

Acknowledgment.—This research has been carried out under Contract AT(45-1)-244 between the U. S. Atomic Energy Commission and Oregon State College. It has also derived support in part from a

grant-in-aid from General Research funds administered by the Graduate Council of Oregon State College.

CORVALLIS, OREGON

RECEIVED JULY 18, 1951

[CONTRIBUTION FROM THE CANCER RESEARCH LABORATORY, MEHARRY MEDICAL COLLEGE]

Separation of the Radioisotopes of Silver and Palladium

BY GEORGE ROUSER¹ AND P. F. HAHN

A rapid quantitative method for the separation of small amounts of the isotope Ag^{111} from large quantities of palladium is described. Isotopes other than Pd^{111} are reported in the palladium fraction.

Pure palladium dissolves sparingly in concentrated nitric acid even after prolonged heating unless the material is finely powdered. Although a mixture of concentrated nitric and sulfuric acids will dissolve palladium slowly, a more rapid solution is preferable. The use of either of these methods may lead to the production of refractory palladium oxides if heating is not carefully controlled. The method to be described using aqua regia as solvent has been used with success^{2,3} and is thus reported here in more detail.

The palladium was received as a 1-g. sheet after neutron bombardment in the Oak Ridge pile. Carrier silver nitrate (5–25 mg.) was added to a test-tube and allowed to coat the sides. The 1-g. palladium sample, containing an estimated 100 millicuries, was then added and treated with aqua regia with gentle heating for 1–2 minutes. Failure to add carrier before dissolving the sample resulted in varying losses depending upon the amount of carrier added (15% when 25 mg. of carrier was added). The acid suspension was then treated with concentrated ammonia and warmed until all of the material was in solution. The silver was reduced to the metal with an aqueous solution of ascorbic acid. The metallic silver was separated by centrifugation, washed with water and dissolved in nitric acid. After evaporation of the nitric acid solution to dryness, silver nitrate

was dissolved in water. Complete removal of chloride and occluded palladium can be ensured by adding ammonia and reducing again with ascorbic acid, although this is generally not necessary.

The procedure up to the evaporation of the nitric acid solution requires as little as 30 minutes. The quantitative nature of the procedure was demonstrated by the failure to recover any appreciable amount of radio-silver after repeating the procedure a second time on the same palladium sample.

Attempts to separate the silver as the chloride resulted in lower yields and increased the operating time because of difficulties involved in removing the chloride. The material obtained as a chloride precipitate was also contaminated with palladium to a greater extent, as shown by following the decay curve of the sample.

When the radioactive decay of the sheets was followed with a Lauritsen electroscope, the presence of material with an apparent half-life of about 17 hours finally decaying to a stable 21-hour period was found. This apparent half-life of 17 hours has been observed by Seiler⁴ to be due to the simultaneous decay of $\text{Pd}^{107,109}$ (half-life 13 hours) and Pd^{112} (half-life 21 hours). The final 21-hour period results from the decay of Pd^{112} after complete decay of $\text{Pd}^{107,109}$. Two days after removal from the pile 95% of the radioactivity was present in the palladium fraction. Since Pd^{111} (half-life 26 minutes), parent of Ag^{111} (half-life 7.5 days), had completely decayed at this time the activity was due to the two other palladium isotopes mentioned. Both of the latter isotopes of palladium decay to short lived silver isotopes and it is necessary to allow the silver fraction to stand overnight in order for them to decay and leave a pure sample of Ag^{111} .

(1) Atomic Energy Fellow in the Biological Sciences. Present address: Department of Biochemistry, University of Rochester School of Medicine and Dentistry, Rochester, New York.

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RECEIVED AUGUST 22, 1951

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