SHORTER COMMUNICATIONS

NATIVE PALLADIUM FROM COLOMBIA

A. D. WESTLAND

Department of Chemistry, University of Ottawa, Ottawa

F. E. BEAMISH

Department of Chemistry, University of Toronto, Toronto

A quantitive analysis of native palladium has not been recorded in the literature. Even had such an analysis been made, it is very likely that it would have been of doubtful accuracy, for no proved method of analysis was available until recently. The authors have developed a scheme for the analysis of small grains of the platinum group minerals and have used it to investigate iridosmine and native osmium (Westland & Beamish, 1957, 1958*a*, 1958*b*). In the course of that work, a sample of native palladium was encountered. A total analysis of this mineral has now been carried out, the details of which are reported in the present communication.

A mineral grain weighing 8 mg., for which the locality "Colombia" was given, was the subject of this investigation. An arc spectrogram taken in an Hilger medium quartz spectrograph revealed the presence of lead, tin, gold, silver, iron, copper, and nickel in addition to platinum group metals but lines other than the "raies ultimes" were afforded by only the first two of these. Provision had therefore to be made in the analysis for the estimation of lead and tin.

The analysis was carried out on two separate samples: one for the minor platinum group constituents and one for palladium, tin, and lead. The first followed the procedure used previously (Westland & Beamish, *loc. cit.*); the second was treated by a procedure, the description of which follows.

In order to ascertain the reliability of microgravimetric estimations of tin and lead, alloys of these two metals were prepared by vacuum fusion. As little as 0.0003 mg. of tin were recovered from one of these by the nitric acid method (Kolthoff & Sandell, 1952). Taken: 0.00029 mg.; recovered, 0.00036 mg. Recovery of the lead as the molybdate (Furman, 1939) was quantitative for milligram amounts. Palladium was determined photometrically with potassium iodide (Fraser & Beamish, 1954).

Procedure for Base Metals and Palladium

Tin was separated as stannic acid upon heating a nitric acid solution of the constituents. Palladium was extracted from the solution and determined as described by Fraser & Beamish (*loc. cit.*). After destruction of organic matter in the aqueous phase with fuming nitric acid and 30% hydrogen peroxide, lead was precipitated with ammonium molybdate and determined. Table 1 shows the recoveries of palladium and lead from synthetic solutions.

TABLE 1. RECOVERY OF PALLADIUM AND LEAD FROM SYNTHETIC SOLUTIONS

No.	Weight of Pd taken (mg.)	Weight of Pd recovered (mg.)	Weight of Pb taken (mg.)	Weight of Pb recovered (mg.)
1	1.00	0.92	5.00	5.04
2	1.00	1.00	5.00	5.04
3	1.00	1.00	5.00	5.02

Analysis of the Mineral

A photomicrograph of the mineral grain is shown in Fig. 1. Siliceous material which was adhering to the metal was removed by leaching with hot hydrofluoric acid. Analysis of the mineral commenced by breaking it into several pieces. One of these weighing 1.97 mg. was corroded by high temperature chlorination (Westland & Beamish, 1958*b*) and analysed for precious metals. A second fragment weighing 3.91 mg. was opened up by treatment with aqua regia as it was only slowly attacked by hot concentrated nitric acid. After expelling oxides of nitrogen by repeated evaporation with concentrated hydrochloric acid, the constituents were separated and determined. The composition found is given in Table 2.

The relatively large concentration of lead is in distinct contrast to native platinum and iridosmine in which iron is generally the principal

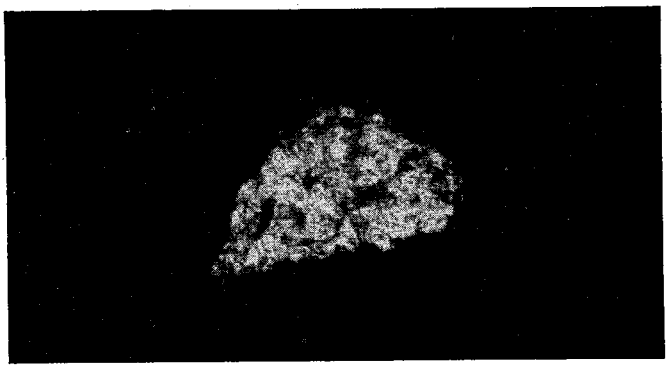

FIG. 1. Native palladium, Colombia, 15×.

TABLE 2. COMPOSITION OF THE MINERAL PALLADIUM

Element	Per cent
Palladium	86.2
Platinum	1.6
Rhodium	3.0
Iridium	0.2
Ruthenium	0.2
Osmium	0.7
Lead	8.1
Insoluble residue (stannic acid?)	0.5
Total	100.5

base metal constituent. This is probably associated with a large solid solubility of lead in palladium but a small solubility in platinum. It seems to be at least 10 per cent in the former (Ruer, 1907) but is believed to be small in the latter case (Doerinckel, 1907).

The lattice constant as determined by x-ray diffraction using Cu-$K\alpha$ radiation was $a = 3.91$ Å. This compares fairly closely with $a = 3.88$ Å for pure palladium metal despite the significant percentage of lead present. Like previously examined specimens of native palladium, the mineral possessed a hardness of 4–5.

ACKNOWLEDGMENT

The authors wish to thank Dr. C. Frondel of the Harvard Mineralogical Museum for providing the specimen.

REFERENCES

DOERINCKEL (1907): Metallographische Mitteilungen aus dem Institut für anorganische Chemie der Universität Göttingen XLVII. Über einige Platinlegierungen, *Z. anorg. u. allgem. Chem.*, **54**, 333.

FRASER, J. G., BEAMISH, F. E., & MCBRYDE, W. A. E. (1954): Separation of palladium from lead and colorimetric determination of palladium with potassium iodide, *Analytical Chemistry*, **26**, 495.

FURMAN, N. H. (1939): *Scott's Standard Methods of Chemical Analysis*, 5th Ed., 506, D. Van Nostrand, New York.

KOLTHOFF, I. M., & SANDELL, E. B. (1952): *Textbook of Quantitative Analysis*, 3rd Ed., 667, MacMillan, New York.

RUER, R. (1907): Über die Legierungen des Palladiums mit Blei, *Z. anorg. u. allgem. Chem.*, **52**, 345.

WESTLAND, A. D., & BEAMISH, F. E. (1957): The separation and determination of the platinum metals on a microscale, *Mikrochimica Acta*, 1957, 625.

——— (1958*a*): The chemical analysis of iridosmines and other platinum-metal minerals, *Am. Mineral*, **43**, 503.

——— (1958*b*): Use of chlorine in the attack of noble metals, *Analytical Chemistry*, **30**, 414.