M-19	2.365	90.48	0.00801	0.04740	17.23	0.185 ± 0.034	0.062
M-20	2.190	56.73	0.00993	0.08345	12.52	0.331 ± 0.074	0.139
M-21	3.473	96.96	0.01043	0.06907	13.68	0.383 ± 0.043	0.064
M-22	2.089	89.79	0.01203	0.06365	14.20	0.119 ± 0.029	0.055
M-23 ^b	1.548	92.31	0.00999	0.03568	13.95	0.023 ± 0.016	0.036
M-24 ^b	1.661	94.20	0.01187	0.06182	17.05	0.051 ± 0.024	0.052
M-25	1.807	92.16	0.00622	0.06479	14.92	0.145 ± 0.051	0.106
M-26	1.909	97.53	0.00660	0.05893	17.82	0.146 ± 0.043	0.086
M-27	2.066	92.13	0.01343	0.04553	12.58	0.071 ± 0.018	0.035
M-28	2.055	92.43	0.01285	0.06178	27.48	0.115 ± 0.025	0.049

^a Values are the activity obtained \pm SD^b Below minimum detectable level (MDL)

1996). Estimation of the radiation-induced health effects associated with the intake of radionuclide in the body is proportional to the dose delivered by the radionuclide while resident in the various organs. By considering that the samples used in this study do not consumed merely in their production area, the average activity concentration was used for the dose estimation. By these explanations, with regard to the average activity concentration of ^{90}Sr in the milk and milk powder samples (0.225 ± 0.042 and 0.216 ± 0.024 Bq kg^{-1} , respectively) and the rate of milk consumption (75 kg year^{-1} for milk consumption in adults reported by the Milk Industry of Iran, 63 g day^{-1} for milk powder consumption in children at the breast younger than 5 months old, and 158 g day^{-1} for children at the breast older than 5 months old), the effective dose of ^{90}Sr due to milk

consumption was calculated as $472.50 \text{ nSv year}^{-1}$ for adults and $1,142.39\text{--}2,865.04 \text{ nSv year}^{-1}$ for infants.

4 Discussion and conclusion

The aim of the present study was monitoring the background level of radioactivity in milk, which is a reliable indicator of the general population intake of certain radionuclides, since it is consumed fresh by a large segment of the population and contains several of the biologically significant radionuclides (ERD 2001). By considering this purpose, although many rapid methods have been developed for the determination of strontium in milk, we used a combination of two common methods

Table 2 Data indicating the activity concentration of ^{90}Sr (Bq kg^{-1} , dry weight) in the milk powder samples and accuracy results

Sample ID	Gross beta count rate (cpm)	Chemical recovery of Yttrium (%)	Analyzed ash weight (kg)	Ash to dry weight ratios	The decay time (h)	Activity concentration of radio Strontium ^a (Bq kg^{-1})	Minimum detectable Level (Bq kg^{-1})
MP-1	2.500	93.81	0.00523	0.02710	61.00	0.283 ± 0.030	0.052
MP-2	2.466	86.97	0.00791	0.02737	10.67	0.115 ± 0.021	0.037
MP-3	1.723	88.71	0.01132	0.03262	14.50	0.034 ± 0.014	0.031
MP-4	2.218	86.04	0.00994	0.02873	13.62	0.078 ± 0.017	0.032
MP-5	3.035	95.13	0.01184	0.04199	16.28	0.171 ± 0.022	0.035
MP-6	2.939	89.07	0.00611	0.02057	14.63	0.161 ± 0.022	0.036
MP-7 ^b	1.633	85.52	0.01053	0.03761	17.47	0.035 ± 0.018	0.039
MP-8	8.253	91.05	0.00576	0.02341	11.60	0.790 ± 0.043	0.042
MP-9 ^b	1.368	89.37	0.00681	0.02702	12.95	0.005 ± 0.018	0.042
MP-10	2.145	74.61	0.02610	0.08392	13.12	0.092 ± 0.021	0.041

^a Values are the activity obtained \pm SD^b Below minimum detectable level (MDL)

routinely used in monitoring process with some modifications. The methods were well established and suitable for our experiments. The quality control and assurance of the combined methods were also obtained by standard samples prepared with an IAEA-certified reference materials (Milk-152) to ensure its quality. The quality control and assurance of the two methods have also been investigated in some researches (Bem et al. 1991; IAEA 1993a), and our results confirmed their data too (our assay gave the average chemical recovery above 91%, and the average of the method efficiency was obtained 85% which is in accordance with the previous investigations). Moreover, rapid methods are required for the application of the EURATOM regulation, in emergency situations, in order to screen a large number of milk samples in a short period of time and to minimize the contamination of all the dairy food chain. The EURATOM regulation No. L2218/89 published in the Official European Community Journal on the July 22, 1989 (Règlement EURATOM 1989) gives the limits of radio strontium activity in infant food and dairy products as 75 and 125 Bq kg^{-1} . This regulation defines the levels for radionuclides in foods, which are set into power in early case of nuclear emergencies. For these situations, by considering the high allowable concentration limits, a low detection limit is not of first importance in comparison with the analytical time (Baron et al. 2004; Chang et al. 2004; Horwitz et al. 1992; Jassin 2005; Tait et al. 1997, 1999; Lee et al. 2002; Mikulaj and Svcc 1993) and fast methods are required, but in the present study, by the aim of the detection of very low background concentrations, it is not necessary to use new methods, which are also very expensive because of the low regeneration of the exchange and affinity resins.

A lot of studies have been carried out to estimate the transmission of strontium from soil forage to cow milk, and their finding has been used for predicting radionuclide concentration in food stuffs and dose impact to man. Although these studies have shown that the values are strongly influenced by many physical, chemical, and biological factors such as geographic site, plant, and cow species, they are in agreement that the strontium transmission to food chain is highly efficient (Comar and Wasserman 1964; Paasikallio et al. 1994). In this study, by considering the strontium concentrations in the analyzed milk samples ($0.225 \pm 0.042 \text{ Bq kg}^{-1}$), we can deduce that the strontium level in the studied area is obviously low. However, to get more accurate results, some other studies should be carried out to determine the strontium-90 concentration in forages and soil samples or probably estimation of the transfer factor for this radionuclide.

Data obtained here can also provide an opportunity to verify any impact from the ingestion of strontium-90 in the people who consume milk. The amount of calculated effective dose ($472.50 \text{ nSv year}^{-1}$ for adults and $1,142.39\text{--}2,865.04 \text{ nSv year}^{-1}$ for infants) show that infants under the age of one are more sensitive but all the doses are still a very small fraction of the natural background average annual dose received by human ($\approx 2.4 \text{ nSv year}^{-1}$), so they are sufficiently too low to pose a risk to human health.

Since ^{90}Sr is an artificial radioisotope and can occur only during uncontrolled nuclear activities (such as Chernobyl accident, weapon fallout and), so in this research, a suitable method for monitoring process was investigated and useful information of the amount of ^{90}Sr deposition over Tehran province was obtained. These data will be useful in emergency situations.

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