A Rapid Method for Strontium-90 Analysis by HDEHP Solvent Extraction - Application to Large Volume Sea water Samples

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Abstract

The application of HDEHP solvent extraction to large volume sea water samples is described. The results obtained by this technique compares favourably with those obtained by the conventional procedures.

Résumé

L'application de l'extraction par solvent HDEHP, à des échantillons d'eau de mer de grand volume, est décrite. Les résultats obtenus par èctte technique sont favorablement comparés avec ceux obtenus par les méthodes conventionnelles.

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One conventional method for 90 Sr determination in environmental samples uses fuming nitric acid for the separation of strontium from large amounts of calcium. This procedure is complicated and time consuming and becomes particularly cumbersome where large numbers of samples are to be analyzed and/or when the size of the sample is large (for example 60 l of sea water). A more rapid method involving solvent extraction of 90 Y (the daughter product of 90 Sr) using di-2-ethylhexyl phosphoric acid (HDEHP) and the tertiary amine methyltricapryl ammonium chloride (Aliquat 336) has been shown to produce satisfactory results in the measurement of small volumes of sea water measured in the intercalibration programme. The application of this rapid method to large volumes of sea water has been successful. Samples can be counted within 2-3 hours after the start of the analysis, thereby eliminating the 90 Y ingrowth period of 15 days normally required in the conventional procedure. Recoveries of 70-80 % can be obtained as compared to 40-60 % using the conventional method.

The procedure adopted for large volume sea water samples is as follows :

1. 30 kg of sea water is adjusted to pH 1 - 1.5 with concentrated HC1. Fifteen milligrams of yttrium carrier is added, mixed well and the solution equilibrated for 24 hours.

2. The sample is then extracted, by vigorous stirring, with 250 ml of HDEHP solution (0.45 M in n-neptane) for 2 minutes. After phase separation, the aqueous phase is discarded and the organic phase is washed 5 times each for 1 minute with 100 ml of 0.5 M HC1. All aqueous washes are discarded.

3. Yttrium is stripped from the organic phase 5 times each for 1 minute with 50 ml of 9M HC1 and the aqueous solution drained into a clean 500 ml separatory funnel. Two hundred and fifty ml of aliquat 336 (30 % v/v in toluene) is added and the solution extracted by shaking for 2 minutes.

4. The aqueous phase is then drained into a 1000 ml beaker and concentrated NH_4OH is added to precipitate $Y(OH)_3$. The solution is heated to boiling, then allowed to cool and finally filtered through

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fine filter paper. The precipitate is washed several times with a dilute solution of NH₄OH. The precipitate is then dissolved from the filter paper with 6M HCl and a second Y(OH₃) precipitation performed by the addition of concentrated NH_4OH . The resulting mixture is again boiled, allowed to cool and again filtered through a fine filter paper.

5. This second filter is dried and mounted for β^- counting. Following repeated countings to verify the radiochemical purity of the ⁹⁰Y, the precipitate is ignited in a crucible at 800° C to constant weight and the yield is determined by weighing the resultant Y_2O_3 .

Seaweed samples have also been analyzed by the above procedure with the modification of adding yttrium carrier to the dried sample, igniting the resulting mixture in a muffle furnace and dissolving the resulting ash in concentrated HNO₃. Following the adjustment of the pH 1-1.5, the remaining steps are those described above. Table 1 lists the results obtained on samples analyzed in the intercalibration programme by the conventional method. The results indicate reasonable agreement.

RAPID METHOD			CONV	CONVENTIONAL METHOD	
Sample size		⁹⁰ Sr found		Sample size	
Seaweed AG-I-1 Sea water SW-I-2 Atlantic water	10g 11 201	$\begin{array}{rrrr} 10.3 \ \pm \ 0.7 \ pCi/g* \\ 56.0 \ \pm \ 0.6 \ pCi/kg \\ 0.11 \ \pm \ 0.05 \ pCi/kg \end{array}$	10g 51 501	$\begin{array}{rrr} 10.2 \ \pm \ 0.2 \ \ pCi/g* \\ 53.6 \ \pm \ 0.3 \ \ pCi/kg \\ 0.11 \ \pm \ 0.05 \ \ pCi/kg \end{array}$	

TABLE 1.: Comparison of 90Sr analyses by rapid and conventional methods. *Errors represent propagated errors at lσ.