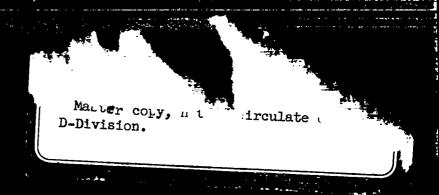
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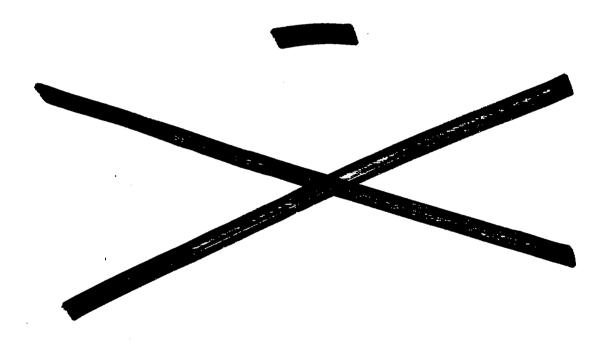
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THE PREPARATION OF PLUTONIUM METAL BY THE REDUCTION OF THE OXIDE WITH GRAPHITE

WORK DONE BY:

D. Lipkin

M. L. Perlman

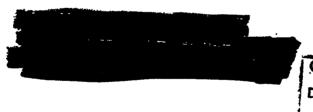
S. I. Weissman

REPORT WRITTEN BY:

D. Lipkin

M. L. Perlman

S. I. Weissman



CLASSIFICATION CANCELLED

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For The Atomic Energy Commission

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Chief, Declassification Branch ...



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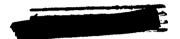


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ABSTRACT

Plutonium metal of high purity has been prepared by the evaporation of the metal from a mixture of oxide and graphite.





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THE PREPARATION OF PLUTONIUM METAL BY THE REDUCTION OF THE OXIDE WITH GRAPHITE

When a mixture of plutonium oxide and graphite is heated in vacuum, carbon monoxide corresponding in amount to the oxygen in the oxide is evolved. If the stoichiometrical amount of graphite is used, the following reaction takes place at 1200° to 1500°C:

$$20 = PuO_{Q} = Pu (vapor) + 200$$
 (1)

When an excess of graphite is present, the reaction which occurs at about the same temperature is

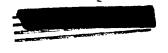
$$(2+x)C + PuO_2 = 2CO + PuC_x$$
 (2)

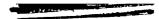
If the mixture is subsequently heated to 1900°C the carbide decomposes.

$$PuC_x = Pu (vapor) + xC$$
 (3)

The reactions were carried out in inductively heated crucibles of graphite, tantalum, or tungsten. The metal vapor was condensed on a thin tantalum "derby hat" held tightly onto a water-cooled copper finger.

The reductions with excess graphite, all on a 50 mg scale, were carried out in each of the three crucible materials. Because of its low vapor pressure tungsten seemed to be most suitable. In these experiments the graphite and oxide were ground together and heated first at 1200° to 1500°C until all the CO was pumped off (Equation 2); this reaction took about an hour. The temperature was then raised to 1900° to 2200°C in order to distill the metal out of the carbide. The yields of metal varied from 30-58 per cent; the experiments showed that these yields could be increased by increasing the duration and temperature of the distillation. A spectroscopic analysis of metal obtained by this method follows:

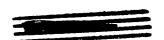




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		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
Lithium	Not detected	(<1 ppm)
Beryllium	n n	(<1 ppm)
Sodium	Blank	(< 5 ppm)
Magnesium	Ħ	(<5 ppm)
Aluminum	Not detected	(< 3 ppm)
Potassium	п п	(<15 ppm)
Calcium	Blank	(< 5 ppm)

The only crucible material used in the experiments with a stoichiometric amount of graphite was tantalum. The reactants were ground together and heated to 1200° to 1500°C. In this temperature range the CO and metal distill out together at a rate controllable by the temperature. This reaction was completed in about an hour. The yields were roughly quantitative, but there was always some back reaction caused by the hot CO impinging upon the deposited metal. Attempts were made to improve the quality of the product by using a reaction chamber designed to permit redistillation of the first product. The work was terminated during these attempts.



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