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TECHNOLOGY - PLUTONIUM

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THERMAL DECOMPOSITION OF PLUTONIUM (IV) OXALATE AND HYDROFLUORINATION OF PLUTONIUM (IV) OXALATE AND OXIDE

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AUGUST 1, 1956

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Technology - Plutonium
(M-3679, 18th Ed.)

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OXALATE AND HYDROFLUORINATION OF
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August 1, 1956

HANFORD ATOMIC PRODUCTS OPERATION
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Work performed under Contract No. W-31-109-Eng-52 between
the Atomic Energy Commission and General Electric Company

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THERMAL DECOMPOSITION OF PLUTONIUM (IV) OXALATE AND
HYDROFLUORINATION OF PLUTONIUM (IV) OXALATE AND OXIDE

INTRODUCTION

Plutonium (IV) oxalate precipitation has been used as a purification step for plutonium and as an intermediate step in the preparation of plutonium tetrafluoride. Little study has been made of the decomposition path of plutonium (IV) oxalate when heated. Plutonium oxide produced by calcination of plutonium (IV) oxalate shows a marked difference in reactivity towards fluorination with hydrogen fluoride, depending on the calcination conditions. The work described in this report was done to determine the path of decomposition of plutonium (IV) oxalate and to determine the factors affecting the reactivity of the oxide with hydrogen fluoride.

SUMMARY AND CONCLUSIONS

Plutonium (IV) oxalate hexahydrate decomposes slowly with a decrease in oxalate content during storage at room temperature (21-25 C). Thermal decomposition in an inert atmosphere proceeds by the following steps: (1) loss of five waters of hydration at 80 C to form the monohydrate; (2) loss of the final water of hydration at 150-160 C; (3) decomposition to plutonium (III) oxalate at 160-200 C; (4) decomposition of plutonium (III) oxalate at 260-275 C; and (5) decomposition of intermediate compounds resulting in plutonium dioxide at 500 C. Decomposition in air follows the same path, but the presence of oxygen increases the rate and decreases the temperature required to initiate decomposition of the plutonium (III) oxalate. At times, exothermic reactions raise the cake temperature several hundred degrees. Heating with infra-red bulbs results in complete calcination of thin cakes (4 mm) of plutonium (IV) oxalate.

Plutonium (IV) oxalate can be fluorinated directly with anhydrous hydrogen fluoride-oxygen mixture to plutonium tetrafluoride. A final temperature of 500 C is necessary to avoid formation of the trifluoride. Optimum

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conditions for hydrofluorination of plutonium oxide are calcination of the oxalate to the oxide below 480 C and hydrofluorination at 500 C. Under these conditions, greater than 90 per cent conversion is obtained in one hour with 0.1 cm cakes. Calcination of the oxide at temperatures above 480 C results in much slower fluorination. The fluorination reaction proceeds in two phases; the very fast reaction with the surface of the oxide particles; and, second, the slower reaction as hydrogen fluoride diffuses into and reacts with the interiors of the particles.

EXPERIMENTAL

The weight loss of plutonium (IV) oxalate during storage was followed with a Chain-O-Matic balance. The proper dessicant was placed in the balance case, and all openings were sealed with tape. Thermal decomposition was studied in apparatus which allowed continuous weighing of the cakes with a Roller-Smith precision balance. The gas from the decomposition was either collected in gas sample bottles or passed through an absorption train. Gas analysis was made with Orsatt type apparatus or by weight gain of the components of the absorption train (Anhydrone, Ascarite, etc.). The oxalate to plutonium ratios were determined by metathesizing a weighed amount of powder with 4 M KOH, neutralizing with sulfuric acid, and filtering the plutonium hydroxide out. The solution was then acidified to 2 M H₂SO₄ and titrated with standard potassium permanganate. The plutonium hydroxide was dissolved in nitric acid and plutonium determined by radio-assay.

The hydrofluorination studies were carried out in apparatus which allowed continuous weighing of the sample during the reaction (Figure 1). The oxide or oxalate was placed in a solid bottomed platinum boat, 1 x 2 x 2 cm, for the deep cake experiments. The boats or trays were then placed in a suitable rack made of gold and platinum. A mixture of ten per cent oxygen - 90 per cent anhydrous hydrogen fluoride, with a flow rate of one liter per minute, was used as the fluorinating gas.

Particle sizes were determined by X-ray analysis by the Product Metallurgy Unit.

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Munsell color charts were used as standards for color determinations.

PROPERTIES OF PLUTONIUM COMPOUNDS

Plutonium (IV) Oxalate

Plutonium (IV) oxalate is precipitated from pure solutions in 2-4 M nitric acid as the hexahydrate $(\text{Pu}(\text{C}_2\text{O}_4)_2 \cdot 6\text{H}_2\text{O})$ (molecular weight 523). The cake densities vary from 0.4 to 0.7 g Pu/cc, with an average density of 0.6 g Pu/cc. The color varies from 2.5 Y 8/4 to 2.5 Y 7/10 on Munsell⁽¹⁾ color scale, with a normal color of 2.5 Y 7/6 (tan). The particle size is approximately five to seven microns. A free-flowing oxalate is obtained by precipitation from pure solutions and air drying. Too rapid precipitation or precipitation from impure solutions may produce fine clay-like thixotropic oxalates.

Plutonium Dioxide

Plutonium dioxide prepared from plutonium (IV) oxalate by thermal decomposition of the oxalate varies in color, depending upon the maximum temperature of calcination. A Munsell scale color of 7.5 Y 5.5/5 (olive green) at a calcination temperature of 300 C, and 7.5 Y 8/8 (yellow-green) at 620 C, have been observed. The bulk densities also vary with calcination temperature; typical densities of 1.89 g Pu/cc are obtained at 300 C and 2.6 g Pu/cc at 620 C. The particle size is approximately two to five microns.

Plutonium Trifluoride

Plutonium trifluoride obtained from hydrofluorination or freonation of plutonium (IV) oxalate is purple-blue (Munsell colors 7.5 PB 1/7 to 7.5 PB 8/2) with a bulk density of 1.5-2.8 g Pu/cc, normally 2.1 g Pu/cc.

Plutonium Tetrafluoride

Plutonium tetrafluoride obtained by hydrofluorination of plutonium(IV) oxalate or plutonium oxide is salmon-pink (Munsell colors 7.5 YR 8/4 to 10.0 YR 8/4), with a bulk density of 1.2-1.9 g Pu/cc, normally 1.6 g Pu/cc. The particle size is two to ten microns.

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DECOMPOSITION OF PLUTONIUM OXALATE

Decomposition During Storage At Room Temperature

In an extremely dry atmosphere, like that over magnesium perchlorate (in which the water in 210 liters of air is unweighable) plutonium oxalate hexahydrate is converted to the monohydrate (chocolate-brown color) in ten days. Storage of the hexahydrate over fused sodium hydroxide (0.002 mg water per liter of air) caused no change in color, although decomposition occurred. Figure 2 shows the per cent unreacted plutonium oxalate (considering the final product as plutonium dioxide) with time. Storage in a moist atmosphere resulted in a change of color from tan to olive-green. Slow decomposition of the oxalate occurs during storage in either a dry or a moist atmosphere, probably due to destruction of the oxalate by alpha bombardment. Figure 3 shows the oxalate content versus time.

Thermal Decomposition of Plutonium (IV) Oxalate in an Inert Atmosphere

In an inert atmosphere, plutonium (IV) oxalate hexahydrate is converted to the monohydrate at 80 C. The final water of hydration is slowly lost from 80 to 150 C, when the anhydrous oxalate is produced. The anhydrous plutonium (IV) oxalate decomposes rapidly at 160 to 200 C, and then more slowly until the plutonium (III) oxalate is produced at 250 C. Decomposition of plutonium (III) oxalate begins at 260 to 275 C, producing a mixture of unidentified compounds. These are completely converted to plutonium dioxide at 500 C. The monohydrate does not appear as a definite step in Figure 4, which shows the thermal decomposition of plutonium (IV) oxalate, because the temperature was raised too rapidly in this run. Figure 5 shows the oxalate to plutonium ratio during the decomposition.

Treatment of the intermediate products from the decomposition of plutonium (III) oxalate with 3 M H_2SO_4 resulted in the evolution of 0.04 moles of carbon dioxide and 0.01 moles of oxygen per mole of plutonium. Treatment with 4 M KOH caused the evolution of 0.01 moles of oxygen per mole of plutonium. These results indicate the presence of traces of carbonate and

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peroxide oxygen. Approximately one per cent elemental carbon and 0.6 moles of oxalate per mole of plutonium were also found. The presence of such a large amount of oxalate suggests the presence of plutonium oxy-oxalate ($\text{Pu}_2\text{O}_2\text{C}_2\text{O}_4$) or some similar compound. Analysis of the gases evolved during the decomposition of plutonium (IV) oxalate to oxide has shown the presence of both carbon dioxide and carbon monoxide in large quantities. The decomposition follows the equations:

Wöhler and Schuff⁽²⁾ found that the alkaline earths decompose nearly quantitatively to the carbonates, according to the equation $\text{MeC}_2\text{O}_4 \longrightarrow \text{MeCO}_3 + \text{CO}$ with a small amount of carbon being formed from the reaction $2\text{CO} \longrightarrow \text{CO}_2 + \text{C}$, also described by Ott⁽³⁾. D'Eeye and Sellman⁽⁴⁾ found that thorium oxalate also decomposed to the carbonate. Günther and Rehaag⁽⁵⁾ found that neodymium oxalate decomposes to the peroxide-dioxalate in high yields, and decomposition of sodium, barium, calcium, and thorium oxalates yielded traces of the metal peroxides. Since only traces of both carbonate and peroxy oxygen were found, the main decomposition path of plutonium (III) oxalate must not be the same as those given by these authors.

The temperature, rather than the time at a particular temperature, is the more important factor in the degree of decomposition, as is shown in Figure 6.

Decomposition in Air or Oxygen

In air or oxygen, plutonium (IV) oxalate hexahydrate loses five waters of hydration at ca. 80°C to form the monohydrate, which is, in turn, converted

to the anhydrous form between 80 and 150 C. The anhydrous oxalate begins decomposition to the plutonium (III) oxalate at 130 to 150 C. Plutonium (III) oxalate undergoes a rapid exothermic reaction in air or oxygen, commencing at ca. 150 to 200 C, which occasionally causes a cake temperature rise of several hundred degrees. The cake temperature rise usually occurs with thixotropic (clay-like) oxalate cakes. (Figures 7 and 8 show the cake temperatures during conversion of two thixotropic cakes.) Since little shrinkage occurs in the conversion of plutonium (IV) oxalate to the (III) oxalate, the thixotropic oxalates form a hard cake with no cracks and allow air to contact only the surface of the cake. A number of thixotropic oxalates have shown a relatively large amount of plutonium (III) oxalate in the interior of the cake (determined by color and oxalate content) after heating to ca. 150 C. When the plutonium (III) oxalate begins to decompose, considerable shrinkage occurs, causing crack formation in the cake and allowing greatly increased contact with air. Figure 9 shows the effect of oxygen on plutonium (III) oxalate decomposition, both in temperature rise and in weight loss. The reaction in other similar runs has been arrested by flooding the reaction vessel with argon, and re-initiated by again admitting oxygen, showing that the exothermic reaction is oxygen dependent. The heat produced in the exothermic reaction is a result of the burning of carbon monoxide produced in the decomposition reaction $(1/2 \text{ Pu}_2(\text{C}_2\text{O}_4)_3 \longrightarrow \text{PuO}_2 + \text{CO}_2 + 2\text{CO})$, with the reaction starting at a fairly low temperature due to a catalytic effect of the plutonium oxide surface.

The various steps of the decomposition of plutonium (IV) oxalate hexahydrate in air have been observed visually during calcination of a thin cake with infra-red light. The colors of the various decomposition products are quite distinctive. The hexahydrate is tan, the monohydrate a chocolate-brown, and the anhydrous oxalate is a light tan. Plutonium (III) oxalate is turquoise-blue. When the plutonium(III) oxalate began decomposition, a yellow, unidentified compound appeared, and then the cake appeared to burn, much like coals from a fire, with occasional sparks.

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Cakes ca. 0.4 cm thick have been calcined to plutonium oxide in a half hour with infra-red light.

The temperatures at which the various decomposition reactions occur could only be approximated. As indicated by the temperature given, the temperature at which plutonium (III) oxalate is formed in air and decomposes is not known, accurately, but small amounts probably form at the lower temperature given (130 C) and decompose at temperatures as low as 150 C.

DIRECT HYDROFLUORINATION OF PLUTONIUM (IV) OXALATE

Hydrofluorination of plutonium (IV) oxalate was demonstrated by Facer and Lyon⁽⁶⁾, who dried the oxalate, then fluorinated at 130 C, with a final heating at 500 C in an anhydrous hydrogen fluoride-oxygen mixture.

Hydrofluorination of plutonium (IV) oxalate below 100 C produces the tetrafluoride (shown by the pink color), but water, excess hydrofluoric acid, and oxalic acid remain in the cake (this increase in weight is shown in Figure 10). Heating at 300 C in a hydrogen fluoride-oxygen atmosphere converted the tetrafluoride into a compound with a molecular weight of 289 to 290. This latter compound was also observed when the oxalate was placed in a hydrogen fluoride-oxygen atmosphere and heated as rapidly as possible to 300 C or 500 C (runs 238 and 239, respectively, as shown in Figure 10). This compound is rapidly converted to plutonium trifluoride at 300 C. The trifluoride is converted to the tetrafluoride when heated at 500 C in hydrogen fluoride oxygen gas mixture.

HYDROFLUORINATION OF PLUTONIUM DIOXIDE

The reactivity of plutonium oxide with hydrogen fluoride is greatly influenced by the maximum temperature of calcination used to convert plutonium (IV) oxalate to the oxide. An initial fast reaction takes place during hydrofluorination of plutonium oxide. The rate of this reaction is nearly the same for all plutonium oxides of the same cake depth. The temperature of calcination determines when this fast reaction is replaced by a much slower reaction. Oxides which have been calcined at temperatures above 480 C have

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a very short initial fast reaction time, while oxides calcined at 480 C have a much longer fast reaction time (Figures 11, 12, 13, and 14).

The reactivity of the oxide is changed slightly, depending upon the plutonium (IV) oxalate calcined. Thixotropic oxalates (which seem to have smaller particle size) produce the most reactive oxides.

The temperature of calcination, rather than the time heated at a particular temperature, has the major effect on oxide reactivity. Very little change in reactivity was noted when an oxalate was calcined for only half a minute, or for 90 minutes at the same temperature (480 C), as shown in Figure 15.

Hydrofluorination temperature does not affect the rate of reaction to as great a degree as calcination temperature. Hydrofluorinations at temperatures above 500 C slightly increase the per cent of oxide converted to the tetrafluoride in one hour (Figures 11, 12, 13, 14, and 16). This effect is indistinct at low temperatures of calcination (300 C) but becomes more definite at higher temperatures; i. e., 500 C and above, as shown in Figure 16.

The depth of the cake influences the slope of the fast reaction (Figure 17). This is due to a difference in the time of diffusion of hydrogen fluoride into the cake, which would be greater in the thick cakes, and produce a slope change.

The effect of the temperature of calcination may be due to sintering and recrystallization of the plutonium oxides produced at temperatures over 500 C. Table I shows an increase in crystallite size, measured by X-ray line broadening, between 300 C oxide and 600 C oxide.⁽⁷⁾ Although crystallite sizes range from 110 to 300 Angstrom units, actual particle size may be many times larger, as reported for uranium dioxide by Anderson, Harper, Moorbatch, and Roberts.⁽⁸⁾ This difference in crystallite size would result in 2.6 times as much surface area with the 300 C oxide as that of the 600 C oxide. The initial fast reaction during hydrofluorination is with the surface

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of the particle. The slow reaction results from the diffusion of hydrogen fluoride into the interior of the particles. Figure 18 shows that the rapid decrease in the extent of the initial fast reaction, which is observed at 500 C, accompanies a decrease in relative surface area, also at 500 C. An additional indication that the initial reaction is with the surface of the particle is the change of slope of the fast reaction with increased cake depth, with little change in the slow reaction rate (Figure 19). The bulk density also increases from 1.8 to 2.6 grams of plutonium per cubic centimeter for 300 C and 600 C oxides, indicating increased particle size in the 600 C oxide.

Optimum conditions for hydrofluorination of plutonium dioxide are calcination of the plutonium (IV) oxalate to the oxide at temperatures not over 480 C, and hydrofluorination at 500 C. Under these conditions, 93 to 98 per cent conversion to the tetrafluoride is accomplished in one hour (Figure 9). Greater conversion (98 to 100 per cent) may require as much as two hours longer. Although calcination and fluorination at 300 C may result in slightly greater conversion to the tetrafluoride, the product may cause excessive bomb pressures in the reduction step, as indicated from reduction studies by Lyons⁽⁹⁾, who found that the bomb pressures doubled for fluorides produced at 300 C as compared with 600 C fluorides.

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REFERENCES

- (1) Munsell Book of Color, Vols. I and II, Munsell Color Company, Inc. (1952).
- (2) Wöhler, L. , and Schuff, W. , "Silicides of the Alkaline Earths", Z. Anorg. Allgem. Chem., Vol. 209 (1932), p. 57.
- (3) Ott, S. , Z. Physik. Chem. , Vol. 109 (1924), p. 1.
- (4) D'Eye, R. W. M. , Sellman, P. G. , The Thermal Decomposition of Thorium Oxalate. AERE-CR-1286, November 2, 1953 (Unclassified).
- (5) Günther, P. L. , and Rehaag, H. , "Thermal Decomposition of Oxalates, I: The Formation of Peroxides through Thermal Decomposition of Oxalates in Vacuum", Chem. Ber. , Vol. 71, (1938), p. 1771.
- (6) Facer, J. F. , Lyon, W. L. , and Weidenbaum, B. , Preparation of Plutonium Tetrafluoride by Direct Hydrofluorination of Plutonium Oxalate. HW-20164, February 1, 1951. (Secret).
- (7) Pallmer, P. G. , Crystallite Sizes of PuO₂ Powders. HW-44585, July 27, 1956. (Unclassified).
- (8) Anderson, J. S. , Harper, E. A. , Moorbath, S. , Roberts, L. E. J. , The Properties and Microstructure of Uranium Dioxide; Their Dependence upon the Mode of Preparation. AERE-CR-886, August 19, 1953. (Secret).

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TABLE I
EFFECT OF TEMPERATURE ON
PLUTONIUM OXIDE CRYSTALLITE SIZE

<u>Temperature of Calcination</u>	<u>Diameter^(a) of Crystallite (A°)</u>	<u>Surface Area</u> <u>Volume</u> of Oxide Produced at Temp. T ^(b)	
		<u>Surface Area</u> <u>Volume</u>	of Oxide Produced at 600 C
600°C	280	1.0	
550 C	250	1.1	
510 C	170	1.7	
480 C	110	2.6	
300 C	110	2.6	

(a) Diameter of crystallite determined by X-ray method. Actual particle size may be many times this figure. See references 7 and 8.

(b) This is a calculated value based on the assumption that all particles are spherical.

FIGURE 1

Weighing Apparatus for Study of Plutonium (IV)
Oxalate Decomposition; and Hydrofluorination
of Plutonium (IV) Oxalate and Plutonium Oxide.

FIGURE 2

Decomposition of Plutonium (IV) Oxalate During
Storage at Room Temperature

FIGURE 4
Thermal Decomposition of Plutonium (IV)
Oxalate in Argon

FIGURE 5
Oxalate Content During Thermal Decomposition
of Plutonium (IV) Oxalate in Argon

FIGURE 6
Effect of Heating Time on Decomposition
of Plutonium(IV) Oxalate in Argon

FIGURE 7

Plutonium (IV) Oxalate Decomposition
in Air Atmosphere (Thixotropic Cake)

FIGURE 8

Plutonium (IV) Oxalate Decomposition
in Air Atmosphere (Thixotropic Cake)

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FIGURE 9

Decomposition of Plutonium (III)
Oxalate in Oxygen Atmosphere

FIGURE 10

Hydrofluorination of Plutonium (IV) Oxalate

FIGURE 11
Effect of Calcination Temperature
on The Hydrofluorination of PuO_2

FIGURE 12

Effect of Calcination Temperature
on The Hydrofluorination of PuO_2

FIGURE 13
 Hydrofluorination of PuO_2 - Effect
 of Calcination Temperature

FIGURE 14
Hydrofluorination of PuO_2 - Effect
of Calcination Temperature

FIGURE 15
Hydrofluorination of PuO_2 - Effect
of Calcination Time

FIGURE 16

Hydrofluorination of PuO_2 - Effect of Calcination Temperature and Hydrofluorination Temperature

FIGURE 17

Hydrofluorination of PuO_2 - Effect of Cake Depth

FIGURE 18

Effect of Calcination Temperature on Surface Area of PuO₂ and Reaction of PuO₂ with Hydrogen Fluoride

FIGURE 19
Hydrofluorination of PuO₂ - Effect of Cake Depth