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Richard J. Roberts 12/1/52

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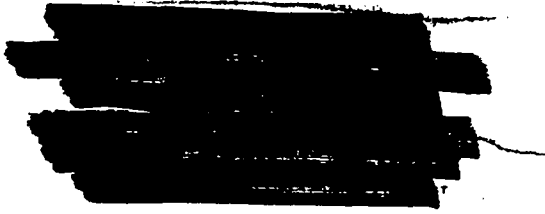
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THE ELECTROLYTIC PREPARATION OF THIN FILMS OF PLUTONIUM OXIDE

(Preliminary)

M. L. Miller

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Richard J. Roberts Oct. 10-21-46
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Thin films of plutonium oxide, up to 0.1 mg of metal per cm², have been prepared electrolytically on platinum. The method depends on the use of the plutonyl ion in alkaline solution. It is presumed that on reduction at the cathode, a thin layer of insoluble hydroxide is deposited. This is subsequently ignited to the oxide.

Method; The arrangement for electrolysis and the electrolyte solution are those described by M. Kahn (UCRL - 612). The solution consists of a KOH-K₂CO₃ mixture containing 1.78 g of KOH (Baker's analyzed) plus 4.0 g of K₂CO₃ per 200 cc of solution. To 15 cc of this is added 5 cc of water containing the plutonyl ion, as nitrate. Electrolysis is carried out with a rotating platinum anode at 3v and a current density of approximately 10 ma/cm². The course of the electrolysis is followed by removing small amounts (appr. 7 microliters) at hourly intervals for counting. When the count has dropped to less than one tenth of its initial value, or if it begins to rise, electrolysis is stopped and the plate washed and ignited. The electrolysis usually requires 2-3 hours. The amount of material on the plate must be determined by counting, since as much as 15-25% of extraneous material may be deposited. About 85% of the metal is recovered on the plate; the remainder probably being

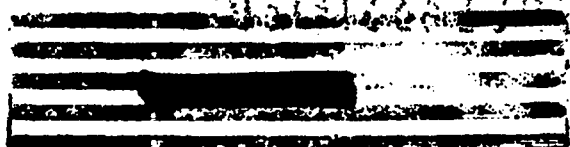


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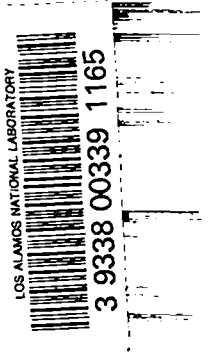
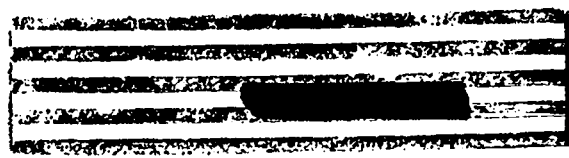
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in the form of an insoluble precipitate on the sides of the chimney.

Whether the carbonate serves any useful purpose or whether its omission or a change in its concentration would increase the efficiency of the electrolysis is one of the many factors yet to be investigated.

The foils so prepared show interference rings and are very similar in appearance to electrolytically prepared U_3O_8 foils.



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