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T. M. Mooney / J.D. Briggs 6-25-03

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M. T. Cain,
Engineering Department

HWIR-411 AEC CASE S-105-70

The following information is in response to your letter of December 18, 1953, to O. F. Hill concerning HWIR-411-AEC Case S-105-70.

1. Bulk density of plutonium trifluoride prepared by reaction between plutonium III oxalate or plutoniumIV oxalate and dichlorodifluoromethane has been measured and found to be 3.5 grams per cc. This is equivalent to 2.8 grams plutonium per cc. This may be compared with the bulk density of plutonium tetrafluoride prepared by present Hanford process methods, which varies between 1.6 and 2.2 grams per cc, the average value being 2.0 grams per cc. This is equivalent to 1.5 grams plutonium per cc.

2. Bulk density of plutonium trifluoride prepared in a two step reaction consisting of (a) low temperature (not over 400 C) decomposition of plutoniumIV oxalate, and (b) subsequent reaction between the resultant plutoniumIV oxide and dichlorodifluoromethane, averages 3.4 ± 0.3 grams per cc, or the equivalent 2.5 grams plutonium per cc. This may be compared with the bulk density of plutonium tetrafluoride stated in section 1.

3. As a specific example of the one step process, 1.35 grams plutonium were precipitated as plutoniumIV oxalate from plutonium nitrate (Hanford A.T.) solution. The oxalate slurry was washed with 0.1 M nitric acid - 0.1 M oxalic acid mixture, then water, and was decanted to leave a slurry containing about 30 percent by volume of water. The wet slurry was transferred to a platinum reaction boat. The slurry was dried in a stream of argon

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(about one-half lineal foot per minute) at 105 C, requiring about one hour. The chemically bound water was removed by continuing the argon flow and raising the temperature to 225 C for 1-1/2 hour. The argon was replaced with Freon-12 (flow rate about 1/3 lineal foot per minute) and the temperature raised to 425 C for two hours. The reaction system was cooled to below 100 C before exposure of the resultant plutonium trifluoride to air. Product plutonium trifluoride weighed 1.67 grams and analyzed 15.7 weight percent fluorine and 0.1 weight percent chlorine. Theoretical weight percent fluorine in plutonium trifluoride is 19.2 percent. Material balance of the analysis was 94.3 percent. Bulk density of final product was estimated to be 3.5 ± 0.3 grams per cc.

4. As a specific example of the two step process, 30.04 grams of plutonium were precipitated as plutonium IV oxalate from plutonium nitrate (Hanford S.T.) solution, washed free of nitrate ion, and decanted. The slurry of plutonium IV oxalate was transferred to platinum reaction boats with a slurry depth of approximately one-half inch, and dried three hours in a stream of air (1/2 lineal foot per minute) at 125 to 150 C. The oxalate cake was converted to reactive plutonium oxide by raising the temperature to 275 to 300 C for one hour, continuing the air stream. Care was taken that the heat of reaction did not elevate the cake temperature above 400 C. The gas stream was shifted from air to Freon-12 (dichlorodifluoromethane) at about 1/3 lineal foot per minute. The temperature was raised to 400 C for two hours. The resultant plutonium trifluoride was cooled to below 100 C before exposure to air. Product plutonium trifluoride weighed 98.9 grams and analyzed 61.0 ± 0.4 percent plutonium, 18.2 ± 0.5 percent fluorine, and 0.4 ± 0.3 percent chlorine. Bulk density of final product was 3.35 grams per cc.

5. Studies were also made to determine the usefulness of other chlorofluorinated hydrocarbons, including Freon-13, Freon-21, Freon-22, Freon-113, Freon-114, and carbon tetrafluoride. None appeared as practical as Freon-12.

The formal report on this work has finally been distributed. It is HW-30040, "Plutonium Trifluoride; Preparation by Reaction with Freon-12, and Bomb Reduction to Metal". The Chicago patent group was included on the distribution list. Other reports having reference to this work include HW-24150, pp. 41-46, HW-24139, pp. 31-32, and HW-25166, pp. 36-37.

Due to organizational changes which have occurred during the past year, any requests for further information should be directed to me for most expeditious answer.

W. E. Roake

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