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DIRECT CALCINATION - FLUID BED CHLORINATION
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PARAMETERS IN THE CONVERSION OF PLUTONIUM NITRATE TO PLUTONIUM TRICHLORIDE BY A DIRECT CALCINATION - FLUID BED CHLORINATION PROCESS

By

M. J. Rasmussen
G. L. Stiffler
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Research and Engineering Operation
Chemical Processing Department

May 31, 1961

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PARAMETERS IN THE CONVERSION OF PLUTONIUM NITRATE
TO PLUTONIUM TRICHLORIDE BY A DIRECT CALCINATION -
FLUID BED CHLORINATION PROCESS

I. INTRODUCTION

The purpose of this study is to describe the presently known parameters for the direct calcination of plutonium nitrate in an agitated bed, and the conversion of plutonium dioxide to plutonium trichloride by a fluid bed process. The report includes a description of the process and the demonstration equipment. The study of variables is discussed. The parameters are described and the plan for demonstration runs is presented.

The report is mainly concerned with the reactivity of plutonium dioxide, which was found to be the key factor in obtaining complete conversion of the oxide to the trichloride. Discussion is aimed at clarifying the variables of concern. Finally, limits will be set for the equipment and operating parameters for the direct calcination and fluid bed conversion. These values apply to the laboratory demonstration unit, but should be readily susceptible to scaleup.

II. SUMMARY AND CONCLUSIONS

Variables in the direct calcination of plutonium nitrate to dioxide have been explored and the effects on reactivity determined. Reactivity was evaluated by chlorination rate in thermobalance and tube furnace tests. The significant effects are due to differences in the oxide particle shape, size, and density. These are influenced by variables such as residence time, temperature, chemical additives, and thermal cycling.

Parameters for a continuous direct calcination process in an agitated bed reactor are presented, and include (1) sizing for minimum residence time, (2) temperature of 250 C, (3) agitator speed of 160 rpm to optimize particle size, (4) use of sulfate or oxalate additive, and (5) feed rate of two liters per hour.

Fluidized bed chlorination parameters include (1) 500 C operation, (2) gas flow of 0.1 to 0.5 feet per second, (3) sizing to produce residence time of four hours, and (4) two stages with cooling between.

Large-scale demonstration equipment is on hand and demonstration runs are expected during the next six months.

III. DISCUSSION

A. Process Description

1. Chemistry

The direct calcination of plutonium nitrate solution includes

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the evaporation of excess water and free nitric acid, the removal of water of crystallization at about 70 - 130 C, and decomposition of the nitrate at 130 - 225 C. The thermogravimetric curve of Drummond and Welch⁽¹⁾ represents the reaction events as they occur in sequence with slowly increasing temperature.

When solution is added dropwise onto a hot powder bed at 275 C, the processes occur almost instantly and local temperatures vary rapidly.

The following theoretical equation is used to represent the calcination step and dissociation of nitrogen dioxide:⁽²⁾

That this is only an approximation is known. Thus the product powder analyzes 0.072 percent nitric acid and <0.05 percent water.

The plutonium dioxide is fluidized and chlorinated homogeneously by a stream of phosgene gas passing through the powder at 500 C. Phosgene gas was selected as the most reactive chlorination reagent⁽³⁾ tested. The primary reaction is as follows:

Conversion must be at least 95 percent complete to provide quality product for the reduction step.

The plutonium trichloride product is hygroscopic. A dry atmosphere of dew point -20 C or better must be provided.

2. Direct Calcination Equipment

A trough-type calciner was selected because (1) favorable experience with UNH calcination and (2) criticality problems appeared readily solvable.

The experimental continuous calciner (Figures 1 and 2) is essentially a stainless steel trough 12 inches long, 3 inches in diameter, and 3 inches high. The plutonium oxide bed is agitated by means of a non-conveying T-bar scraper-blade-type agitator located concentrically with the trough. Three assemblies of two blades each are placed at equal intervals around the circumference of the shaft so that only one blade enters the powder at any instant.

A spacing between blade assemblies provides for the placement of a feed injector and thermocouple. The level of the bed is maintained by means of an overflow weir such that the agitator is about one-half submerged.

The bed is heated by two coils of nichrome wire. One coil supplies heat to the eight-inch feed section, and the other heats the four-inch finishing section. The coils are rated at 2000 and 1000 watts respectively. The power input to each coil is controlled by a "Variac" variable transformer.

Off-gas is discharged through a two-inch settling chamber located in the calciner cover opposite the powder discharge end. Vapors leaving the calciner are further cleaned in a cyclone separator followed by a ceramic filter. Operating conditions are measured by thermocouples located in the PuO_2 at the feed point and at the discharge end. Selected temperatures can be maintained by balancing the power input with the rate of feed addition.

The material of construction is 304L stainless steel.

3. Fluidized Bed Reactor

A previous study⁽³⁾ provided operating data on a vibrating tube reactor for chlorinating plutonium dioxide. This process used as feed the very fine plutonium dioxide resulting from calcination of plutonium oxalate. The fluid bed conversion process, however, is intended to be used in a processing complex consisting of the direct calcination of plutonium nitrate to dioxide, fluid bed conversion to plutonium trichloride, and electroreduction to the metal. Hence, the requirements are different.

A fluid bed reactor was picked for the following reasons: (1) the particle size of directly-calcined oxide was expected to be relatively large, hence a vibrating tube could not be applied; (2) the large holdup time of a fluid bed reactor could be used to advantage in obtaining complete chlorination of the less-reactive powder; (3) better temperature control was anticipated; and (4) fluid bed equipment was expected to be free of the startup uncertainties found with vibrating tube reactors.

The experimental fluid bed reactor is constructed⁽⁴⁾ of Hastelloy C from nominal two-inch diameter Schedule 40 pipe. A temporary section of Vycor permits observation of fluidization. Gases flow in sequence through rotameters, gas preheater, filter (to prevent contamination back-flow), gas distribution plate, powder bed, settling chamber, primary filter, secondary filter, and thence to a caustic scrubber. (See Figures 3 and 4.)

Heat is supplied by means of asbestos-covered nichrome wire wrapped around the equipment. Each unit is a 1000 watt heater controlled by a Variac. Temperatures are registered via chromel-alumel thermocouples on a strip chart recorder, except that the powder bed temperature is registered on a controller-recorder which controls temperature in the bed area.

Startup of this unit will be for batch chlorinations. Additions of a second stage and continuous powder flow equipment are pending.

B. Variables Affecting The Reactivity Of Plutonium Dioxide

It was found early that plutonium dioxide formed continuously by direct calcination of plutonium nitrate tended to be relatively-unreactive toward phosgene, compared with oxide from oxalate. Also, conversions to trichloride leveled off far below completion. At least 95 percent conversion is necessary to provide good feed for an electro-reduction step.

This section discusses the variables and factors affecting the reactivity of plutonium dioxide. Due to difficulties in experimenting with the large equipment, many of the tests were made in stirred beaker runs with tube furnace chlorinations.

1. Structure Of Particles

Photomicrographs of the unreactive powder from continuous calcination runs showed a relatively-round particle, presumably with a layered structure. This results from successive evaporations of drops of liquid on a particle already formed. Powder from calcination of oxalate does not show this structure, nor does batch calcined oxide. The reactivities of these materials are compared in Table I.

TABLE I

CHLORINATION REACTIVITY OF PLUTONIUM DIOXIDE

<u>Source</u>	<u>Percent Chlorination In 20 Minutes</u>
Oxide from oxalate	95
Oxide from batch calcined nitrate	61
Oxide from continuous calcined nitrate	20

The non-layered type of structure as formed from calcination of oxalate particles is more reactive. Hence, successive buildup on particles is to be minimized.

Best guess is that an open structure is required. Access to the core of the particle by gas penetration of pores is probably the key to complete conversion, since at least 20 percent of the material is rapidly converted in all cases.

2. Particle Size

Plutonium dioxide formed in the agitated bed calciner was sampled and ball milled.

Chlorination tests using a thermobalance* were used to show the effects of particle size on reaction rate. Chlorination was more rapid on the small particles, but the reaction still did not go to completion.

TABLE II

EFFECT OF PARTICLE SIZE ON CHLORINATION

<u>Particle Size</u>	<u>Percent Conversion</u>	
	<u>1 hour</u>	<u>4 hours</u>
{ 80% 105-270 μ , 19% 49-105 μ }	19	44
100% <49 μ	79	79

Besides affecting reactivity, particle size also affects the off-gas filtration problem. More important, fluid bed reactors function well only with a certain range of particle sizes.

The particle size desired is about 200 microns with only trace-amounts smaller than 100 μ or larger than 500 μ . However, tests in a one-inch diameter fluid bed indicate that fluidization can be achieved with 50 percent of the particles 105-250 μ diameter and 45 percent in the range of 49-105 μ . Powders finer than this are expected to require a stirrer in the fluid bed.

3. Calciner Residence Time

The effect of residence time at temperature was investigated in batch calcinations of plutonium nitrate on a beaker-scale. The cessation of nitrogen dioxide fumes was taken as zero time and the powder was then stirred and sampled at intervals. Chlorination tests were run in a tube furnace and percent conversion determined. The lower residence times resulted in the best conversions, as shown in Table III.

(*) Thermobalance runs were performed by R. Sorenson, Process Chemistry Operation.

TABLE IIICHLORINATION REACTIVITY AS A FUNCTION OF CALCINATION TIME

<u>Calcination Time</u> <u>Hours At 300 C</u>	<u>Percent Conversion</u>		
	<u>1 Hour</u>	<u>4 Hours</u>	<u>5 Hours</u>
0.1	86	87	--
2	61	86	--
6	18	72	75
20	6	40	57

Presumably the continued heating at even this low temperature caused crystal growth⁽⁵⁾ and the removal of the crystal imperfections which enhance chemical reactivity. Apparently these samples do not reach equilibrium in a reasonable time.

A high calcination temperature will also favor unwanted sintering of the oxide. Hence, the lowest feasible temperature for shortest possible time is preferred. Temperatures in the range 230 - 275 C appear optimum.

4. Thermocycling

One method of improving conversion is thermocycling to induce cracking, pore formation, and access to the interior of the particles.

In one test, partially-chlorinated oxide was cooled to < 100 C between two 20-minute treatments with phosgene at 500 C. Conversion after the first treatment was 20 percent, and 52 percent after the second. Further tests of this effect will be done.

5. Additives

Additives were suggested by the fact that plutonium dioxide from oxalate has such a high reactivity. Sulfate as an additive has been useful in improving UO₃ reactivity. Plutonium sulfate is readily converted to plutonium trichloride by phosgene under standardized conditions.

The use of additives in calcination to increase plutonium dioxide powder reactivity has been actively investigated. Data from small-scale tube furnace chlorinations and from thermobalance runs are shown in Table IV. The oxide samples were obtained from beaker-scale continuous calcination runs in which the additive is introduced

to the calcination beaker along with the plutonium nitrate.

TABLE IV
EFFECT OF ADDITIVES ON THE REACTIVITY OF PuO₂

<u>Additive</u>	<u>Moles Per Mole Pu</u>	<u>Powder Bulk Density g/cc</u>	<u>Predominant Particle Size, Microns</u>	<u>Reactivity*, Percent Conversion To PuCl₃</u>	
				<u>Tube Furnace**</u>	<u>Thermo- balance</u>
None	---	5.0	175	63	--
H ₂ C ₂ O ₄	0.2	6.0	75	66	47
H ₂ C ₂ O ₄	1.0	---	75	--	84
H ₂ C ₂ O ₄ Starting bed	2.0)	5.0	75	100	98
H ₂ C ₂ O ₄ Solution	1.0)				
(NH ₄) ₂ SO ₄	0.2	3.0	800	71	85
(NH ₄) ₂ SO ₄	0.5	2.7	1000	100***	85

(*) Tests run on a standard size fraction.

(**) Twenty-minute time cycle.

(***) Tests for remaining sulfate were negative.

Both additives tried produced an improvement in powder reactivity. Sulfate addition caused formation of large particles of low-bulk density. Oxide from the standard continuous oxalate process was completely converted in both tests.

Density has not been investigated as a separate variable. Density is a factor in reactivity in that a less-dense particle is usually more reactive. Powder density affects the holdup in a calciner of fixed geometry. Low density is desirable to ease design problems for critical mass safety.

C. Process And Equipment Parameters

1. Agitated Bed Calciner

This section describes those parameters which can now be fixed. Reference is to a laboratory demonstration complex of minimum capacity of 250 grams per hour plutonium.

a. Size - Holdup

The calciner is essentially a trough 3 inches in diameter, 3 inches high, and 12 inches long. The calciner residence time of the powder was found to have a significant effect upon powder reactivity. The residence time must be minimized. By adding cylindrical blocks 1.75 inches in diameter to the 0.625-inch agitator shaft, the holdup of the calciner

has been reduced to about 230 cc. This bed volume is equivalent to a residence time of about two hours. It is impractical to decrease the bed volume much further.

b. Temperature

The reactivity of calcined plutonium dioxide decreases with an increasing calcination temperature, due to perfection of the crystal lattice. Consequently, the calciner should be operated to maintain a bed temperature of about 250 C, which is just above the 220 C decomposition temperature of $\text{Pu}(\text{NO}_3)_4$.

c. Agitation

Agitator speed will affect both the particle size of the plutonium dioxide powder and the rate of heat transfer between the powder and the calciner wall. The particle size of the powder will decrease with increasing agitator speed due to attrition; the heat transfer coefficient will increase. An agitator speed of 160 rpm (a linear velocity of 2.1 ft/sec) will produce particles having a diameter of about 170 microns. The heat transfer coefficient at this speed is about 57 btu/hr·ft²·°F.

d. Additives

The addition of certain chemicals to the calciner simultaneously with the feed solution can greatly improve the reactivity of calcined PuO_2 . Additives to be recommended include any of the following:

- (1) 0.1 mole of oxalic acid per mole of plutonium.
- (2) 1.58 mole of oxalic acid per mole of plutonium.
- (3) 0.5 mole of ammonium sulfate per mole of plutonium.

Chemical additives in solution are added to the feed solution at a point in the feed line just preceding the calciner. The volume of off-gas is minimized by using saturated solutions of the additives.

e. Feed Rate

The furnace which heats the calciner has an output of 3000 watts. This output is sufficient to calcine standard plutonium nitrate solution, containing 250 g of Pu per liter, 4 M HNO_3 , at a rate up to two liters an hour. The output of the calciner can be increased considerably, however, if the feed solution is concentrated to the satura-

tion point of about 600 g per liter.

2. Fluidized Bed Chlorinator

a. Temperature

The optimum temperature for the reaction is 500 C. Reaction rates are apparently not improved by higher temperature, but corrosion may become serious. At 450 C, the reaction rates are only slightly reduced.

b. Gas Flow

The gas flow rate required for good fluidization is a superficial velocity (assume column empty, but at observed temperature) of 0.1 to 0.5 ft/sec in the fluid bed. Good fluidization has been demonstrated at 0.15 ft/sec velocity. A powder with high density, small particles, may well require the 0.5 ft/sec flow rate of argon or phosgene.

Argon is used to start or shut down the fluid bed column. This provides isolation of test periods and a means of removing phosgene from the product. Phosgene will normally be used undiluted, although it can be diluted with argon to slow the reaction rate during startup.

c. Residence Time

The plutonium dioxide in the vibrating tube reactor⁽³⁾ required three times the stoichiometric amount of phosgene for completion of the chlorination. Assuming the same factor for the two-inch fluid bed, the chlorination rates were calculated for a few typical flow rates (Table V, Column 2).

Powder residence times were based on further assumptions (1) a nine-inch bed depth expanding to 10 inches during operation and (2) powder bulk densities would be bracketed by the values 3 g/cc (sulfate additive) to 6 g/cc (highest observed value); i.e., plutonium densities 2.1 g/cc for low-density chloride to 5.4 g/cc for high-density oxide.

d. Number Of Stages

For initial test purposes only one column of two-inch diameter was built for a batch-type operation. At this time, it appears that two stages will be necessary for 95 percent chlorination. This also permits use of thermocycling (cooling to <100 C) in the reservoir supplying

the second stage.

TABLE V

RESIDENCE TIME OF POWDER IN FLUID BED

Phosgene Gas Flow ft/sec	Plutonium Chlorination Rate, g/hr	Residence Time - Hours	
		Bulk Density	Bulk Density
		3 g/cc	6 g/cc
0.15	245	4.3	10.9
0.3*	590	1.8	4.5
0.5*	817	1.2	3.3
Pu in bed		1043 g	2681 g

(*) The figures on these lines are not corrected for the increased bed expansion expected at these flow rates.

D. Plans

1. Calcination

Further studies on a large scale using the agitated bed calciner will be started as soon as small-scale reactivity testing is completed. Runs will use the best combination of time, temperature, and additive indicated in small tests to give a reactive product.

2. Fluid Bed Chlorination

The prototype fluid bed of 2" diameter has been tested for operability as a single stage. When reactive oxide from the calciner is available, the chlorination rate can be adequately tested. Multiple-stage operation (thermocycling) will be simulated by intermittent operation of the single column. If good chlorination is achieved, multiple-stage operation with continuous powder flow will be tested.

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FIGURE 1

PLUTONIUM NITRATE CALCINER

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FIGURE 2
PLUTONIUM NITRATE CALCINER
(Lid Removed)

FLUID BED CHLORINATOR

FIGURE 3

FIGURE 4
FLUID BED CHLORINATOR
(Single Stage)