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FINAL REPORT
PRODUCTION TEST 234-5
PLANT PROCESS EVALUATION PRECIPITATION
OF PLUTONIUM IV OXALATE

BY
W. B. KERR
TECHNICAL SECTION
ENGINEERING DEPARTMENT

DECEMBER 3, 1952

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Technology - Hanford Processes

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FINAL REPORT
PRODUCTION TEST 234-5
PLANT PROCESS EVALUATION
PRECIPITATION OF PLUTONIUM IV OXALATE

By

W. B. Kerr

Process
Separations Technology Unit

December 3, 1952

HANFORD WORKS
RICHLAND, WASHINGTON

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FINAL REPORT
PRODUCTION TEST 234-5
PLANT PROCESS EVALUATION
PRECIPITATION OF PLUTONIUM IV OXALATE

I. INTRODUCTION

Prior to January 1952, the processes for the preparation of metallic plutonium in the 234-5 Building affected by this Production Test included the following steps:

- a. Receipt of the 231 Building product effluent (AT) solution. This was approximately 1.5 molar tetra and hexavalent plutonium nitrate in nitric acid solution.
- b. Reduction of the plutonium to the trivalent state by hydriodic acid, then precipitation as plutonium (III) oxalate.
- c. Conversion of the plutonium (III) oxalate to plutonium (IV) fluoride by anhydrous hydrogen fluoride and oxygen at elevated temperatures.
- d. Reduction of the plutonium (IV) fluoride to plutonium metal by calcium.

The hydriodic acid as used in step "b" and the iodine which results from that step were very corrosive and required special construction materials. Glass, glass-lined steel, tantalum, and plastics had been used as construction materials for the equipment in which hydriodic acid was used. However, maintenance and replacement costs of this equipment, as well as normal construction materials unavoidably exposed to HI vapors, were very high. For this reason, a development program was pursued at Hanford Works and Los Alamos ^(1, 2) to replace the Pu (III) oxalate precipitation process with one requiring less corrosive chemicals.

This objective, along with process simplification, appeared feasible when a process for converting plutonium peroxide to plutonium tetrafluoride was developed concurrently by the Hanford and Los Alamos development groups. Production Test 234-3⁽³⁾ was a process evaluation of the laboratory procedures for the hydrofluorination of plutonium peroxide and was reported in reference #4. The metal produced by this method was of adequate purity but several disadvantages were inherent in the process. Those disadvantages were:

- a. The filtered cake and tetrafluoride had a low bulk density.
- b. It was necessary to dehydrate the plutonium peroxide prior to heating or hydrofluorination to prevent splattering and caking. A methyl alcohol wash was used to remove the water from the filtered peroxide; however, this introduced safety hazards in handling process streams.
- c. The reduction yields were erratic - ranging from 90.8 to 97.6 per cent.

Inasmuch as the performance characteristics of the peroxide process revealed several deficiencies an alternate process, plutonium (IV) oxalate, which had received some laboratory study with encouraging results, was carried through development to evaluate it with respect to precipitation, dehydration, and hydrofluorination characteristics. This "plus 4" process eliminated the hydriodic acid, as did the peroxide process. In addition, it eliminated the use of alcohol, produced a tetrafluoride having a bulk density approximately twice that produced by the peroxide process (this increased bulk density was expected to result in consistently high reduction yields), and produced a supernate that could be recycled to the E-4 Tank of the 224 Building without further processing.

The current report has been written to record the data and present the conclusions derived from production testing of the "plus 4" oxalate process in production plant equipment as outlined in reference #5.

II. OBJECTIVES

The object of the work described in this report was to adapt the laboratory procedures for precipitating and hydrofluorinating plutonium (IV) oxalate to plant equipment on 160 and 350 gram scale. This adaptation was done by making minor process changes in concentration, digestion time, rate of chemical addition, etc.

III. SUMMARY AND CONCLUSIONS

The IV process, discussed below, was shown to be satisfactory for adoption for plant use. Nineteen batches containing 160 grams of plutonium and five batches containing 300 to 350 grams of plutonium were purified by the plutonium (IV) oxalate precipitation process in the 234-5 Building. Seven batches containing a maximum of 350 grams of plutonium were purified by the same process in the 231 Building, collected on a filter, and transferred to the 234-5 Building. All of the materials mentioned above were processed in the RG Line hydrofluorination equipment.

Significant observations regarding the plutonium (IV) oxalate process were:

- a. The reduction yields and purity of the reduced metal were satisfactory. Yields averaged 98.4 per cent and ranged from 96.1 to 100.2 per cent.
- b. The purification cycle using settling and decanting procedures was 5* hours for the Pu (IV) compared with 3* hours for the Pu (III) oxalate process on the 160 gram batch; however, the Pu (IV) process on a 350 gram batch saved 3* hours/Kg of Pu over that required by the Pu (III) process on a 160 gram batch.
- c. The hydrofluorination time cycles for the (IV) process were 6-1/2* hours for a filtered cake and 8-1/2 hours using a slurry compared with 9-1/2* hours for the III process.

* These times are approximate.

- d. The untreated supernates are chemically suitable for recycle to the 224 Building although treatment may be necessary to achieve plutonium accountability. Samples of the supernatant solutions indicated 0.3 to 0.4 per cent of the batch was present, but a material balance between the 231 and 234 Buildings indicated that about 8 per cent should be in the supernatants. Treatment of the supernates may eliminate this discrepancy. Waste losses and accountability discrepancies will be evaluated in the Final Report of Production Test 231-14.
- e. The tetrafluoride produced by hydrofluorinating the (IV) oxalate was a free-flowing powder with an average bulk density of 1.5 g/cc ranging from 1.22 to 1.95. This compared very favorably with the bulk density of about 1.0 g/cc by the (III) oxalate process and approximately 0.7 g/cc using alcohol-washed peroxides.

IV. RECOMMENDATIONS

These recommendations were made following analysis of the observations and data:

- a. That a plutonium (IV) oxalate precipitation and subsequent filtration process be adopted for the RMA Line.
- b. That the hydrofluorination process shown in curve "C" - Figure "1" be used as the process at RMA start-up.
- c. That further work be done as part of Production Test 231-14 to achieve adequate accountability control for the (IV) process.
- d. That the plutonium (IV) purification and hydrofluorination processes be adopted in the RG Line and that the early installation of a filter for collecting the plutonium (IV) precipitate be completed.

V. EXPERIMENTAL

A. General

The scope of this test included the production of plutonium (IV) oxalate from regular 231 Building AT solution, the hydrofluorination of the oxalate to the tetrafluoride, and the evaluation of these processes. The purification and hydrofluorination processes were tested in 234-5 Building equipment with minor revisions.

During the latter part of the test, it was deemed desirable to develop a hydrofluorination process for filtered plutonium (IV) oxalate. This material was produced in the 231 Building from dissolved second cycle peroxide cake as part of another production test⁽⁶⁾. A satisfactory process was developed for hydrofluorinating this filtered cake in the RG Line equipment.

B. Equipment

The special equipment and minor modifications to existing equipment required for this test were:

- (1) Three special temperature controller cams for the hydrofluorination furnace were supplied and modified during the test. Typical temperature curves are given in Figure 1, curves A, B, and C.
- (2) The purification hood was revised to permit decanting supernatant liquids directly to the recycle can.
- (3) Special solutions required for this test were introduced to the purification equipment via existing piping.

C. Solutions

The special solutions required for this test are listed below with permissible ranges.

- (1) Hydrogen peroxide - $2.0 \pm 0.2\%$, $3.0 \pm 0.3\%$, and $6.0 \pm 0.6\%$.
(2) Oxalic Acid - $1.0 \pm 0.03\%$ M.
(3) Wash Solution - two concentrations were used.
 2.0 ± 0.05 M HNO_3 - 0.45 ± 0.05 M oxalic acid
 and 1.5 ± 0.05 M HNO_3 - 0.45 ± 0.05 M oxalic acid.

D. Process

The process steps found to be satisfactory for purification and hydro-fluorination using the (IV) process are given below.

1. Purification

For 160 gram batch

- a. The plutonium nitrate (AT) solution, as received from the 231 Building, was transferred to the Purification Reactor by means of distilled water flushes of the shipping container (Sample Can).
- b. The Reactor contents were diluted to 2100 cc.
- c. One liter of 3 per cent hydrogen peroxide (2 per cent for one batch) was added to the solution in the Reactor and agitated for 30 minutes. The solution at this point had a calculated H^+ concentration of less than 2.0 g/l.
- d. A total of 1.8 liters of 1.0 M oxalic acid was added in the following order:
 - Add 900 cc. in 10 minutes followed by 10 minutes agitation.
 - Add 600 cc. in 5 minutes followed by 5 minutes agitation.
 - Add 300 cc. in 5 minutes followed by 15 minutes agitation.
- e. The precipitate was allowed to settle for 30 minutes.
- f. The supernatant liquid was decanted to the Recycle Can.
- g. Six liters of acid wash were added and agitated for 5 minutes.

- h. Steps e and f (settling and decanting) were repeated.
- i. Steps g and h (acid wash) were repeated twice.
- j. The precipitate was transferred (slurried) into a flask for transfer to the hydrofluorination operation.

For the 350 gram batch

- a. The diluted volume in the Reactor prior to addition of hydrogen peroxide was 3500 cc.
- b. The hydrogen peroxide concentration was increased to 6 per cent.
- c. The total oxalic acid volume was calculated for each batch to give an approximate 0.065 M oxalic acid concentration after the plutonium had been precipitated.
- d. The precipitate was transferred to two flasks.

2. Hydrofluorination

For 160 gram batch

- a. The oxalate was transferred into the hydrofluorination boats and most of the liquid removed by decanting.
- b. The loaded boats were placed in the hydrofluorination furnace and dried by the cycle listed below.

<u>Temperature C*</u>	<u>Time</u>	<u>Air flow</u>
130	1 hour	1.0 liter/min.
190	1-3/4 hour	1.0 liter/min.
300	1 hour	1.0 liter/min.

- c. The dried oxalate was hydrofluorinated at 300 C* for one hour and at 600 C* for two hours. Gas flow during hydrofluorination was:

HF-600 gm/hr and O₂ - 250 ml/min.
(see curve B, Figure 1)

* All temperatures are relative because of the location of the indicating thermocouple outside the furnace reaction tube (See Figure 2).

For 350 gram batch

The drying and hydrofluorination process for the 350 gram Purification batch was the same as the process for the 160 gram batch. This was possible because the charge was divided for transferring and hydrofluorination.

For filtered IV oxalate

The drying portion of the hydrofluorination cycle was reduced to one hour at 190 C* and one hour at 300 C*. (See Curve C - Figure 1 for the recommended cycle).

3. Reduction and Casting

Two 160 gram or one 350 gram batch of plutonium tetrafluoride is reduced by calcium to form elemental plutonium buttons. The plutonium buttons were combined to form a casting composed only of test material.

E. Sampling

The samples used for evaluation of this test were:

<u>Sample Code</u>	<u>Material</u>	<u>Analyze for</u>
1. SN-1	composited purification process liquid wastes	Pu
2. DC-1	Pu IV oxalate	impurities
3. B-1 and 2	Reduced metal button	impurities
4. MC-1 and 2	Cast metal	impurities

VI. DISCUSSIONA. Process

The process steps initially determined from laboratory scale studies of the IV oxalate process were:

* All temperatures are relative because of the location of the indicating thermocouple outside the furnace reaction tube (See Figure 2).

1. For the Purification process

- a. Adjustment of the plutonium to the tetravalent state by the addition of hydrogen peroxide to the nitrate solution to a concentration of 0.7 per cent H_2O_2 .
- b. Precipitation of Pu (IV) oxalate by the addition of enough oxalic acid to give a calculated 0.05 M oxalic acid solution in the supernates after precipitation.
- c. Washing of the precipitate with 2 M nitric - 0.04 M oxalic acids solution.

2. For the Drying and Hydrofluorination process

- a. Drying of the plutonium (IV) oxalate.
- b. Hydrofluorination of the plutonium (IV) oxalate (HF flow, 500 g/hr; O_2 flow, 100 ml/min) at 300 C for 1 hour and 600 C for 2 hours as shown by curve A of Figure 1.

Note: These temperatures are relative only and do not represent cake temperatures due to the position of the thermocouple on the outside of the furnace tube. (See Figure 2).

During this test, some process revisions were necessary and some critical steps noted. These are mentioned in the following paragraphs as well as other items observed during the test.

1. Purification

- a. The addition of oxalic acid for IV oxalate precipitation should require approximately 30-45 minutes. Rapid addition results in fine voluminous precipitates which settle slowly and which may pass through a filter. (Note P.T. #2, Table I). Therefore oxalic acid for all succeeding runs was added in three batches over a 35-minute period to be sure that the strike was not too rapid.

- b. Waste loss data as collected for this test and shown in Table I are not dependable. Analysis of the liquid wastes indicates 0.3 to 0.4 per cent waste loss, but material balances indicate approximately 8 per cent. This discrepancy may be due to post-precipitation with the precipitate not being included in the waste sample. At the time of this test, the discrepancy was thought to be due to factors other than this test. Unsatisfactory material balances resulted for the bulk of the material shipped to the 234-5 Building over an extended period which included this test. Complete waste loss data will be included in the Final Report of Production Test 231-14.
- c. It was found desirable to increase the H_2O_2 concentration to 1.0 per cent since with only 0.7 per cent the solution was blue-green, indicating that the plus IV oxidation state had not been reached.
- d. The concentration of the acid wash solution was changed from 2 M nitric - 0.45 M oxalic to 1.5 M nitric - 0.45 M oxalic acid at the suggestion of the development group. This did not improve the material balance nor did it affect the apparent waste loss.
2. Drying and Hydrofluorination (dry chemistry)
- a. It was found unnecessary to dry the oxalate before hydrofluorination. Drying to a damp cake followed by an hour at 300 C before starting hydrofluorination as shown by curve B, Figure 1 made no noticeable difference in the fluoride characteristics (Compare P. T. runs 14 and 15 with runs 1 to 13 Table I). This change shortened the time cycle by approximately one hour.

- b. An increase in oxygen flow from 100 to 250 ml/min. but retaining the HF flow of 500 gm/hr resulted in the production of fluoride having a consistently high bulk density - averaging approximately 1.5 and ranging between 1.2 and 1.9 as shown by runs 16 to 27 in Table I.
- c. The drying portion of the dry chemistry cycle was modified for processing oxalate which had been collected on a filter. This modification reduced the low temperature drying time because there was little chance of spattering from this drier material. This cycle is shown by Curve C - Figure 1.

B. Reduction Yields

The reduction yields from the plutonium tetrafluoride produced under these test conditions were consistently high, averaging 98.4 per cent and ranging from 96.1 per cent to 100.2 per cent. Yields are calculated by this formula:

$$\text{Red. Yield} = \frac{\text{Wt. of Metal - added elements}}{\text{Wt. of Pu in tetrafluoride}^*} \times 100$$

* Assuming tetrafluoride was 100 per cent PuF_4 .

This yield compares very well with long-time average reduction yields of 97.5 per cent.

C. Purity

The metal produced according to this test was satisfactory for further fabrication. Tables II and III are summaries of the analyses performed on this metal.

Table IV is a summary of the impurities found in plutonium IV oxalate as produced on the 160 and 350 gram scale. These data were not used to evaluate the test but are included for future reference.

REFERENCES

- (1) K. K. Kennedy and W. L. Lyon, "Plutonium Peroxide Process for the Purification Building", Document HW-18092, SECRET, 6-15-50.
- (2) W. L. Lyon, "Laboratory Evaluation of One and Two Cycle Peroxide Processing of F-10-P Solutions on the Basis of Metal Purities Obtained", Document HW-21691, SECRET, 7-19-51.
- (3) W. B. Kerr, "Production Test 234-3 Direct Hydrofluorination of Plutonium Peroxide", Document HW-21026, SECRET, 5-23-51.
- (4) W. B. Kerr, "Final Report Production Test 234-3 Process Evaluation - Hydrofluorination of Plutonium Peroxide", Document HW-22802, SECRET, 11-15-51.
- (5) W. B. Kerr, J. M. Hay, "Production Test - 234-5 Plant Process Evaluation Precipitation of Plutonium (IV) Oxalate", Document HW-23203, SECRET, 1-8-52.
- (6) M. J. Szulinski, G. V. Packer, "Demonstration of 231 Building Filter Boat Station with Plutonium IV Oxalate and Plutonium Peroxide Flowsheets", Document HW-23416, SECRET, 1-30-52.

W. B. Kerr

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TABLE I

Process Data and Observation

PT #	Batch Size (grams)	ppt. Vol.	SN-1 Total Units	Density PuF ₄	REMARKS				
					Purification	Dry Chemistry			Reduction ⁽⁴⁾ Yield
						Gas ⁽¹⁾ Ratios	Heating ⁽²⁾ Curve	Notes of ⁽³⁾ fluoride conditions	
1	160	425	0.64	1.466	Slow oxalic addition	1	Curve A	Pink fluoride	98.8
2	160	810	0.414	----	Oxalic acid addition in approx. 5 min.	1	A	Blue-green fluoride	98.9
3	160	300		----	Oxalic acid	1	A	Blue-green fluoride	98.9
4	160	450	1.035	1.098	added in 3 por-	1	A	Pink fluoride	98.8
5	160	600	0.965	1.169	tions over 30	2	A	Blue-green fluoride	98.7
6	160	310	0.408	1.525	to 45 min. period-all suc- ceeding batches.	2	A	Pink fluoride	98.7
7	This run omitted because of operational error.								
8	160	375	1.720	1.250		1	A	Pink fluoride	97.8
9	160	400	1.720	1.286		1	A	Pink fluoride	98.2
10	160	325	1.396	0.786	1	1	A	Pink fluoride	98.2
11	160	400	1.396	1.001		1	A	Pink color-soft lumps	98.6
12	160	300	1.177	1.116		1	A	Blue-Part	96.1
13	160	450	0.637	1.369		1	A	Pink fluoride	96.1
14	160			0.913		1	Curve B	Pink fluoride	96.1
15	160		1.205	1.36		1	B	Blue both boats	100.2
16	160			1.22		2	B	Pink fluoride	100.2
17	160		1.156	1.62		2	B	Pink-soft lumpy	97.9
18	160			1.41		2	B	Pink with small unit blue	97.9
19	160		1.441	1.95		2	B	Pink	97.9
20	350			1.67		2	B	Pink	98.9
21	350		1.057	1.42		2	B	Pink	98.6
22	350			1.53		2	B	Pink	98.0
23	350		1.187	1.38		2	B	Pink	98.1

TABLE I (contd.)
Process Data and Observation

PT #	Batch Size (grams)	ppt. Vol.	SN-1 Total Units	Density PuF ₄	REMARKS					Reduction ⁽⁴⁾ Yield
					Purification	Dry Chemistry			Notes of ⁽³⁾ fluoride conditions	
						Gas ⁽¹⁾ Ratios	Heating ⁽²⁾ Curve			
24	350		0.500	----		2	B	Pink	97.6	

----- All following material delivered for drying and hydrofluorination as a filtered IV -----
oxalate cake

25				1.42		2	Curve C		Pink	99.5
26				1.35		2	C		Pink	98.2
27				1.13		2	C		Pink	100.1
28				----		2	C		Pink	99.1
29				----		2	C		Pink	98.6
30				----		2	C		Pink	99.2
31				----		2	C		Pink	98.7

(1) Gas Ratio

1. HF at 500 gm/hr O₂ at 100 ml/min.
2. HF at 500 gm/hr O₂ at 250 ml/min.

(2) See Figure 1 for heating curves.

(3) Normal tetrafluoride is a fine, free-flowing pink powder.

(4) Two or more test runs on 160 gram batch size combined to make one reduction.

$$\text{Yield} = \frac{\text{Button Wt.} - \text{added elements}}{\text{Pu in tetrafluoride (calculated)}} \times 100$$

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TABLE II
IMPURITY DATA (1)

METAL (BUTTON) FROM HYDROFLUORINATED PU IV OXALATE

	IV Oxalate 160g batch (2)			IV Oxalate 350g batch (3)			IV Oxalate filtered (4)		
	Ave.	Max.	Min.	Ave.	Max.	Min.	Ave.	Max.	Min.
Al	< 19*	< 70	< 13	< 25	< 50	13	< 41*	< 50	< 20
Be	< 0.04*	< 0.08*	< 0.03	< 0.03*	< 0.03	< 0.03	< 0.05*	< 0.1	< 0.02
Ca	< 41	< 100	< 13	< 14	20	13	< 24	50	< 8
Cr	< 1.5	5	< 1	< 1	1	< 1	< 43*	< 50	< 20
Fe	184	242	135	164	245	80	202	245	180
La	80	200	10	172	500	< 20	170	500	< 30
Li	< 1*	< 1	< 1	< 1*	< 1	< 1	< 1*	< 1	< 1
Mg	63	100	20	120	200	50	< 143	200	< 50
Mn	10	20	< 2	21	50	5	39	100	10
Na	16	25	< 5	< 5	5	< 5	< 6	10	< 5
Ni	< 40	200	< 20	422	500	< 20	< 36	50	< 20
Si	9	20	5	12	20	10	11	20	5
B	.7	1.7	0.1	.56	1.0	0.1	.4	.8	.1
F	5.0	8.4	1.7	4.7	8.0	0.2	3.4	6.4	1.9

* No positive results

(1) All values expressed parts per million (ppm) Pu.

(2) 9 batches

(3) 5 batches

(4) 7 batches

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TABLE III
IMPURITY DATA (1)

CASTINGS - METAL FROM PU IV OXALATE

	<u>IV Oxalate (2)</u> <u>160g batch</u>		<u>IV Oxalate (3)</u> <u>350g batch</u>	
	<u>MC-1</u>	<u>MC-2</u>	<u>MC-1</u>	<u>MC-2</u>
Al	13	20	<20	<20
Be	< 0.03	< 0.03	< 0.08	< 0.08
Ca	13	13	<30	<30
Cr	2	2	< 1	< 1
Fe	205	205	155	185
La	130	50	130	130
Li	< 1	< 1	< 1	< 1
Mg	20	20	20	20
Mn	20	20	10	10
Na	15	15	< 5	< 5
Ni	100	100	<20	<20
Si	20	10	10	10
B		0.2		0.7
F		4.4		5.4
C		220		220

(1) All values parts per million (ppm) Pu.

(2) One casting

(3) One casting

TABLE IV
IMPURITY DATA ⁽¹⁾
PLUTONIUM IV OXALATE

	IV Oxalate 160g batch ⁽²⁾			IV Oxalate 350g batch ⁽³⁾			IV Oxalate filtered ⁽⁴⁾		
	Ave.	Max.	Min.	Ave.	Max.	Min.	Ave.	Max.	Min.
Ag	< 2	10	< 1	< 2	5	< 1	< 1	1	< 1
Al	< 28	< 50	< 20	28	50	< 20	< 20*	< 20	< 20
As	< 1*	< 2	< 1	< 1*	< 1	< 1	< 1*	< 1	< 1
Ba	< 17	50	< 5	107	200	20	154	500	20
Be	< 0.4*	< 2	< 0.03	< 0.05*	< 0.08	< 0.03	< 0.07*	< 0.1	< 0.02
Bi	> 768	> 1000	200	375	1000	100	380	500	200
Ca	149	500	30	437	1000	50	120	200	100
Cd	< 91	400	< 50	125	200	50	50	50	50
Ce	< 525	< 2000	< 200	< 207*	< 300	< 130	< 576*	< 1000	< 200
B	15.7	100	1	13	20	2	2	5	1
Cr	< 5.9	20	< 1	9	20	1	31	100	10
Cu	31	200	10	6	10	5	30	100	10
Fe	221	650	65	< 100*	< 100	< 100	269	400	115
Hg	< 2965*	< 12000	< 800	< 1175*	< 1300	< 800	< 2460*	< 4000	< 800
In	< 11*	< 20	< 10	< 7.5*	< 10	< 5	< 10*	< 10	< 10
K	230	1000	20	325	500	100	110	200	50
La	2773	10,000	50	1650	2000	1300	2080	4000	100
Li	< 2.6*	< 16	< 1	< 1*	< 1	< 1	< 1*	< 1	< 1
Mg	63	200	5	117	200	20	60	100	50
Mn	< 9.2	50	< 2	13	20	2	29	100	5
Na	< 145	500	< 25	937	2500	250	400	500	250
Ni	< 78	500	< 20	43	50	20	80	200	50
P	> 468	> 1000	> 100	300	500	100	120	200	100
Pb	43	100	10	57	100	20	60	100	50
Sb	< 104	< 200	50	< 125	200	< 100	< 100*	< 100	< 100
Si	16.6	50	< 5	10	20	5	52	200	10
Sn	< 3.2	20	< 1	< 1*	< 1	< 1	< 1*	< 1	< 1
Sr	< 8.6	< 30	< 2	< 4.3	< 5	< 2	< 9	< 16	3
Tl	< 543*	< 2000	< 130	< 257*	< 300	< 130	< 514*	< 100	< 130
V	< 105*	< 200	< 100	< 100*	< 100	< 100	< 100*	< 100	< 100
Zn	< 55	100	< 50	< 75*	< 100	< 50	< 50*	< 50	< 50

* No positive values

(2) 15 batches

(1) All values expressed as parts per million (ppm) Pu.

(3) 5 batches

(4) 5 batches.

FIGURE II

UNCLASSIFIED

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HW-26450

ABSTRACT

Tetravalent plutonium was precipitated as an oxalate, hydrofluorinated to the tetrafluoride, and reduced to the metal in plant equipment. This process was recommended for regular plant use.