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ISOTOPIC ABUNDANCES OF PALLADIUM

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MASS SPECTROMETRY LABORATORY
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ABSTRACT

The recently developed mass spectrometer high temperature source oven has been applied to the determination of the isotopic abundances of palladium as part of a program for the assaying of the separated isotopes of the platinum metals. Discrepancies with previously published data have been found and the newly determined isotopic abundances of natural palladium are reported.

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ISOTOPIC ABUNDANCES OF PALLADIUM*

John R. Sites, George Consolazio,** Russell Baldock

For some time we have concerned ourselves with developing a high temperature ion source oven for use in a mass spectrometer so that isotopic analyses could be performed on materials in either the elemental form, or on the more stable compounds as found in the Stable Isotope Research and Production Division's inventory of separated isotopes. The recent successful separation of many of the isotopes of the platinum metals group by means of the calutron has been reported by Bell, Love, Normand, and Prater.[1]

In preparation for performing the isotopic analyses on material collected during the separation of the isotopes of an element, the natural abundances have been redetermined. We have found some variations from the measurements on palladium reported by Sampson and Bleakney,[2] and compiled by Bainbridge and Nier.[3] In an earlier report,[4] Dempster reported the dis-

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* Presented at the American Physical Society meeting at Albuquerque, New Mexico, September 4, 1953.

** Watertown Arsenal, Watertown, Massachusetts.

1. Bell, W. A., Jr., L. O. Love, C. E. Normand, and W. K. Prater, "Electromagnetic Separation of Pd, Pt, and Ru isotopes." Bull. Amer. Phys. Soc., 28, p 24 (1953).

2. Sampson, M. B., and W. Bleakney, Phys. Rev. 50, p 732 (1936).

3. Bainbridge, K. T., and A. O. Nier, "Relative Isotopic Abundances of the Elements." Preliminary Report No. 9, National Research Council.

4. Dempster, A. J., Nature 136, p 65 (1935).

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covery of the six isotopes of palladium but gave only an estimate of the abundances. No other investigations of the isotopes of palladium have been found in the open literature.

The mass spectrometer used in this investigation has been previously described.[5] Briefly, it is an 8 inch radius, 60° sector type instrument of all stainless steel construction. The ion current is measured using a 10^{10} ohm input resistor to a vibrating reed electrometer which feeds into a multiple range Leeds and Northrup Speedomax recorder.

The ion source oven used in this investigation was developed by Mr. J. R. Walton of our group, and has likewise been previously described.[6] The essential features of the oven are shown in Figure 1. The center opening is 1/8 inch long and 0.020 inch in diameter. Two mil platinum, or tantalum, sheet stock is cut in a thin strip and is formed in a die, first U-shaped, and then finished around a 0.020 inch mandrel. Pins of platinum, or tantalum, are inserted and spot welded to the legs. The legs serve both for mechanical support and as electrical conductors for self-heating. The oven is mounted on a subassembly which in turn is attached to the ionization chamber of a permanently mounted source. The open side of the oven is parallel to and directed towards the ionizing electron beam from a distance of 1/16 inch.

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5. Baldock, Russell, and J. R. Sites, "A Versatile All-Metal 60° Mass Spectrometer," Y-755.

6. Walton, J. R., "High Temperature Mass Spectrometer Oven." ORNL-1473.

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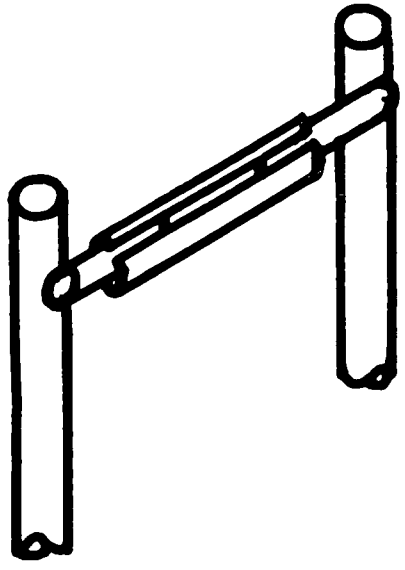

FIGURE 1. HIGH TEMPERATURE FILAMENT SOURCE OVEN

For each run, about a half a milligram of natural palladium metal powder was placed in a tantalum filament source oven. At 1350°C the first palladium ions were observed. At 1560°C, the melting point of palladium, the ion beams were quite intense; and mass spectrometer operation and recording continued smoothly up to 2200°C, near the boiling point. No apparent alloying took place between the palladium and the tantalum oven. The filament source oven temperature was held at various settings above the melting point for over two hours, and near 2200°C for twenty-five minutes with no indication of sample exhaustion. Altogether sixty-four groups of ion peaks were recorded and the relative abundances were calculated for each group. The 95% confidence interval was determined for each series, and for the grand average. Figure 2 shows a bar graph of the isotopic abundances of the palladium isotopes. The cross bars indicate the abundances reported by Sampson and Bleakney.[2] Figure 3 is a photograph of typical scans of natural palladium, and of palladium enriched in the mass 108 isotope. The small ion peak at mass 103 in the enriched material scan may be due to contamination by a little rhodium. Since there is no mass 103.5 ion peak present it could not be due to doubly charged lead. In Table I are listed the data of Sampson and Bleakney, the new values reported here, and the isotopic abundances of the palladium collected in the mass 108 position. Sampson and Bleakney stated that they believed that their results were good to 1%.

Chemically determined values for the molecular weight of palladium

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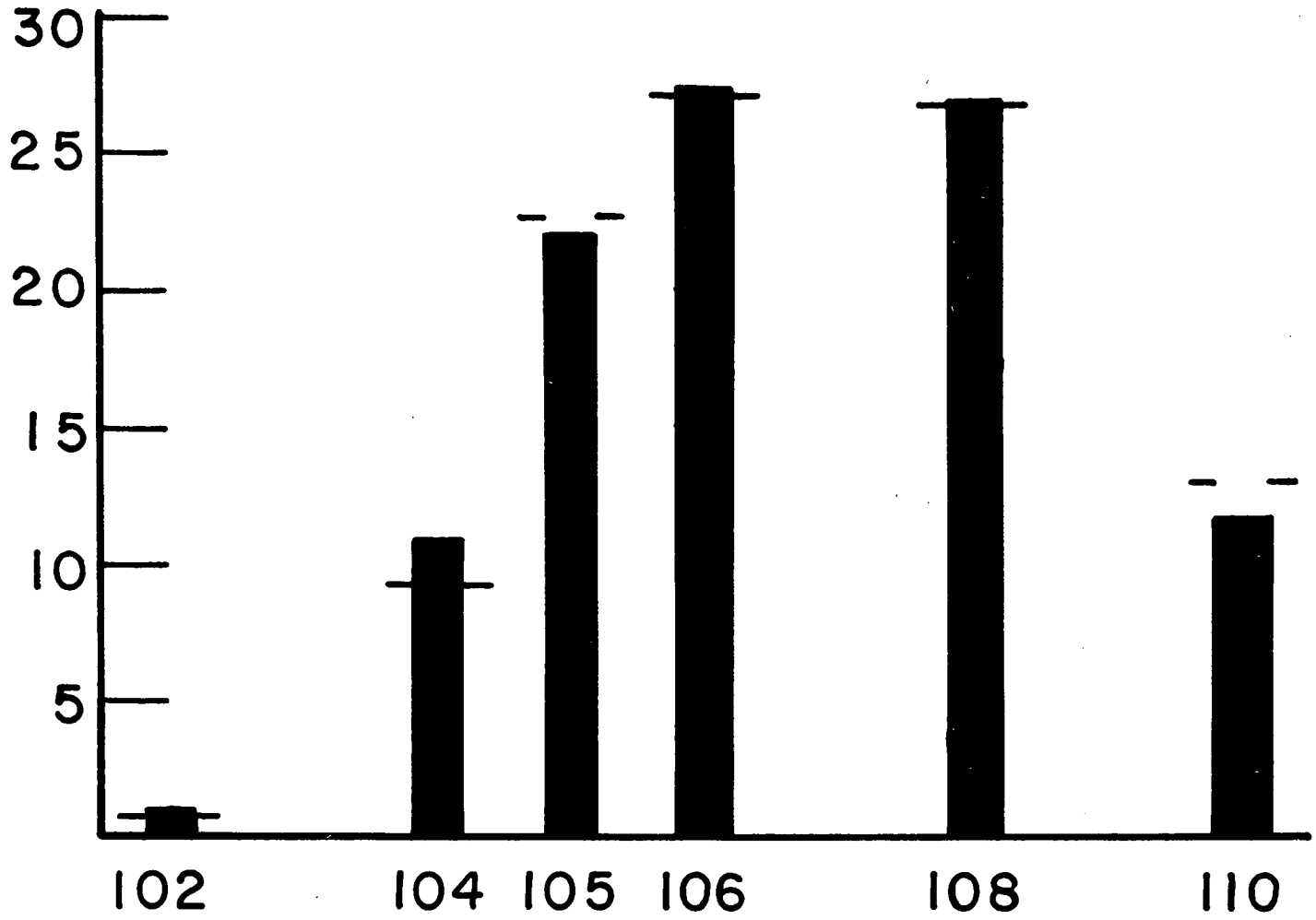

FIGURE 2. GRAPH OF ISOTOPIC ABUNDANCES OF NATURAL PALLADIUM

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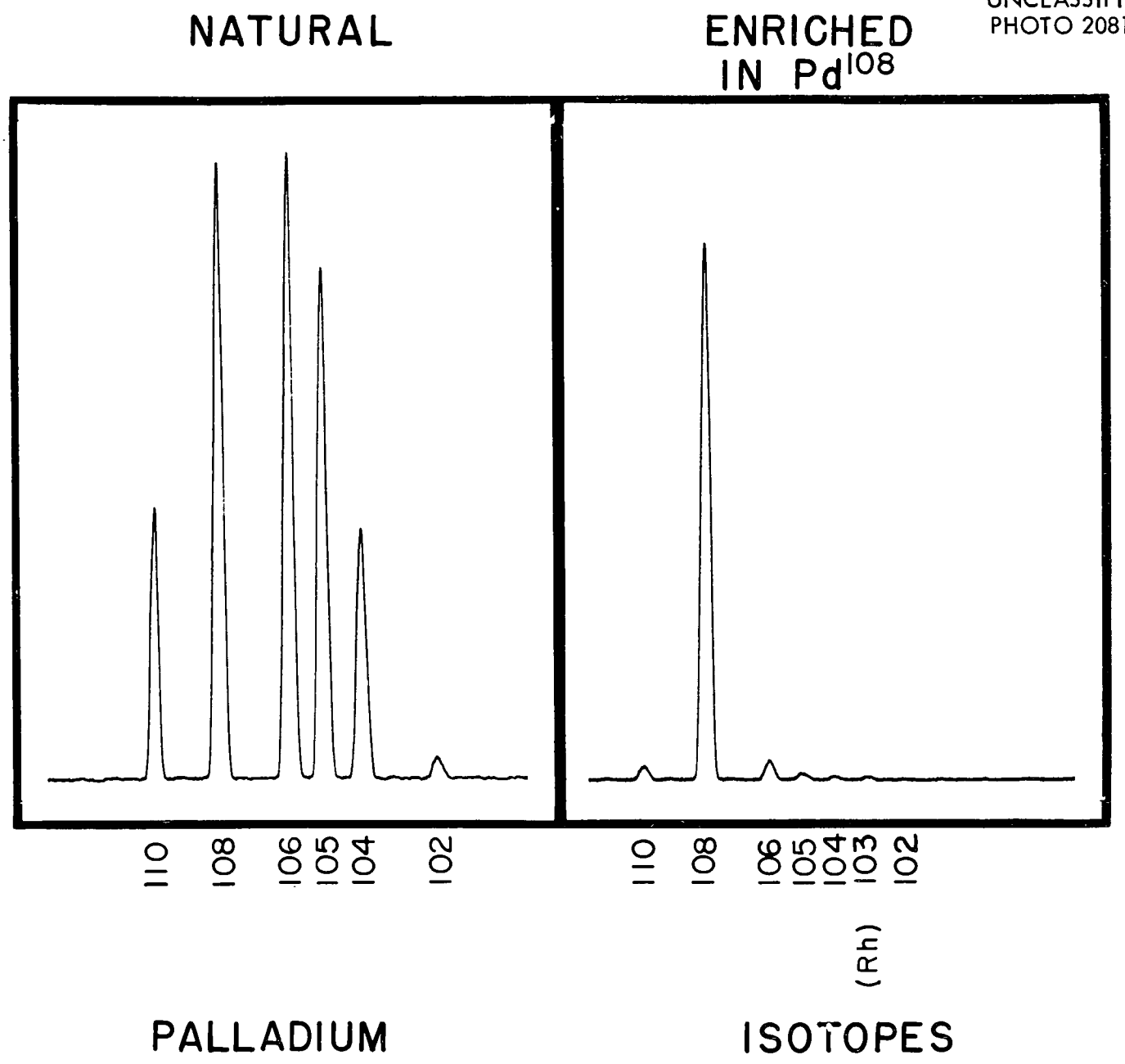

FIGURE 3. TYPICAL SCANS OF NATURAL AND ENRICHED PALLADIUM

TABLE I

Palladium Isotopic Abundance Data

Mass	Sampson and Bleakney (In %)	This Report (In %)	Enriched (In %)
102	0.8	0.96 ± 0.01	0.03 ± 0.01
104	9.3	10.97 ± 0.02	0.20 ± 0.05
105	22.6	22.23 ± 0.03	0.63 ± 0.04
106	27.2	27.33 ± 0.03	2.84 ± 0.04
108	26.8	26.71 ± 0.03	94.19 ± 0.11
110	13.5	11.81 ± 0.03	2.08 ± 0.04

have been reported as ranging from 106.4 to 106.9.[7, 8, 9] Sampson and Bleakney reported a value of 106.6. Using the mass defects reported by Halsted[10] and the new isotopic abundance values reported here, a molecular weight of 106.5 ± 0.1 is obtained.

The standard precautions to be taken in measuring isotopic abundances as described by Bainbridge and Nier[3] have been taken into account. The ion detector was carefully checked for linearity. Magnetic scanning was used throughout, with an electron-collimating magnet being required to attain sufficient ion beam intensity. The natural palladium used was obtained from two vendors: Secon Metals Corporation, 228 E. 45th Street, New York 17, New York, and Goldsmith Brothers, 111 North Wabash Avenue, Chicago, Illinois. No isotopic differences could be detected in the metal from the two sources. As previously indicated, no variation of isotopic abundances was found over a wide spread in temperature. In the natural palladium runs, no interferring background, such as doubly charged mercury or lead ions, could be detected.

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7. Baxter, G. P., O. Honigschmid, and P. LeBeau, J. Amer. Chem. Soc. 53, p 541 (1936).

8. Kemmerer, G. I., J. Amer. Chem. Soc. 30, p 1701 (1908).

9. Shinn, O. L., J. Amer. Chem. Soc. 34, p 1448 (1912).

10. Halsted, R. E., Phys. Rev. 88, 666 (1952).