Extraction of ³⁵sulphur from pile-irradiated potassium chloride

Introduction

³⁵S (half-life 88 days) is widely used as a tracer in agricultural and biochemical studies. It is generally obtained in the carrier-free state by the ³⁵Cl $(n, p)^{35}$ S reaction using KCl as the target material. ³⁵S thus produced is radiochemically contaminated with ³²P (formed by the ³⁵Cl $(n, a)^{32}$ P reaction), from which it must be separated.

A number of methods have been reported by different $\operatorname{authors}^{1,2,3}$ for the separation of ${}^{35}S$ from various targets, *viz.* CCl_4 , KCl, NaCl, FeCl₃ etc. In the conventional method⁴, followed in many countries, the pile-irradiated KCl is dissolved in water and the solution passed down a cation-exchange column; K⁺ ions are adsorbed on the column while ${}^{32}P$ and ${}^{35}S$ pass out with the effluent solution as phosphate and sulphate ions. ${}^{32}P$ is then removed either by coprecipitation with La(OH)₃, or by passage through a column containing aluminium shavings.

This paper describes a new method for the separation of ³⁵S from pile-irradiated KCl in which the separation of carrier-free ³⁵S has been much simplified through the use of an anion-exchange column. The separation of ³⁵S from ³²P and K⁺ is effected in a single step and ³⁵S is obtained in a radiochemically pure form. The method has been tried with success for the isolation of millicurie amounts of ³⁵S and is being adopted for the routine production of this isotope at the Trombay Establishment.

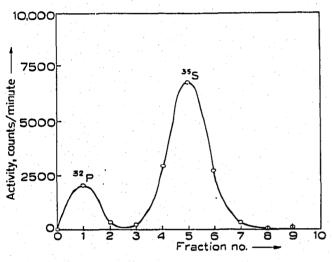
Experimental details

15 g samples of KCl were irradiated with neutrons, in the Apsara reactor, Trombay, at a flux of $3 \cdot 10^{11} n/\text{cm}^2/\text{sec}$ for about 100 h. The ⁴²K (half-life 12.4 h) formed by the ⁴¹K(n, γ)⁴²K reaction was allowed to decay before processing. A negligibly small amount of ³⁶Cl is formed during such a short irradiation.

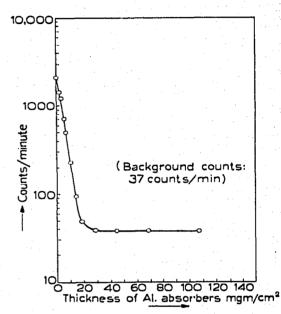
Measurements of radioactivity were made using an end-window type G.M. counter (window thickness 2.5 mg/cm^2) in conjunction with a scaling unit.

The irradiated KCl was dissolved in water and made up to a known volume, which served as a stock solution for all the experiments. A suitable aliquot was pipetted out and diluted with an appropriate volume of water, so as to give a solution approximately 0.1 M in KCl. A small amount of phosphate carrier (5 mg) is added in order to reduce (i) the adsorption of ³²P on glass, and subsequently (ii) the contamination of ³⁵S by ³²P during the acid elution. This solution was passed down an anion-exchange column containing the resin Amberlite IRA-400 (40-60 mesh) in the chloride form. The effluent was collected separately and checked for any activity. The column was next washed with 100 ml distilled water and the effluent again checked for activity. A certain amount of ³²P was detected in both these fractions, indicating that it is poorly fixed on the column. Finally the column was eluted with 0.1 M HCl and fractions of about 50 ml each were collected separately. The activity in each of these fractions was measured. 0.2 ml from each fraction was deposited on a glass planchet and then evaporated to dryness under an infra-red lamp. They were counted in an end-window G.M. counting assembly, first without an absorber, then with an aluminium absorber of thickness 30 mg/cm² interposed between the source and the counter window. Such an absorber cuts off all β radiations from ³⁵S (E_{\max} 0.17 MeV), so that any contamination due to ${}^{32}P(E_{max} I.7 MeV)$ would easily be detected. It was found that the first two or three fractions (100-150 ml) contained the ³²P free of any ³⁵S, and that the later fractions contained pure ³⁵S. A typical elution curve is shown in Fig. 1. The curve shows two distinct peaks with good resolution, which indicates the possibility of se-

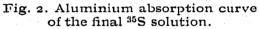
parating radiochemically pure, carrier-free ³⁵S from irradiated KClusing an anion exchanger.







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The recovery of ³⁵S has been found to be almost quantitative. The column was checked for any residual activity on it by elution with concentrated HCl. No activity was detected in the effluent solution.

Radiochemical purity

In Fig. 2 is shown the aluminium absorption curve taken on an aliquot from a mixture of all the fractions containing pure ³⁵S. It is evident that the ³⁵S obtained is radiochemically pure.

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