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PREPARATION OF 35-SULPHUR LABELLED SULPHUR TETRAFLUORIDE

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SUMMARY

The synthesis of ^{35}S-labelled sulphur tetrafluoride from ^{35}S-labelled elemental sulphur and iodine pentafluoride is reported.

INTRODUCTION

The preparation of sulphur tetrafluoride is well documented [1,2], and $S^{18}F_4$ is readily prepared by 18-fluorine exchange between SF_4 and alkali metal [^{18}F]-fluorides [3]. However the synthesis of $^{35}SF_4$, which was required for a ^{35}S (β^-_{max}=0.167MeV, $t_{\frac{1}{2}}$=87.4d) tracer study of the fluoride ion catalysed chlorofluorination of SF_4[4], has apparently not been reported. The most obvious route is via the reaction of sulphur dichloride with sodium fluoride in acetonitrile [1,2], but this is not very suitable for $^{35}SF_4$ as the maximum radiochemical yield attainable is only 33.3%.

An alternative route is the fluorination of elemental sulphur by iodine pentafluoride, a reaction first mentioned by Moissan [5], and reinvestigated more recently, equation (1), [1].

$$5S + 4IF_5 \rightarrow 5SF_4 + 2I_2 \qquad (1)$$

The synthesis of $^{35}SF_4$ by this means, on the millimole scale with a specific activity suitable for tracer work, is now reported.

EXPERIMENTAL

All manipulations involving volatile compounds were performed in flamed out Pyrex or passivated Monel metal vacuum lines to avoid hydrolysis and to ensure adequate shielding of emitted β^- radiation.

Rhombic [^{35}S]-sulphur (1mCi, specific activity $<$ 30mCi mg-atom^{-1}, The Radiochemical Centre, Amersham) was diluted with inactive S_8 (7.0mmol) by dissolution in dry CS_2. The solution was transferred to a Monel metal pressure vessel (90cm^3, Hoke Inc.) and CS_2 removed under vacuum. Iodine pentafluoride (44.8mmol, Fluorochem Ltd.) which had been purified by successive treatments with predried NaF then Hg metal, was added by vacuum distillation. The mixture was allowed to react at 373K for 5h, then 473K for 48h. After reaction, volatile material was removed at 195K and condensed in a similar vessel over predried NaF. The yield of $^{35}SF_4$ at this stage was 67%.

The product was purified by reaction with BF_3 at 195K forming the adduct $^{35}SF_3^+BF_4^-$ [6]. Unreacted material was removed by pumping at this temperature, and the adduct decomposed by adding the calculated quantity of dry diethyl ether at 195K [7]. The i.r. spectrum of the $^{35}SF_4$ (33.8mmol, 60% yield) produced, contained no bands attributable to SF_6, SOF_2, or SiF_4. Heating the IF_5, I_2, $^{35}S_8$ mixture which remained produced a further quantity of $^{35}SF_4$.

^{35}S Activity was determined using an evacuable Pyrex vessel (421cm^3) fitted with an end-window Geiger Muller tube. $^{35}SF_4$ was diluted with purified, inactive SF_4 (1:4 ratio) to obtain an acceptable working specific activity, approximately 75 counts s^{-1}mmol^{-1}. A linear count rate _vs._ pressure relationship was obtained over the range 60-200 torr.

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