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EFFECTS OF ACID CONCENTRATION AND FEED-PARTICLE SIZE
ON THE DISSOLUTION OF PLUTONIUM-BEARING MATERIALS

THE DOW CHEMICAL COMPANY
ROCKY FLATS DIVISION
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U.S. ATOMIC ENERGY COMMISSION
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G. F. Molen

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Effects of Acid Concentration and Feed-Particle Size on the Dissolution of Plutonium-Bearing Materials

G. F. Molen

Abstract: The dissolution of plutonium-bearing residues in a nitric acid-calcium fluoride solvent system is described. The acid concentration, mesh size, initial solid-to-liquid ratio and initial feed material concentration are evaluated in terms of the effects they have on the dissolution of these residues. The reproducibility of ash dissolution results is also measured.

INTRODUCTION

Many plutonium-bearing materials are generated as a result of producing, fabricating, and machining plutonium metal. These materials include incinerator ash (ash resulting from the incineration of plutonium contaminated burnables), glovebox floor sweepings (plutonium-containing material that is inadvertently dropped on the floor of a glovebox), and reduction slag (slag resulting from the bomb reduction of PuF_4 with calcium metal). To recover the plutonium, the plutonium-bearing materials are dissolved in nitric acid containing calcium fluoride. The plutonium-containing solution is purified by anion exchange. The material is often difficult to dissolve in this solvent system. The residue remaining after dissolution is called heel. Thus there are three types of plutonium-bearing heels which must be considered. They are incinerator ash heel, glovebox floor sweepings heel, and reduction slag heel.

In an earlier study¹ the variables considered were; the length of the reflux period, the drying temperature of heels, the solid-to-liquid ratio, the addition of fluoride during dissolution, and the effectiveness of a multiple contact system. The feed materials used for that study were crushed incinerator ash, incinerator ash heel, glovebox floor sweepings, and glovebox floor sweepings heel.

The purpose of this study was to determine the variables affecting the dissolution of the feed materials.

The feed materials used for this study were crushed reduction slag and crushed incinerator ash. The variables considered were the effects of acid concentration, feed-particle size, initial solid-to-liquid ratio, and initial feed material concentration.

SUMMARY AND CONCLUSIONS

The acid concentration, feed-particle size, reproducibility of results, initial solid-to-liquid ratio and initial feed material concentration were evaluated in terms of the effects they have on the dissolution of plutonium-bearing materials.

Two types of plutonium-bearing materials were used for feed. They were crushed reduction slag and crushed incinerator ash. The effect of nitric acid concentration between 3.0 and 15.0M on the dissolution of these materials was determined. The results showed that acid concentration had little effect on dissolution of crushed reduction slag. However, the dissolution of crushed incinerator ash was affected by acid concentration; maximum plutonium recovery was obtained when 9.0M HNO_3 was used.

The effect of feed-particle size on the dissolution of crushed reduction slag was evaluated by testing unscreened slag, >12-mesh slag, <12->18-mesh slag and <18-mesh slag. The results showed that the greatest plutonium recovery was obtained from small feed-particle sizes. Slag smaller than 35-mesh was too reactive to contain in the reaction vessel. Slag smaller than 18-mesh was considered best for dissolution.

The ability to reproduce incinerator ash dissolution results was measured by repeating a set of experiments five times.

The data for the acid concentrations showed a range of plutonium recovery from 68.2%-83.8% for 6.0M HNO_3 and from 54.3%-96.0% for 15.0M HNO_3 . The results are not considered reproducible, but average values do show a reproducible trend. This inability to reproduce results is undoubtedly because of the heterogeneous nature of crushed incinerator ash.

¹ G. F. Molen and R. O. Wing, "The dissolution of Plutonium-Bearing Residues in a Nitric Acid-Calcium Fluoride Solvent System," The Dow Chemical Company, Rocky Flats Division, RFP-776, November 22, 1966.

A five contact method for dissolution was used to evaluate the effect of initial solid-to-liquid ratio and initial feed material concentration. The results showed that for an initial solid-to-liquid ratio greater than 75 g/l, four or more contacts were required for maximum plutonium recovery regardless of the initial feed material concentration.

RECOMMENDATIONS

The following recommendations from RFP-776 are still considered valid.

1. A multiple contact system consisting of three contacts is recommended. The first contact should be with "used" acid for one hour of refluxing; the second contact should be with fresh acid for a reflux period of 90 minutes; and the third contact should be with fresh acid for one hour of refluxing.
2. An initial solid-to-liquid ratio of 100 g/l for the three contact system is recommended.
3. A calcium fluoride concentration of 0.2M for each contact is recommended.
4. The reflux period for each contact should not exceed 90 minutes.
5. The drying temperature of heels that have to be reprocessed should never exceed 650°C.

The following recommendations are based on this study.

1. Nine molar nitric acid is recommended instead of twelve molar nitric acid.
2. If a multiple contact system cannot be used, a solid-to-liquid ratio of 75 g/l or less, for each dissolution cycle, is recommended.
3. It is recommended that, for any dissolution system, the initial solid-to-liquid ratio be decreased as the initial feed material concentration is increased.
4. When dissolving crushed reduction slag or any similar material, it is recommended that the particle size of the material be less than 18-mesh.

EXPERIMENTAL PROCEDURES

Dissolution of Reduction Slag: Crushed reduction slag (50.0 g) was mixed with 500 ml of nitric acid in a one-liter Florence flask of Pyrex glass which rested on a Thermolyne Stirplate that both heated and stirred (magnetically) the solution. The stirring speed (set on high) was held constant throughout all the experiments. Refluxing conditions (110°-115°C) were maintained for one hour by a water-cooled Liebig condenser. For reduction slags which contained large amounts of sand (SiO_2), the condenser had to be removed periodically and freed of the deposits of silicon dioxide formed by the hydrolysis of the volatile silicon tetrafluoride. At the end of the reflux period (one hour), the hot solution was filtered through a medium-porosity glass filter and the undissolved residue was washed with 200 ml of 0.5M HNO_3 . The undissolved residue was then air dried. The entire residue was accurately weighed, dried at 800°C, reweighed, and analyzed for plutonium content by x-ray fluorescence. The filtrate volume was measured, sampled, and analyzed for plutonium by radio-assay or x-ray fluorescence.

Dissolution of Crushed Incinerator Ash: Crushed incinerator ash (50.0 g) was mixed with 500 ml of nitric acid, containing 0.2M CaF_2 , as described in the dissolution of reduction slag. All other procedures were the same.

Five Contact Method for Dissolution: Crushed incinerator ash was refluxed (110°-115°C) with 500 ml of 12M HNO_3 -0.2M CaF_2 for one-half hour. At the end of the reflux period the hot solution was filtered, the filtrate volume measured, sampled, and analyzed for plutonium. The undissolved residue was quantitatively returned to the reaction flask. Five hundred milliliters of fresh 12M HNO_3 -0.2M CaF_2 were added and the solution was again refluxed for one-half hour. This procedure was repeated until five contacts of the original feed material had been made. After the fifth contact the solution was filtered and the filtrate volume was measured, sampled, and analyzed for plutonium content. The undissolved residue was air dried, accurately weighed, dried at 800°C, reweighed, and the plutonium content determined by x-ray fluorescence.

DISCUSSION OF RESULTS

Effect of Acid Concentration on Dissolution of Reduction Slag: The plutonium concentration in the

feed material was held constant (1.5-1.8% Pu) and the nitric acid concentration was varied from 3M to 15M by intervals of three. The results, which are presented in Table I, show that the dissolution of unscreened reduction slag in nitric acid is little affected by the acid concentration. The average plutonium recoveries varied from 82.4% for 3M HNO₃ to 87.4% for 12M HNO₃.

All undissolved residue concentrations (expressed in % Pu) were less than 0.7% Pu which is the present discard level for material of this category. The solubility of slag was governed by the solubility of the major component, calcium fluoride. Under the experimental conditions used, the slag had a maximum solubility (in one hour) of ~36 g/l in 12M HNO₃.

Effect of Mesh Size on Dissolution of Reduction Slag:

The crushed reduction slag was screened through Tyler equivalent 12- and 18-mesh sieves and divided into three fractions; greater than 12 mesh, less than 12 mesh but greater than 18 mesh, and less than 18 mesh. The different fractions were then dissolved as previously described. These results are tabulated in Table I. The plutonium concentration in the feed materials varied from 1.4% to 2.3%.

The results show that for a given mesh size there was very little difference in plutonium recovery for various concentrations of acid. However, the results show a trend toward increasing plutonium recovery with decreasing mesh size because of the increased surface area. Reduction slag of 35 mesh was so reactive that it could not be contained in the dissolution vessel. This was due to both the heat of reaction and the small amounts of calcium metal which were present.

The data indicate that screening to less than 18-mesh was best for plutonium recovery. Nine-to-twelve molar nitric acid is optimum for total solid solubility (35-39 g/l). The solubility of reduction slag as a function of nitric acid concentration is shown in Figure 1.

Effect of Acid Concentration on Crushed Incinerator Ash Dissolution: The effects of acid concentration on the dissolution of crushed incinerator ash are summarized in Table II and Figure 2. The feed material averaged 5.3 to 5.9% plutonium. The acid concentrations used were 3.0, 6.0, 9.0, 12.0 and 15.0M HNO₃. The results show that 9.0M HNO₃ is optimum for both total solid dissolution and plutonium recovery.

TABLE I. The Effect of Acid Concentration and Mesh Size on The Dissolution of Crushed Reduction Slag.

Solid-to-Liquid Ratio - 100 g/l, Reflux Period - One Hour						
Nitric Acid Conc. (M)	Sieve Size (Mesh) ^a	No. of Runs	Initial Feed Conc. (% Pu)	Plutonium Recovered (%)	Solid Dissolved (%)	Undissolved Residue Conc. (% Pu)
3.0	unscreened	3	1.5	82.4	27	0.4
6.0	unscreened	3	1.6	83.2	32	0.4
9.0	unscreened	3	1.6	83.5	35	0.4
12.0	unscreened	3	1.7	87.4	36	0.3
15.0	unscreened	3	1.8	84.1	31	0.4
3.0	>12	3	1.4	88.7	25	0.2
6.0	>12	3	2.3	74.1	25	0.2
9.0	>12	3	1.4	86.4	36	0.3
12.0	>12	3	1.5	90.7	37	0.2
15.0	>12	3	1.5	87.1	30	0.3
3.0	<12>18	3	1.6	87.6	26	0.3
6.0	<12>18	3	1.8	83.3	30	0.3
9.0	<12>18	3	1.6	88.1	36	0.3
12.0	<12>18	3	1.7	90.8	37	0.2
15.0	<12>18	3	1.7	89.0	30	0.3
3.0	<18	3	1.8	92.0	27	0.2
6.0	<18	3	1.5	92.8	34	0.2
9.0	<18	3	1.9	94.7	36	0.2
12.0	<18	3	1.8	94.6	39	0.2
15.0	<18	3	1.7	96.2	30	0.1

^a Tyler equivalent designations.

FIGURE 1. The Solubility of Crushed Reduction Slag as a Function of Nitric Acid Concentration.

TABLE II. The Effect of Nitric Acid Concentration on The Dissolution of Crushed Incinerator Ash.

Solid-to-Liquid Ratio - 100 g/l, Reflux Period - One Hour, Calcium Fluoride Conc. - 0.2M

Nitric Acid Conc. (M)	No. of Runs	Initial Feed Conc. (% Pu)	Plutonium Recovered (%)	Solid Dissolved (%)	Undissolved Residue Conc. (% Pu)
3.0	2	5.5	42.0	21	4.9
6.0	2	5.3	75.9	34	1.9
9.0	2	5.9	86.2	41	1.3
12.0	2	5.7	81.3	35	1.7
15.0	2	5.8	69.7	31	2.7

Reproducibility of Crushed Incinerator Ash Dissolution

Results: A series of experiments was designed to determine the reproducibility of the dissolution results. Crushed incinerator ash was used and the procedure was the same as previously described. Each experiment was repeated five times (one of the samples for 6.0M HNO₃ was lost). The plutonium concentration in the feed material had a range of 4.8 to 6.5% with a minimum variation of 0.9% Pu and a maximum variation of 1.5% Pu for any given acid concentration. The results are shown in Table III. The plutonium recovered for a given acid concentration has a minimum variation of 15.6% for 6.0M and 9.0M HNO₃, and a maximum variation of 41.7% for

15.0M HNO₃. The solid dissolved for a given acid concentration has a minimum variation of 7% for 12.0M HNO₃ and a maximum variation of 28% for 15.0M HNO₃. From this information it is apparent that the dissolution results are not reproducible. This is due to the heterogeneous nature of the crushed incinerator ash. The average results of the dissolution behavior indicate that 9.0M HNO₃ is the optimum acid concentration.

Effect of Initial Solid-to-Liquid Ratio Using the Five Contact Method for Dissolution: The initial solid-to-liquid ratio for these experiments was varied from 25 to 500 g/l for feed material containing 11.5 to 13.9% Pu.

FIGURE 2. The Effect of Nitric Acid Concentration on the Dissolution of Incinerator Ash.

TABLE III. The Determination of the Ability to Reproduce Crushed Incinerator Ash Dissolution Results.

Solid-to-Liquid Ratio - 100 g/l, Reflux Period - One Hour, Calcium Fluoride Conc. - 0.2M

Nitric Acid Conc. (M)	Initial Feed Conc. (% Pu)	Plutonium Recovered (%)	Solid Dissolved (%)	Undissolved Residue Conc. (% Pu)
3.0	4.8	47.9	18	3.0
3.0	5.3	25.0	32	5.8
3.0	5.5	45.1	18	3.7
3.0	6.1	32.3	29	5.8
3.0	6.3	30.5	29	6.1
Avg.	5.6	36.2	25	4.9
6.0	4.8	73.4	38	2.0
6.0	5.1	68.2	38	2.6
6.0	5.4	79.0	36	1.8
6.0	5.7	83.8	28	1.3
Avg.	5.2	76.1	35	1.9
9.0	5.2	79.1	42	1.9
9.0	5.4	83.6	41	1.5
9.0	6.0	94.7	30	0.4
9.0	6.1	93.5	34	0.6
9.0	6.5	79.2	43	2.3
Avg.	5.1	86.0	38	1.3
12.0	5.5	70.8	37	2.6
12.0	5.6	75.3	30	2.2
12.0	5.6	77.4	41	2.1
12.0	5.9	88.9	34	1.0
12.0	6.4	93.1	34	0.6
Avg.	5.8	81.1	37	1.7
15.0	4.9	63.3	53	2.8
15.0	5.1	77.2	57	2.7
15.0	5.6	54.3	33	3.8
15.0	6.3	58.7	31	3.8
15.0	6.5	96.0	29	0.4
Avg.	5.7	69.9	41	2.7

The results are presented in Table IV. For the same plutonium concentration in the feed material (~12-14%), an increase in the solid-to-liquid ratio from 25 to 500 g/l decreases the percentage of plutonium recovered after one contact by 67%. After two contacts the decrease is 44%, and for the third, fourth and fifth contacts the decrease is 27%, 13%, and 10% respectively. The decrease in plutonium recovery resulting from increasing the initial solid-to-liquid ratio can be

minimized by increasing the number of contacts. The effect of the initial solid-to-liquid ratio on the total percent of dissolved solid after five contacts is illustrated in Figure 3. This curve shows that ratios greater than 75 g/l result in a decreasing total percent of dissolved solid.

Table V shows the data for the percent of remaining plutonium recovered per contact. The plutonium

TABLE IV. The Effect of the Solid-to-Liquid Ratio on the Cumulative Plutonium Recovered Using the Five Contact Method for the Dissolution of Crushed Incinerator Ash.

Reflux Period Per Contact - One-Half Hour, Feed Material - Crushed Incinerator Ash
Calcium Fluoride Conc. - 0.2M per Contact, Nitric Acid Conc. - ~12.5M Per Contact

Initial Solid-to- Liquid Ratio (g/l)	Initial Feed Material Conc. (% Pu)	No. of Runs	Cumulative Plutonium Recovered (%)					Solid Dissolved (%)	Undissolved Residue Conc. (% Pu)
			Cont. 1	Cont. 2	Cont. 3	Cont. 4	Cont. 5		
25	13.9	3	87.9	96.0	97.1	97.7	97.7	42	0.5
50	13.2	3	69.2	90.4	94.6	96.8	98.2	58	1.0
75	13.6	3	72.3	93.4	97.8	99.2	99.6	93	0.8
100	13.1	3	60.2	89.0	96.5	99.2	99.8	85	0.2
150	12.6	3	57.6	84.7	94.3	97.3	98.8	64	0.4
200	11.6	3	40.5	63.3	80.5	84.6	87.4	57	3.4
400	11.5	3	34.4	60.7	75.0	82.1	88.3	51	2.7
500	12.2	3	27.3	53.9	70.9	84.8	87.4	46	2.8

FIGURE 3. The Effect of the Initial Solid-to-Liquid Ratio on the Total Percent of Dissolved Solid Using the Five Contact Method for the Dissolution of Crushed Incinerator Ash.

TABLE V. The Effect of Solid-to-Liquid Ratio on the Plutonium Recovered per Contact Using the Five Contact Method for the Dissolution of Crushed Incinerator Ash.

Reflux Period per Contact — One-Half Hour, Feed Material — Crushed Incinerator Ash
Calcium Fluoride Conc. — 0.2M per Contact, Nitric Acid Conc. — 12.5M per Contact

Initial Solid-to- Liquid Ratio (g/l)	Initial Feed Material Conc. (% Pu)	No. of Runs	Plutonium Recovered (%) per Contact				
			Contact 1	Contact 2	Contact 3	Contact 4	Contact 5
25	13.9	3	88	67	28	20	0
50	13.2	3	69	70	46	34	40
75	13.6	3	72	76	68	64	50
100	13.1	3	60	72	68	78	80
150	12.6	3	58	64	62	54	56
200	11.6	3	40	38	47	21	18
400	11.5	3	34	40	36	28	35
500	12.2	3	27	36	37	48	17

TABLE VI. The Effect of the Feed Material Concentration and the Solid-to-Liquid Ratio on the Cumulative Plutonium Recovered Using the Five Contact Method for Dissolution of Crushed Incinerator Ash.

Reflux Period — One-Half Hour per Contact, Feed Material — Crushed Incinerator Ash
Calcium Fluoride Conc. — 0.2M per Contact, Nitric Acid Conc. — 12.5M per Contact

Initial Solid-to- Liquid Ratio (g/l)	Initial Feed Material Conc. (% Pu)	No. of Runs	Cumulative Plutonium Recovered (%)					Solid Dissolved (%)	Undissolved Residue Conc. (% Pu)
			Cont. 1	Cont. 2	Cont. 3	Cont. 4	Cont. 5		
25	13.9	3	87.9	96.0	97.1	97.7	97.7	42	0.5
25	27.4	3	45.9	90.6	98.2	99.1	99.7	82	0.4
50	10.2	3	64.4	87.1	92.2	96.1	98.4	34	0.2
50	16.1	3	73.9	93.8	97.0	97.5	98.0	83	1.9
75	13.6	3	72.3	93.4	97.8	99.2	99.6	93	0.8
75	37.7	3	62.2	86.6	95.5	98.0	98.7	69	1.5
100	5.7	3	53.2	71.8	83.8	90.8	95.4	83	1.5
100	18.4	3	47.3	80.3	93.7	98.5	99.2	73	0.5
200	4.5	3	40.7	69.9	77.4	84.5	90.3	76	1.8
200	11.6	3	40.5	63.3	80.5	84.6	87.4	57	3.4
400	6.2	3	42.6	63.4	74.0	80.9	86.4	53	1.8
400	11.5	3	34.4	60.7	75.0	82.1	88.3	51	2.7

recovered per contact for a given solid-to-liquid ratio does not follow a pattern. The optimum number of contacts is defined as that contact number in which the plutonium recovery is maximum. The data show that a different number of contacts is optimum for different solid-to-liquid ratios. The data for ratios from 150-500 g/l is too erratic to establish the optimum number of contacts. Probably the optimum number of contacts for ratios greater than 150 g/l is greater than five.

Effect of Initial Feed Material Concentration Using the Five Contact Method for Dissolution: The effects

of the plutonium concentration in the feed material and the initial solid-to-liquid ratio on plutonium recovery are tabulated in Table VI. These results show that the plutonium concentration in the feed material for solid-to-liquid ratios of 25 to 100 g/l is not as important as the solid-to-liquid ratio unless the initial feed concentration is greater than 20% plutonium. For solid-to-liquid ratios ≥ 200 g/l the plutonium concentration in the feed material is not as important as the solid-to-liquid ratio.

Tabulating (see Table VII) the amount of remaining plutonium recovered per contact for these same

experiments shows the optimum number of contacts required as a function of solid-to-liquid ratio and feed material concentrations.

The general trends indicated by the results of these experiments are presented in Table VIII. These

values may be used as guidelines in determining optimum conditions (i.e. solid-to-liquid ratio and number of contacts for a given plutonium concentration in the feed material) for dissolving crushed incinerator ash. These numbers are not meant to be absolute and variations to this behavior are to be expected.

TABLE VII. The Effect of the Feed Material Concentration and the Solid-to-Liquid Ratio on the Plutonium Recovered per Contact Using the Five Contact Method for Dissolution of Crushed Incinerator Ash.

Reflux Period – One-Half Hour per Contact, Feed Material – Crushed Incinerator Ash
Calcium Fluoride Conc. – 0.2M per Contact, Nitric Acid Conc. – 12.5M per Contact

Initial Solid-to- Liquid Ratio (g/l)	Initial Feed Material Conc. (% Pu)	No. of Runs	Plutonium Recovered per Contact (%)				
			Contact 1	Contact 2	Contact 3	Contact 4	Contact 5
25	13.9	3	88	67	28	20	0
25	27.4	3	46	83	81	50	67
50	10.2	3	64	64	39	50	60
50	16.1	3	74	76	52	17	20
75	13.6	3	72	76	68	64	50
75	37.7	3	62	64	66	56	36
100	5.7	3	53	40	42	43	50
100	18.4	3	47	63	68	76	50
200	4.5	3	41	49	25	31	37
200	11.6	3	40	38	47	21	18
400	6.2	3	43	36	29	25	28
400	11.5	3	34	40	36	28	35

TABLE VIII. The Recommended Number of Contacts for the Dissolution of Crushed Incinerator Ash as a Function of the Initial Feed Material Concentration and the Initial Solid-to-Liquid Ratio.

Initial Feed Material Concentration (% Pu)	Initial Solid to Liquid Ratio (g/l)	Optimum Number of Contacts	Initial Feed Material Concentration (% Pu)	Initial Solid to Liquid Ratio (g/l)	Optimum Number of Contacts
≤5	25	1	>15≤25	25	2
≤5	50	1	>15≤25	50	2
≤5	75	5	>15≤25	75	2
≤5	100	>5	>15≤25	100	4
≤5	200	>5	>15≤25	200	>5
≤5	400	>5	>15≤25	400	>5
>5≤10	25	1	>25≤40	25	3
>5≤10	50	1	>25≤40	50	3
>5≤10	75	1	>25≤40	75	4
>5≤10	100	>5	>25≤40	100	>5
>5≤10	200	>5	>25≤40	200	>5
>5≤10	400	>5	>25≤40	400	>5
>10≤15	25	1	>40	25	3
>10≤15	50	2	>40	50	3
>10≤15	75	2	>40	75	4
>10≤15	100	>5	>40	100	>5
>10≤15	200	>5	>40	200	>5
>10≤15	400	>5	>40	400	>5