PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Process for the Isolation of Phosphorus-32 from Irradiated Sulphur

I, THE DIRECTOR IN CHARGE OF THE PRESIDENCY OF COMISION NACIONAL DE ENERGIA ATOMICA, a Government Department of the Argentine Republic, of 8250 Avenida del Libertador, Buenos Aires, Argentina, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for the isolation of phosphorus-32 from irradiated sulphur.

Phosphorus-32 is ordinarily obtained through the reaction $^{32}S(n,p)^{32}P$ by irradiating sulphur with neutrons.

The isolation and purification of phosphorus-32 is performed by various methods; some use the leaching of elementary irradiated sulphur with acids, particularly nitric acid, and extraction with mixtures of acetic acid and anhydride has also been proposed. In these methods the extraction step is followed by a purification process, which is essential to assure the chemical and radiochemical purity of the product.

Another method is based on the diffusion of phosphorus-32 during the transformation from the rhombic to the monoclinic variety of the irradiated sulphur, which occurs at 99.5°C. In practice, after adding a surface-active agent, the finely ground sulphur is boiled in water and the solution thus obtained is recovered.

In a further method which has been in commercial use for a few years, the phosphorus-32 is separated from sulphur by distilling the latter by heating in a nitrogen current and treating the phosphorus-32-containing residue with hydrochloric acid.

Among the more recently proposed separation methods, some use organic solvents to dissolve the irradiated sulphur and then the phosphorus-32 is recovered by means of acidic solutions.

A method has been proposed which is based on the solution of sulphur in hot toluene and the retention of phosphorus-32 in an alumina column; after elution with 0.5 M sodium hydroxide, the product thus obtained is freed of cations, using exchange resins.

All the above methods use the irradiation of elementary sulphur. A method has been described in which the irradiated compound is magnesium sulphate. Starting from this compound, after dissolution, the phosphorus-32 is oxidized to phosphate and precipitated with magnesium hydroxide by the addition of ammonia. The precipitate is then dissolved in hydrochloric acid, the traces of sulphate are eliminated by precipitation of barium sulphate, and the solution is passed through exchange resins for purification from the cations that might be present.

Some of the above mentioned methods require expensive apparatus needing delicate handling, others require a very careful preparation of the material to be irradiated, while most of them require complicated purifying steps to get useable materials.

The present invention provides a method for the isolation of phosphorus-32 from neutron-irradiated sulphur, the method comprising contacting a solution of the irradiated sulphur with a material comprising quartz or a silicon-containing glass or ceramic and then recovering phosphorus-32 that has been retained by the said material. The material is preferably in granules of from 0.3 to 1.0 mm. diameter, and phosphorus-32 can be recovered therefrom by elutriation with water or aqueous mineral acid, e.g. hydrochloric acid. The eluant preferably has an orthophosphate dissolved therein to aid the phosphorus recovery.

The mechanisms of retention of phosprous-32 in said material is not known with certainty, though it may involve an adsorption phenomenon.

The simplicity of the proposed method lends itself to the routine production of phosphorus-32 in hot cells with remote control.

The invention will now be further described, by way of illustration, with reference to the following specific example.

EXAMPLE

In the production of phosphorus-32, elemental sulphur (purified by triple sublimation) is used which has been neutron irradiated in the reactor RA-1. Reactor RA-1 is an annular geometry reactor, moderated with light water, having graphite reflectors. The fuel is uranium enriched with 19.17% ^{235}U.

The fuel elements are curved plates vertically and radially located in the annular neutron moderator.

The inner reflecting central cylinder is made of graphite and is provided with 8 orifices which are the irradiation facilities. Such location of said facilities lends itself to the use of the internal reflector as a neutron trap, as the thermal neutron flux is at a maximum therein (10^{11} neutrons/cm^2 sec.).

The control elements are three stainless steel plated cadmium plates which run vertically and tangentially to the reactor tank at the external reflector.

The external reflector consists in a graphite block frame shaping a structure of $1 \times 1 \times 1$ meter; there is a concrete biological shield surrounding this reflector.

The control is performed through the information received from 3 boron trifluoride proportional counters and 5 uncompensated ionization chambers which form the linear and logarithmic starting and running channels. Four games monitors are provided in addition to give an idea of the ambient radiation.

Rectified carbon disulphide is employed as the solvent for the irradiated sulphur. In spite of the drawbacks exhibited by this compound on account of its volatility and toxicity, it is chosen for its high solvent power for sulphur which permits operation with relatively small volumes of liquid and at room temperature.

The irradiated sulphur solution is passed through 14 mm diameter, 100 mm height columns filled with powdered soda-lime glass previously cleaned free of oils and greases and carefully dried. The particle diameter is from 0.3 to 1.0 mm. The column is washed with 50 ml carbon disulphide and the traces of solvent remaining in the column are removed by the passage therethrough of air for 10 minutes. In these conditions, more than 99% of the phosphorus-32 is retained in the column.

The elution is effected by the passage of 50 ml of three-times-distilled water at room temperature at the rate of 2 ml/min.; 90% of the phosphorus-32 retained in the column is eluted in this way.

The radioactive purity of the product obtained, as determined by electrophoresis and the beta-disintegration curve, is greater than 99.5%.

Chemical purity as determined by emission spectroscopy complies with the requirements of phosphorus-32 solutions for medical use.

An investigation of radio-chemical purity performed after sterilizing the preparation, shows that 99.5% of phosphorus-32 is in the form of orthophosphate.

The above described process has been selected on the basis of its optimum efficiency and the advantages of the product thereof, after performing tests in which the type of retaining material of the columns and the elution solutions were altered.

The retaining properties of powdered porcelain, quartz, and borosilicate glass have been studied. In these cases, the retention of phosphorus-32 was also very high, but did not reach the value obtained with soda-lime glass.

It has been shown in a study of the particle size of the material used for retention that the phosphorus retention is lower for particles larger than those used in the above Example.

Various elution solutions for phosphorus-32 were tested, some of which were acidic, with or without an orthophosphate carrier. The overall efficiency of the process was never higher than 90%. On this account, thrice-distilled water is preferably chosen as the eluant.

WHAT I CLAIM IS:—

1. A method for the isolation of phosphorus-32 from neutron-irradiated sulphur, the method comprising contacting a solution of the irradiated sulphur with a material comprising quartz or a silicon-containing glass or ceramic and then recovering phosphorus-32 that has been retained by the said material.

2. A method according to Claim 1, in which the irradiated sulphur is dissolved in carbon disulphide for contact with the material.

3. A method according to Claim 1 or 2, wherein the material is boro-silicate glass.

4. A method according to Claim 1 or 2, wherein the material is soda-lime glass.

5. A method according to Claim 1 or 2, wherein the material is porcelain.

6. A method according to any of Claims 1 to 5, wherein the material is in the form of particles having a size of from 0.3 to 1.0 mm diameter.

7. A method according to any of Claims 1 to 6 including the step of passing air through the material to eliminate solvent therefrom before recovery of the phosphorus-32.

8. A method according to any of Claims 1 to 7, wherein phosphorus-32 retained by the material is recovered by treatment with a mineral acid in aqueous solution.

9. A method according to Claim 8, wherein the mineral acid is hydrochloric acid.

10. A method according to any of Claims 1 to 7, wherein phosphorus-32 retained by the material is recovered by treatment with water.

11. A method according to Claim 8, 9 or 10, wherein the acid or water has dissolved therein a metal orthophosphate to aid in recovery of the phosphorus-32.

12. A method according to any of Claims 1 to 11, wherein the sulphur has been purified by triple sublimation prior to irradiation.

13. A method for the isolation of phosphorus-32 substantially as hereinbefore described in the Example.

14. Phosphorus-32 prepared by a method according to any of Claims 1 to 13.

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