in a vacuum, b. p. 156–157° (uncorr.) at 10 mm. (uncorr.). The yields are above 50% but vary considerably with the time of heating. Bis-(β-phenylaminoethyl)-amine is also formed but in smaller amounts.

N-Monophenylpiperazine is insoluble in water and soluble in all common organic solvents, d_4^{20} 1.0621; *neut. eq.* calcd. 162.1; found, 165.0. The hydrochloride of N-monophenylpiperazine was used for analysis for nitrogen. Calcd.: N, 14.11. Found: N, 14.01.

Other mono N-substituted amines have been prepared by this same method and their properties and derivatives will form the basis of a forthcoming publication.

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RECEIVED AUGUST 20, 1934

THE ISOLATION OF 0.1 GRAM OF THE OXIDE OF ELEMENT 91 (PROTACTINIUM)

Sir:

Using a process described previously [A. V. Grosse, *Ber.*, **61,** 233 (1928); THIS JOURNAL, **52,** 1742 (1930)] we have recently been able to work up large quantities of radium residues from Joachimsthal, Czechoslovakia. Thanks to the courtesy of the Lindsay Light Company, a small plant for the treatment of these residues was rigged up at their factory in West Chicago.

The technical process of concentration consists briefly: (1) an extraction of the residues, consisting principally of SiO_2, Fe_2O_3 and PbO with hydrochloric acid; (2) a melt of the silica residue, containing the protactinium, with sodium hydroxide, and (3) separation of the protactinium with zirconium phosphate from the mixture of basic oxides remaining after the melt is leached.

The starting material contains on the average 0.3 g. of protactinium per metric ton or a concentration of 1 part in 3 millions. The plant product contains the element in a concentration of 1 part in 5000, the yield being about 75%.

The plant product is further concentrated in the laboratory by a series of fractional crystallizations of zirconium oxychloride and zirconium phosphate precipitations. At a concentration of about 10% by weight of protactinium, most of the zirconium can be separated by sublimation of the chlorides; protactinium is then best precipitated directly by means of hydrogen peroxide. The purity of the oxide obtained by this method has been proved by x-ray analysis [H. Beuthe and A. V. Grosse, *Z. Physik,* **61,** 170 (1930)].

A detailed description of the technical and laboratory process will be given later.

Up to the present time, the plant product from about 1 ton of residues was worked up and 0.1 g. of protactinium oxide (Pa_2O_5) isolated in the pure state. Larger quantities will be isolated in the near future.

This work required by its very nature large financial expenditures and it has been made possible only by a generous grant of Mr. Hiram J. Halle of New York City, to whom we express our sincere gratitude. We are much indebted to Mr. A. C. Ratchesky, U. S. Minister to Czechoslovakia, and to Mr. Frank Novotny, U. S. Legation in Prague, for their efficient support in obtaining the raw material from the Czechoslovakian Government, and to Prof. Julius Stieglitz for his continuous help and advice.

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RECEIVED SEPTEMBER 10, 1934

METALLIC ELEMENT 91

Sir:

Isolation from radium residues of about 0.1 g. of protactinium oxide has been accomplished recently [A. V. Grosse and M. S. Agruss, THIS JOURNAL, **56,** 2200 (1934)]. The pure oxide (Pa_2O_5) was converted into the metal by two methods: (1) By bombarding the oxide, on a copper target, with a stream of electrons in a high vacuum. The use of 35,000-volt electrons for a few hours at a current strength of 5–10 milliamperes, splits the oxide into oxygen and the metal, the latter remaining as a shiny, partly sintered, metallic mass, stable in air. (2) By transforming it into a halide (Cl, Br, I) and then cracking it in a high vacuum (10^{-6} to 10^{-5} mm.) on an electrically heated tungsten filament, according to the reaction $2PaX_5 \longrightarrow 2Pa + 5X_2\uparrow$, the general method first introduced by Langmuir [I. Langmuir, THIS JOURNAL, **37,** 1139 (1915)]. The protactinium formed a shiny gray white, partly molten, deposit on the filament. The metal does not oxidize in air, in an expected contrast to metallic radium [Marie Curie and A. Debierne, *Compt. rend.*, **151,** 523 (1910)], and retains its luster for quite a while.