

SHORT COMMUNICATIONS**New possibilities to obtain ^{32}P -labelled inorganic compounds of high specific activity**

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We found that inorganic compounds labelled with ^{32}P such as P_2S_5 , PCl_3 , PSCl_3 , POCl_3 , and H_3PO_4 can be obtained by a new simple method, with good yields and a high specific activity. The carrier free ^{32}P resulting through the nuclear reaction $^{32}\text{S}(n,p)^{32}\text{P}$ from elementary sulphur was concentrated on red phosphorus and active carbon. Then, by chemical reactions with phosphorus pentachloride or elementary sulphur, PCl_3 , POCl_3 , PSCl_3 , and P_2S_5 labelled with ^{32}P are obtained. Carrier free $\text{H}_3^{32}\text{PO}_4$ is obtained by desorption of radiophosphorus from active carbon with 2M HNO_3 at 80°C .

To prepare compounds labelled with ^{32}P for medical, biological, agricultural use, etc. it is necessary to have intermediate inorganic substances such as $^{32}\text{PCl}_3$, $^{32}\text{P}_2\text{S}_5$, $^{32}\text{POCl}_3$, $^{32}\text{PSCl}_3$, $\text{H}_3^{32}\text{PO}_4$ of high specific activity. The neutron irradiation of the respective substances is not employed because of the various products formed by radiolysis and because of the small specific activities obtained⁽¹⁻⁸⁾. Therefore the labelling of these substances was tried by synthesis^(4, 9-20) or isotopic exchange methods.

Because of technological difficulties the labelling by synthesis method was replaced by the isotopic exchange. In the case of the labelling of P_2S_5 by isotopic exchange good results were obtained⁽²¹⁾, but for PCl_3 , PSCl_3 , and POCl_3 the various techniques tried were unsuccessfully. To obtain $^{32}\text{PCl}_3$ and $^{32}\text{P}_2\text{S}_5$ of high specific activity the carrier free ^{32}P remained in the residue from the distillation of irradiated sulphur was employed⁽²²⁾.

From our study effected on the chemical state of radiophosphorus resulting through nuclear reaction $^{32}\text{S}(n,p)^{32}\text{P}$ from elementary sulphur⁽²³⁻²⁸⁾ a new method was devised for the preparation of P_2S_5 , PCl_3 , POCl_3 , PSCl_3 , and H_3PO_4 labelled with ^{32}P . Our experiences make evident the possibility of obtaining these compounds of a high specific activity, with a high radiochemical efficiency and by very simple methods. For this purpose two important observations were contributed.

a) The possibility of radiophosphorus atoms to stabilize in the elementary sulphur target in an oxidized or elementary form, depending on the presence or the absence of oxygen during the irradiation^(26, 27).

b) The ability of red phosphorus and of active carbon to adsorb the carrier-free radiophosphorus from a solution of irradiated sulphur target.

The adsorption of radiophosphorus on red phosphorus⁽²⁵⁾ and on active carbon⁽²⁸⁾ has been studied and an almost complete (99%) concentration of radiophosphorus on these adsorbents was obtained.

A schematic presentation of the new possibilities to prepare of these substances can be sketched (fig. 1).

As it may be seen, there are two principal directions: one of the preparation of $\text{H}_3^{32}\text{PO}_4$ and $^{32}\text{POCl}_3$, another of the preparation of $^{32}\text{P}_2\text{S}_5$, $^{32}\text{PCl}_3$ and $^{32}\text{PSCl}_3$. For irradiation aerated or unaerated sulphur can be used, depending on the substance prepared.

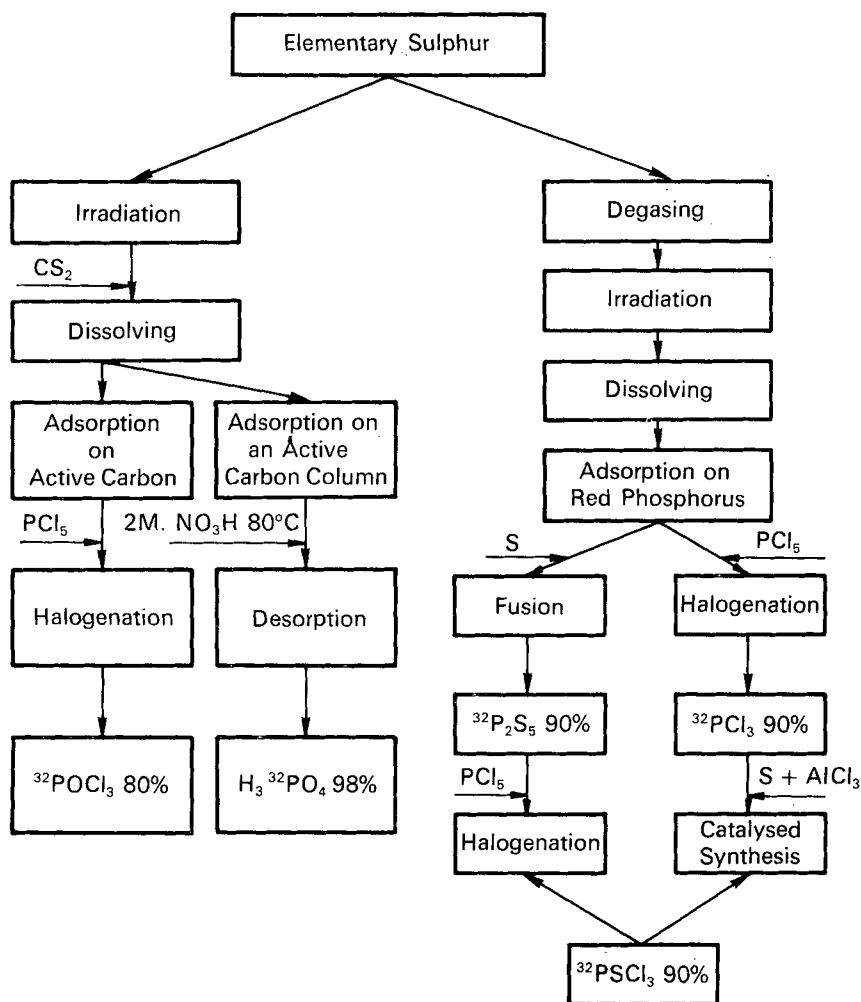


FIG. 1. — Schematic diagram of the chemical procedures.

The adsorption on red phosphorus is achieved by dissolution of the irradiated un-aerated sulphur target in carbon disulphide, then adding an amount of red phosphorus and stirring the mixture by refluxation of the solvent. Then the solvent is removed by decantation and the red phosphorus is employed for the preparation of $^{32}\text{P}_2\text{S}_5$, $^{32}\text{PCl}_3$ and $^{32}\text{PSCl}_3$ in accordance with known techniques.

The adsorption on active carbon can be achieved in two ways for the preparation of $^{32}\text{POCl}_3$ or for the preparation of $\text{H}_3^{32}\text{PO}_4$.

In the case of preparation of carrier-free $^{32}\text{POCl}_3$ into the irradiated aerated sulphur target dissolved in carbon disulphide the active carbon is added and the mixture is stirred as above. After removing of the solvent is added phosphorus pentachloride and a solvent for instance Cl_4C . The carrier-free $^{32}\text{POCl}_3$ is distilled together with solvent. In the case of the preparation of $^{32}\text{POCl}_3$ of high specific activity is added a volume of inactive POCl_3 and by fraction distillation the solvent and $^{32}\text{POCl}_3$ are distilled.

For the preparation of the carrier free $\text{H}_3^{32}\text{PO}_4$ the irradiated sulphur is dissolved in carbon disulphide and it passed on an active carbon column. The desorption of the radiophosphorus is achieved with 2M HNO_3 at 80°C.

The experiment evidences that the technological procedures are very simple and with good yields.

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Synthesis of carbon-14 labeled S-(O, O-diisopropylphosphorodithioate) of N-(2-mercaptoethyl)benzenesulfonamide

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Labeled ^{14}C -benzene S-(O,O-diisopropylphosphorodithioate) of N-(2-mercaptoethyl)benzenesulfonamide of a specific activity of 0.1 mC/m mole was prepared for residue and metabolism studies. The overall yield was 60% of a 95% radiochemically pure product.

S-(O,O-diisopropylphosphorodithioate) of N-(2-mercaptoethyl) benzenesulfonamide* [I] was first prepared and shown to be a selective pre-emergent herbicide by Fanher and Dewald ⁽¹⁾.

To facilitate residue and metabolism studies, [I] was prepared with a carbon-14 label in the benzene ring. The reaction scheme is shown in figure 1.

Uniformly ^{14}C labeled benzene was chlorosulfonated with chlorosulfonic acid in carbon tetrachloride. The resulting benzenesulfonyl chloride was treated

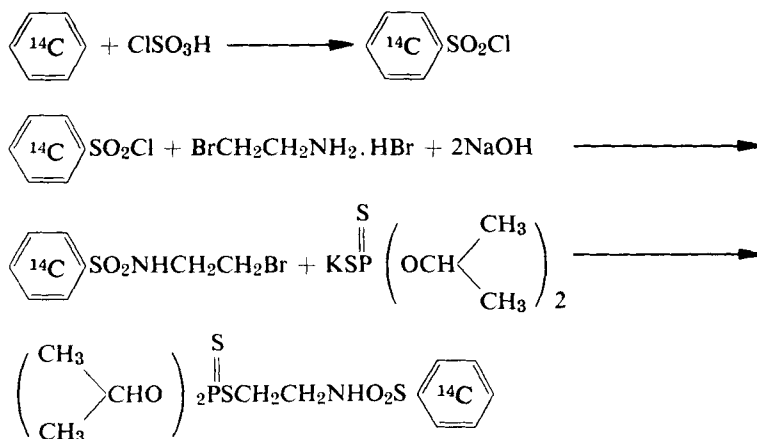


FIG. 1 — The reaction scheme

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