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CHEMISTRY IN THE ELECTROMAGNETIC SEPARATION OF PLATINUM ISOTOPES

W. C. Davis
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CHEMISTRY IN THE ELECTROMAGNETIC SEPARATION OF PLATINUM ISOTOPES

W. C. Davis and C. W. Sheridan

May 17, 1955

JUN 9 1955

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ABSTRACT

The chemistry of platinum is reviewed in relation to its application to the preparation of charge materials, the chemical refinement of the electromagnetically separated isotopes, and the recovery of residues.

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INTRODUCTION

The six known isotopes of platinum (190, 192, 194, 195, 196 and 198) have been separated on a production basis using the Oak Ridge National Laboratory production-scale mass spectrometer.[1] Material was also collected in the 200 mass position in order to search for an additional isotope. In the absence of suitable stable compounds, the metal as sponge was used to supply ions in the machine. The isotopes, which were collected in graphite receivers, were subsequently recovered, refined and made available for research.

CHARGE MATERIAL

All the charge material for electromagnetic separator operations, with the exception of one sample of platinum dicarbonyl dichloride,[2] $Pt(CO)_2Cl_2$, consisted of 10-gram samples of platinum sponge.[3] During the course of the

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[1]Bell, W. A., Jr., Love, L. O., and Normand, C. E., ORNL-1767.

[2]Blanchard, A. A., Chem. Rev., 21, 31(1937).

[3]All the platinum was acquired from the American Platinum Works, Newark, New Jersey.

production run there were some one hundred and thirty (130) such samples used. The run started with 500 grams of platinum sponge in the charge inventory and 200 additional grams of platinum powder was added midway during the run. The powder was found unsuitable as such, however, and was converted to platinum sponge by dissolving the metal in aqua regia and precipitating the complex ammonium salt, ammonium chloroplatinate, which decomposes to sponge at 600°C.

The platinum compound, platinum dicarbonyl dichloride, was prepared by passing carbon monoxide and chlorine over platinum sponge in a glass tube at 300°C. The compound was transferred from the glass tube directly into a charge container and immediately sealed. At no time during the trial run were platinum ions noted on the electrical meters. Upon termination of the run, the charge bottle was opened and a thin layer of platinum metal was found on the inside. Since all of the known platinum compounds are thermally unstable, no other compounds were synthesized.

RECYCLE

While the collection was still in progress, the graphite charge wedges, which contained the greater portion of the unused charge, were being processed. This included igniting the graphite in quartz under oxygen at 800°C, dissolving the residue in aqua regia, and then precipitating and igniting ammonium chloroplatinate. In all, 600 of the original 700 grams was recycled and reused as charge material in order to supply the total of 1300 grams which was necessary for the collection.

Upon completion of the separation all of the exposed metallic parts of the machine were washed and scrubbed with dilute nitric acid. These solutions, along with all the contaminated graphite parts, were processed

to recover the uncollected platinum. The solutions were filtered on folded S and S papers, and the filtrates were tested for platinum. Since platinum is not soluble in any concentration of nitric acid, it was believed that the solutions would be free of dissolved platinum. Several tests proved this true and all filtrates were discarded. The papers, containing the undissolved metal, were ignited along with the graphite parts and the platinum was recovered by methods previously described. Most of the original 700 grams was recovered and is available for future production runs.

ISOTOPE REFINEMENT

The refinement procedure which was used followed conventional analytical methods[4,5,6,7] and included three main steps: 1) ignition of the graphite receivers; 2) solution of the resulting ash; 3) selective and quantitative precipitation of platinum by the use of alcoholic ammonium chloride.

Prior to ignition, each of the receivers was cleaned thoroughly on the outside to prevent contamination of the isotope from normal and/or adjacent isotopic platinum which was deposited there during the separation. The slit through which the ions had entered the receiver was covered with 1/2-inch paper masking tape, and the surfaces of the graphite were rubbed with emery cloth until there was no evidence of a metallic deposit. The tape was removed,

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[4] Hillebrand, W. F., and Lundell, G. E. F., Applied Inorganic Analysis, pp 289, 290, New York, John Wiley and Sons, 1929.

[5] Treadwell, F. P., and Hall, W. T., Analytical Chemistry, Vol. II, pp 137, 145, New York, John Wiley and Sons, 1942.

[6] Schoeller, W. R., and Powell, A. R., The Analysis of Minerals and Ores of The Rarer Elements, pp 254, 280, London, C. Griffin and Co., 1940.

[7] Blackmere, A. P., Marks, M. A., Barefoot, R. R., and Beamish, F. E., Anal. Chem., 24, 1815(1952).

and the receiver was crushed in a Pyrex dish under a double layer of Kodapak.[8] A semi-circular quartz boat, 8" x 2" x 1", after being filled with pieces of the graphite, was placed in a quartz tube and ignited at 650°C in a stream of oxygen. The ignition was continued until all carbonaceous matter had been oxidized.

The ash, containing the platinum isotope, was dissolved in aqua regia and evaporated to incipient crystallization in the presence of excess hydrochloric acid. The resulting salts were alternately moistened and dried with 12M hydrochloric acid to remove any residual nitrate, and the residue was finally dissolved in 100 ml. of 1M hydrochloric acid with gentle warming. The solution was filtered through a 7-cm. No. 42 Whatman filter paper to remove any possible siliceous material, the paper was washed carefully with water, and the filtrate was evaporated slowly to a volume of 100 ml. After the solution had cooled, an excess of solid ammonium chloride was introduced, and an amount of 95% ethanol equal in volume to that of the solution was added. After standing for 24 hours, yellow ammonium chloroplatinate was filtered on an appropriately sized No. 42 Whatman paper. The precipitate was washed on the paper with 20% ammonium chloride solution, and then dried in porcelain and ignited carefully by heating to 800°C in a muffle furnace. The resulting platinum sponge was then placed in a quartz tube and ignited to constant weight at 600°C in hydrogen.

The individual lots of isotopes and the recovered charge material were analyzed spectrographically for approximately 35 elements, including the common elements and the platinum group. The purification procedure was repeated

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[8]Cellulose acetate sheets made by Eastman Kodak Company.

in only one case where analysis indicated excessive impurities. The values reported are semiquantitative, and the lower limits of the spectrographic method for most elements were of the order of 0.01%. The production data are tabulated below.

TABLE I

Isotope	Lot No.	Weight (g.)	Impurities Found (%)								
			Ba	Cu	Fe	Mg	Mn	Ni	Pd	Si	Ti
190	GH 776(a)	0.3446		<0.01		0.01	<0.01			0.01	
192	GH 777(a)	0.2789		0.02		0.005				<0.02	
192	GH 777(b)	0.9112				0.02					
194	GH 778(a)	12.0060			0.05						
195	GH 779(a)	3.2157				None Detected					
196	GH 780(a)	6.9895				None Detected					
198	GH 781(a)	0.4977		0.01	0.02		0.002			0.05	0.02
198	GH 781(b)	1.0496				None Detected					
200	GH 782(a)	0.2798	0.02	0.02	<0.02	0.002				<0.02	
Recovered Charge				0.2	0.05			<0.05T	1.0		

ACKNOWLEDGEMENTS

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