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ABSTRACT

A solvent extraction method, using tributyl phosphate, for rapid separation of Strontium-90 in milk and other food samples has been presented in this report in view of large number of samples received after Chernobyl accident for checking radioactive contamination. The earlier nitration method in use for the determination of ^{90}Sr through its daughter ^{90}Y takes over two weeks for analysis of a sample. While by this extraction method it takes only 4 to 5 hours for sample analysis. Complete estimation including initial counting can be done in a single day. The chemical recovery varies between 80 - 90% compared to nitration method which is 65 - 80%. The purity of the method has been established by following the decay of Yttrium-90 separated. Some of the results obtained by adopting this chemical method for food analysis are included. The method is, thus, found to be rapid and convenient for accurate estimation of Strontium-90 in milk and food samples.

Introduction:

Strontium-90 present in the environment due to fall-out from nuclear explosions constitutes a long term biological hazard because of its long radioactive & biological half lives and chemical similarity to calcium. This radionuclide finds its way to human bone along with calcium and can be potentially hazardous, if present in large amounts. Therefore, it is necessary to determine the levels of Strontium-90 in milk and other food samples to assess the likely hazard to the population.

Strontium-90 is usually determined through its daughter product Yttrium-90 because of its higher beta energy (2.26 MeV) and short half-life (65.0 hrs) compared to Strontium-90 (0.54 MeV, 28.8 Yrs). The half-life of 65.0 hrs of yttrium-90 is sufficiently short to follow decay. This helps in checking the chemical purity of ^{90}Y separated and the chemical procedure in use. It also avoids correction for self-absorption factor which otherwise is necessary for ^{90}Sr measurement.

In the chemical procedure in use in this Laboratory for the separation of strontium and subsequently for yttrium, the sample after dissolution in an acid is concentrated by carbonate precipitation. Strontium is then separated from bulk of calcium by successive fuming nitric acid precipitations under ice-bath. The separated strontium is purified by barium chromate and hydroxide scavengings and stored for yttrium-90 to grow. It takes nearly 14 days to establish near complete equilibrium between ^{90}Sr and ^{90}Y . This is a long period to wait. Besides, the time taken for the chemical separation itself takes 2-3 working days.

After Chernobyl accident, it is mandatory for all food items to be checked for radioactivity contamination levels. To meet the needs for checking large number of samples, an attempt has been made to use solvent extraction method (1,2) which will require a shorter time period than the afore mentioned method of analysis involving more than two weeks. In this method, using tributyl phosphate (TBP), it takes 4-5 hours for sample analysis and the complete estimation including initial counting can be done in a single day. The chemical recovery varies between 80-90% compared to 65-80% by the nitration method (3). This report describes the chemical procedure developed in detail and some of the results obtained adopting this chemical method for food samples. A decay curve for separated yttrium-90 is also shown to establish purity of the method adopted for separation.

Sample Preparation:

Milk samples for analysis, are collected from city's major dairy centres at Aarey and a neighbouring place, Anand. Samples of 10 liters of liquid milk are obtained on monthly basis. Other food samples like food-grains (5kg), fresh vegetables (2.5 kg) and processed food items like tea, milk powder etc. (1 kg) are obtained from local markets. These samples are weighed and dry ashed in a stainless steel container at a temperature of about 350°C to avoid losses. The final ash, free from carbonaceous material, is taken for chemical analysis.

Chemical Procedure:

In this method yttrium-90, from already equilibrated ash solution, is directly extracted with TBP. It is further purified by selective stripping from TBP and back extracted. Finally yttrium is precipitated as an oxalate for counting. The detailed procedure is as under:

Take 15 g sample ash in a beaker and add to it 60 mg strontium and 20 mg yttrium carriers. Dissolve the mixture in 70

ml conc. HNO₃ on a hot plate. After dissolution, filter the solution through whatman filter paper (541). Ignite undissolved carbonaceous residue, if any, along with filter paper in a crucible and dissolve the residue in the same filtrate. Maintain the same volume of the solution i.e., 70 ml. Add slowly and dropwise 130 ml. of fuming nitric acid (96%) under ice-bath and shake thoroughly by stirring with glass rod till white crystals of strontium nitrate appear. Allow the crystals to settle. Decant off the supernate in a 250-ml. separating funnel, leaving crystals in the beaker. Use this precipitate of strontium nitrate for estimating ⁸⁷Sr, if required.

To the separating funnel containing supernate, add 50ml. equilibrated TBP and shake the funnel well for 5 minutes, taking precaution to release the gases formed in the funnel from time to time. (The equilibrated TBP is obtained by shaking the stock TBP with conc. HNO₃). Allow the two layers to separate. Transfer the aqueous layer in to a fresh 100ml. separating funnel, leaving the TBP layer in the original funnel. Add 25ml. TBP to the aqueous layer and shake the funnel again for 5 minutes taking all the precautions as earlier. Allow the two layers to separate. Reject the aqueous layer and add TBP layer to original 250ml. separating funnel. Add 50ml. distilled water and shake well for 5 minutes. Allow the two layers to separate and transfer the aqueous layer to a beaker. To TBP layer, add additional 25ml. distilled water and shake again in an identical manner. Remove aqueous phase and add it to earlier aqueous phase. Add 25ml. 3M HNO₃ to TBP layer and shake once again for 5 minutes. Remove the nitric acid layer and add it to aqueous layer. Reject TBP layer. To a beaker now containing aqueous layer add dil. ammonia solution till alkaline. White ppt. of yttrium hydroxide appears. Cool the solution and filter through whatman 541. Dissolve the ppt. on filter paper in a minimum quantity of 6M HCl. Add 10ml. oxalic acid (8%) to precipitate yttrium as oxalate (pH 1.5-2.0). Warm the ppt. to coagulate. Filter through filter stick using 2.5cms. weighed filter paper disc. Wash the ppt. first with water and then with alcohol. Dry the ppt. under infra-red lamp, weigh it to get chemical recovery and mount for counting, covering it with thin mylar film.

Strontium-89 can be determined in the nitrate precipitate along with Strontium-90. The separated strontium is purified by different scavengings and finally precipitated as carbonate for counting. ⁸⁷Sr is counted along with ⁹⁰Sr and using aluminium cut-off absorber, ⁸⁷Sr is determined.

Counting Technique:

The samples are counted on a standard planchet using a low-level beta counting set-up, having background of about 30

counts per hour. The counting of yttrium oxalate is done 3-4 times at different time intervals to obtain decay curve for yttrium-90. The decay curve is extrapolated to calculate strontium-90 activity in the sample. The counting time for the sample and the background are so selected that the overall error in counting is about 10% for samples having count rate above 0.37 Bq. and less than 20% for samples having lower concentrations.

Results and Discussions:

The chemical procedure described above is rapid and quantitative and yttrium separated is free from contamination of other fission products. The purity of the sample as well as the accuracy of the estimation is checked by following the radioactive decay of the sample. Figure 1 gives a typical decay curve of one of the chemically separated yttrium sample with half-life of 64.0 hrs.

The concentration levels obtained by this new solvent extraction method in some of the samples were compared with the earlier nitration method. Three such milk powder samples were analysed by both these methods. The results of these analyses are given in Table 1. These results compare well with each analysis.

The activity levels of strontium-90 for some of the samples collected from local market are given in Table 2. It can be seen from table that the ^{90}Sr concentration levels in tea samples are higher compared to other food samples. This is because of higher rainfall and consequently higher fallout received at places where tea is grown in India(4).

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Table 1. Results of Strontium-90 Activity Compared by Two Different Methods.

Sample Number	Method used	
	Nitrate Precipitation (Bq/Kg)	Solvent Extraction (Bq/Kg)
Milk Powder (S-2B)	0.16	0.19
Milk Powder (S-S)	1.70	1.50
Milk Powder (M-C)	0.80	0.83

Table 2: Strontium-90 Concentration Levels in Various Food Samples at Bombay.

Sample type	⁹⁰ Sr Conc. (Bq/Kg)
1. Milk	
a) Aarey(Whole)	0.60
b) Consumer Society	0.40
c) Milk Powder-NDDB	
Serial No.28	0.16
" No.29	0.13
" No.31	0.19
" No.32	0.50
" No.34	0.06
" No.35	0.20
" No.G-1	0.60
" No.G-2	0.07
" No.G-14	0.03
2. Vegetables:	
a) Fenugreek (Methi)	0.16
b) Amarnth (Lal Muth)	0.24
c) Sweet potato	0.09
d) Potatoes	0.18
e) Spinach	0.08
3. Pulses & Cereals:	
a) Thoor dal	0.80
b) Masoor dal	0.60
c) Kabli channa	0.15
4. Tea:	
a) Tata Tea (Tree Brand)	12.30
b) Brook Pond (Red Lable)	13.70

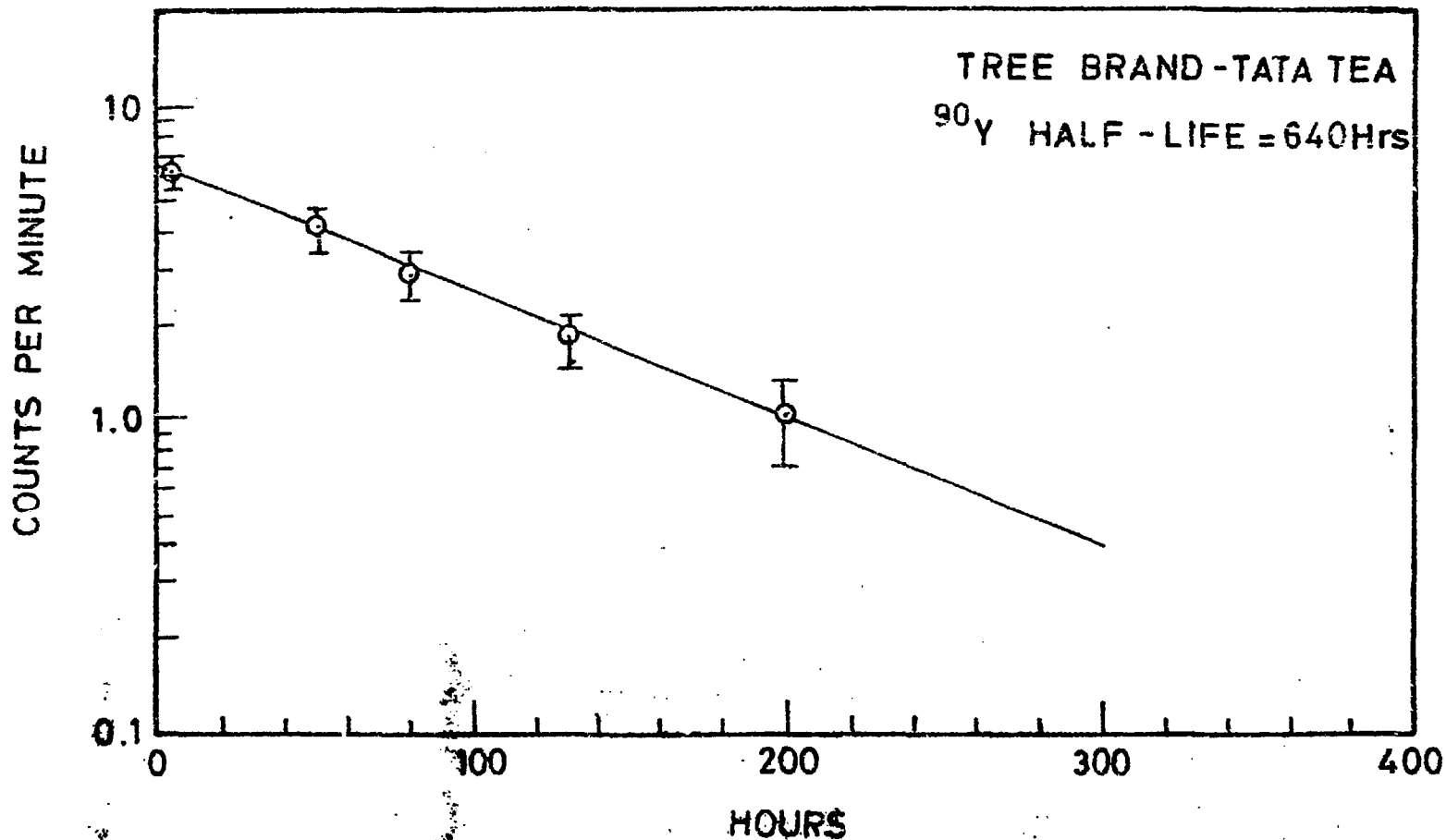


FIGURE: 1. A TYPICAL DECAY CURVE OF YTTRIUM-90 CHEMICALLY SEPARATED USING TBP METHOD.

