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NOTE

The Preparation of High Purity $H_2{}^{35}S$

C.D. Collins* and J.N.B. Bell

Imperial College,
Department of Biology,
Silwood Park,
Ascot,
SL5 7PY

SUMMARY

$H_2{}^{35}S$ was synthesised by the reaction of H_2 with elementary [^{35}S] sulphur at 600°C with 100 mg of pumice added as a catalyst. The resulting gas was purified by placing the preparation vessel in a pack of ice. A yield of 46% was produced.

Key words: hydrogen sulphide, sulphur-35, preparation.

INTRODUCTION

The method of preparation of hydrogen [35]S sulphide described here was developed, in order to provide a pure source of the labelled gas for studies on its uptake and subsequent translocation by crops. This type of work necessitates the use of pure gases in order to avoid the confounding effects of trace gases containing the same label, that might be deposited on crops at a high deposition velocity, thereby resulting in a major artefact. Furthermore it is important to use a gas with a high specific activity so that the radionuclide can be traced throughout the life of a crop, while at the same time avoiding concentrations which might be phytotoxic. In the case of hydrogen sulphide a number of studies have demonstrated phytotoxic effects [1,2].

In normal laboratory circumstances H_2S is prepared by the action of a dilute acid upon iron sulphide using the Kipps Apparatus. However a purer formation can be prepared by the action of the constituent elements with pumice as a catalyst at 600°C [3]. The latter was the method chosen here both for its high purity and the ready availability of high specific activity sulphur 35 (30.5 T Bq g^{-1} atom, Amersham International plc, U.K.). Possible contaminants, H_2S_2 (b.p. 71 °C) and H_2S_3 (b.p. v. high) were removed by placing the reaction vessel after heating in an oven for 6 hrs on a bed of ice, exploiting the much lower boiling point of H_2S (b.p. -60°C). Following this preparation the gas was stored in an aluminium cylinder pressurised with 'O' free nitrogen.

MATERIALS AND METHODS

$H_2{}^{35}S$ was synthesised in a glass vessel similar to that of Kluczewski *et al* (1984) [4] (Fig 1.), but this was enlarged to give a higher overall yield as the gas was to be used for

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*Author to whom correspondance should be sent.

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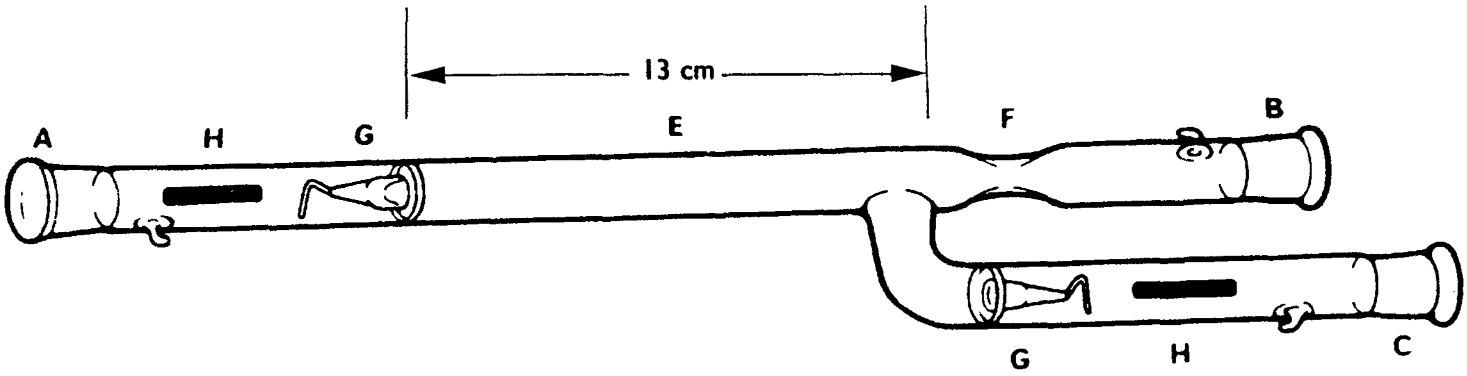

Figure 1. The sulphur-35 plus carrier and pumice catalyst are injected into compartment E, the reaction vessel is then filled with H2 via B. The vessel is then sealed by heating the constriction at F. Following heating, the product is flushed with N2 in through C and out via A, after breaking the seals (G) with glass coated iron slugs (H). The entire construction was made of borosilicate glass (1.8 mm wall thickness), with spring secured B14/23 ground glass connections.

several fumigations over a long time period, thereby requiring more carrier. Elementary sulphur in toluene (185 MBq, Amersham international) was made up to 1 ml with a carrier solution containing 17 mg ml^{-1} sulphur in toluene. Pumice (0.05 g) was placed into the main compartment of the reaction vessel and the sulphur in toluene solution was then injected on to it. 'O' free nitrogen was then passed over this mixture until it had evaporated to leave yellow sulphur crystals. Using a hand held vacuum pump and a three way valve the reaction vessel was evacuated and pressurised with hydrogen 10 times, then evacuated to 0.5 atmospheres to reduce the likelihood of breakage during the subsequent heating.

The reaction vessel was then sealed by heating the constriction with an oxygen and natural gas torch. The vessel was then placed in an oven, which was heated to 600°C and maintained at that temperature for 4 hrs. After the reaction vessel had cooled to room temperature it was packed in ice to reduce the temperature of the vessel and remove any impurities of H_2S_2 (b.p. 71 °C) and H_2S_3 (b.p. v. high). After 15 minutes the contents of the vessel were passed into an evacuated aluminium cylinder at a flow rate of c. 50 cm^3 min^{-1} after the seals at the end of the reaction compartment had been broken. The cylinder was then pressurised with 'O' free nitrogen to c. 2000 psi.

Analysis of the gas from the cylinder gave a mean H_2S concentration of 23.9 $\pm$ 9.20 ppm following three analyses on a Hewlett Packard 5738A gas chromatograph with a flame photometric detector. This resulted in a yield of approximately 46% and a hence a specific activity of 5 GBq g^{-1}.

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