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Decontamination of HEPA Filters: July-September 1977

Don F. Luthy and Edward L. Lewis

February 20, 1978

Monsanto

MOUND FACILITY
Miamisburg, Ohio

operated by
MONSANTO RESEARCH CORPORATION
a subsidiary of Monsanto Company

for the
U. S. DEPARTMENT OF ENERGY

Contract No. EY-76-C-04-0053

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Price: Printed Copy $4.00; Microfiche $3.00

MLM-2491
UC-70

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Contents

Abstract

Dissolution parameters (in various reagents) of americium-241 and plutonium-239 oxide mixtures, uranium-233 oxide, neptunium-237 oxide, curium-244 oxide, thorium-232 oxide, and plutonium dioxide were determined. The reagents used were various concentrations of HNO_3-HF-H_2SO_4, HNO_3-$(NH_4)_2Ce(NO_3)_6$, HNO_3-HF, and HNO_3. Both simulated contaminated HEPA filter media and actual glovebox filter media from spent filters were used. The maximum decontamination factor achieved was 833 using a six-stage dissolution process. Also, plutonium dioxide was fused with sodium carbonate at various elevated temperatures, and a dissolution percentage was determined.

Experimental

I. Americium-241 and plutonium-239[*] mixed oxide dissolution studies

Contaminated filter media were prepared by mixing AmO_2-PuO_2 powder with shredded filter media. Small samples of this mixture were then treated with various leaching agents, including 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$, 12N HNO_3-0.05N HF-0.01N H_2SO_4, and 8N HNO_3. A sample of filter media was placed in a beaker, 250 ml of the leaching agent was added, and the temperature was increased to the boiling point of the solution. Samples were withdrawn periodically and filtered through a 4-5 µm glass filter. The americium-241 and plutonium-239 concentrations were determined by alpha spectrometry. The solution volume and concentration were kept constant by adding acid of the proper concentration to replace evaporated acid. As can be seen in Table 1, the 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$ was an effective leaching agent, dissolving greater than 90% of both AmO_2 and PuO_2 in 11-3/4 hr. On the other hand, the 8N HNO_3 was not a satisfactory leaching agent since only 36% of the PuO_2 dissolved in 13-1/2 hr, although 97% of the AmO_2 dissolved during this time (see Figure 1). The mixture of HNO_3-HF-H_2SO_4 was effective in dissolving the AmO_2 (90% in 11-1/4 hr) but not as effective in dissolving the PuO_2 (80% in 11-1/4 hr). Of the three reagents tested, the 4N HNO_3-0.1M $(NH_4)Ce(NO_3)_6$ is the recommended leaching agent for mixtures of AmO_2-PuO_2.

II. Uranium-233 oxide dissolution studies

These studies were conducted using U_3O_8 mixed with filter media. The oxide had been fired at 950°C for 2 hr. A small sample of the prepared filter media (approximately 3.2 g) was placed into a beaker containing 250 ml of boiling reagent. The reagents used were 8N HNO_3, 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$, and 12N HNO_3-0.05N HF-0.01N H_2SO_4. Samples were withdrawn periodically for alpha analysis. All samples were filtered through 4-5 µm glass filters. The solution volume and concentration were kept constant by adding acid of the proper concentration to replace evaporated acid. As can be seen from

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[*] This material was approximately three parts plutonium-239 oxide mixed with one part americium-241 oxide by weight. It had been fired for 2 hr at 1000°C.

Table 1 - AmO_2-PuO_2 DISSOLUTION TESTS

Acid Test Number	Reagent Composition	Time Heated (hr)	PuO_2 Dissolved (wt%)	AmO_2 Dissolved (wt%)
Am-Pu-1A	4N HNO_3-0.1M CAN[a]	5 3/4	72.9	63.2
Am-Pu-1B	4N HNO_3-0.1M CAN[a]	11 3/4	93.7	111.8
Am-Pu-2A	12N HNO_3-0.05N HF-0.01N H_2SO_4	5 3/4	68.9	72.4
Am-Pu-2B	"	11 1/4	80.2	90.4
Am-Pu-4A	8N HNO_3	7 1/4	37.2	88.9
Am-Pu-4B	"	13 1/2	36.3	96.7

[a] CAN is an acronym for ceric ammonium nitrate.

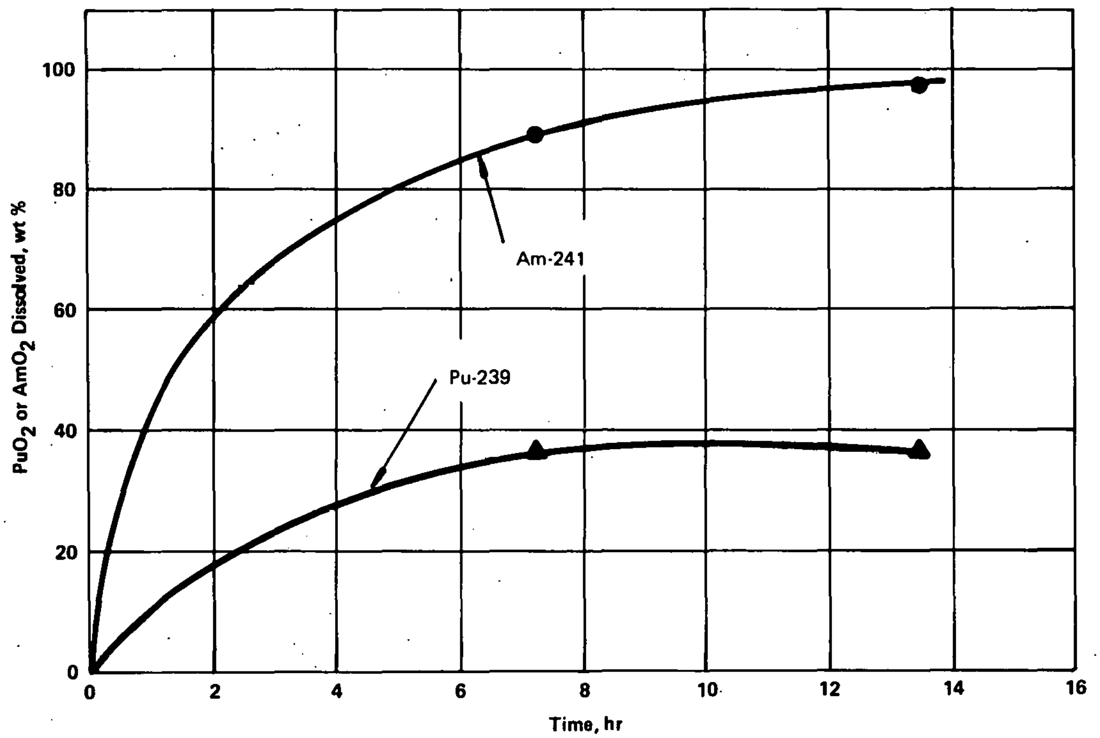

FIGURE 1 - Dissolution rate of Am^{241}-Pu^{239} Oxides in 8N HNO_3

Table 2, in all three cases the dissolution was very rapid and exceeded 90%. For example, the dissolution rate in 8N HNO_3 is depicted in Figure 2. All three reagents would be acceptable dissolution reagents for uranium-233 oxide.

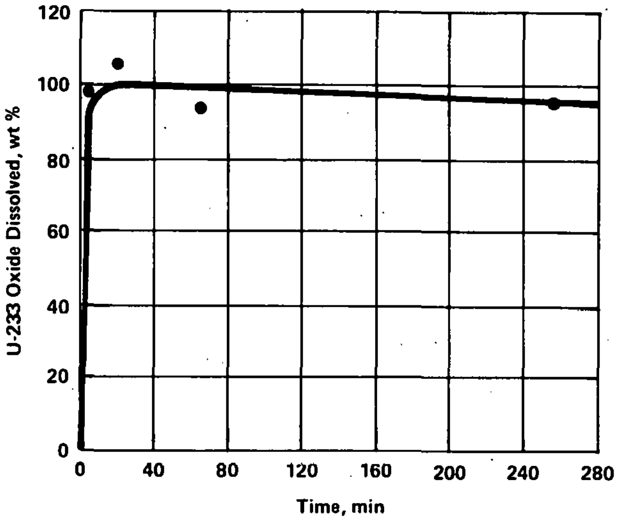

FIGURE 2 - Dissolution rate of U_3O_8 in 8N HNO_3.

III. Sodium carbonate fusion of PuO_2*

These studies were conducted in order to determine the effect of temperature on fusion efficiency. Small samples were prepared by mixing 64 mg of PuO_2 with 2.25 g of Na_2CO_3. The PuO_2-Na_2CO_3 mixture was then placed into a platinum crucible and contents were then heated to

Table 2 - U-233 OXIDE DISSOLUTION TESTS

Acid Test Number	Reagent Composition	Time Heated (min)	U-233 Oxide Dissolved (wt%)
U-3-1	8N HNO_3	5	97.4
U-3-2	"	20	106.1
U-3-3	"	65	93.8
U-3-4	"	255	95.9
U-4-1	4N HNO_3-0.1M CAN[a]	5	89.7
U-4-2	"	15	104.8
U-4-3	"	60	95.2
U-4-4	"	360	95.7
U-5-1	12N HNO_3-0.05N HF-0.01N H_2SO_4	5	100.9
U-5-2	"	15	106.2
U-5-3	"	50	100.1
U-5-4	"	240	102.5

[a] CAN is an acronym for ceric ammonium nitrate.

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*The PuO_2 was fine powder having a composition of 80 wt% Pu-238, 16% Pu-239, 2.5% Pu-240, 0.8% Pu-241, 0.2% Pu-242, plus small amounts of other actinides. It was calcined for 2 hr at 950°C.

the desired operating temperature (1200 and 1300°C) and maintained there for 1 hr. The crucible and melt were cooled to room temperature, and the melt was removed from the crucible and dissolved in 4N HNO_3. As can be seen in Table 3, there is not a significant difference (4.6%) in the two dissolution percentages and it is probable that the difference is due to experimental error. Therefore, it can be concluded that within the temperature range under investigation, the fusion efficiency is approximately 58% (average value).

IV. Neptunium-237 oxide dissolution studies

The NpO_2 was fired for 2 hr at 950°C. Samples of contaminated filter media were prepared by mixing 0.2 g of NpO_2 with 3 g of filter media. The procedure was identical to that used in the uranium-233 oxide dissolution studies (Section II). Three leaching reagents were tested, 8N HNO_3, 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$, and 12N HNO_3-0.05N HF-0.01N H_2SO_4. Table 4 lists the results from these experiments. As can

Table 3 - Na_2CO_3 FUSIONS OF PuO_2

Fusion Test Number	Time Heated (hr)	Operating Temperature (°C)	PuO_2 Solubilized (wt%)	Dissolution Reagent
Pu-44	1	1300°C	56.4	4N HNO_3
Pu-45	1	1200°C	61.0	4N HNO_3

Table 4 Np-237 OXIDE DISSOLUTION TESTS

Acid Test Number	Reagent Composition	Time Heated (min)	NpO_2 Dissolved (wt%)
Np-3-1	8N HNO_3	10	1.3
Np-3-2	"	25	1.9
Np-3-3	"	45	4.1
Np-3-4	"	75	6.1
Np-3-5	"	480	32.3
Np-4-1	4N HNO_3-0.1M CAN[a]	10	93.7
Np-4-2	"	25	97.2
Np-4-3	"	40	88.9
Np-4-4	"	60	89.1
Np-5-1	12N HNO_3-0.05N HF-0.01N H_2SO_4	10	64.7
Np-5-2	"	25	105.2
Np-5-3	"	45	107.4
Np-5-4	"	100	102.3

[a] CAN is an acronym for ceric ammonium nitrate.

be seen, the 8N HNO_3 was unsuccessful in dissolving the NpO_2 rapidly with only 32% solubilized in 8 hr. On the other hand, each of the other two reagents dissolved the NpO_2 rapidly with greater than 90% dissolved after 1/2 hr of boiling (see Figure 3 for dissolution percentage as a function of time). Therefore, both the 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$ and the 12N HNO_3-0.05N HF-0.01N H_2SO_4 acid mixtures would be recommended for dissolution of NpO_2 from filter media.

V. Curium-244 oxide* dissolution studies

Contaminated filter media were prepared by mixing 13 mg of CmO_2 with 3 g of filter media. The experimental procedures were identical to those used for the uranium-233 dissolution studies (Section II). Two leaching reagents were tested, 12N HNO_3-0.05N HF and 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$. The results from these tests are given in Table 5. As can be seen, both reagents dissolved the CmO_2 very rapidly (essentially 100% dissolution in 10 min). Figure 4 shows the dissolution rate for 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$. Both reagents are recommended for dissolution of CmO_2 from contaminated filter media.

VI. Thorium-232 oxide dissolution tests

Contaminated filter media were prepared by mixing 0.24 g of ThO_2 with 3 g of filter media. The experimental procedure was identical to that used in the uranium-233 oxide studies (Section II). The ThO_2 was a fine powder which had been fired at 600°C. The purpose of the experiment was to determine if ThO_2 would dissolve in 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$ similarly to other actinide oxides previously tested (PuO_2, CmO_2, AmO_2, U_3O_8, and NpO_2). As can be seen from the data in Table 6 and Figure 5, the dissolution rate was slow at first (0-7 hr), rapid from 7 to 9 hr, and slow again from 9 to 20 hr. The reason for this is not known at this time, but it is thought that it took 7 hr to attack the surface of the ThO_2 and open the pores so that rapid dissolution could begin. It therefore appears that 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$ would be an acceptable leaching agent for ThO_2 although dissolution time is longer than for other actinides. A point of interest is that in a similar experiment using just ThO_2 (no filter media), the dissolution percentage obtained was only 25% after 20 hr of boiling. The reason for the difference in dissolution percentages (25% versus 100%) is not known at this time.

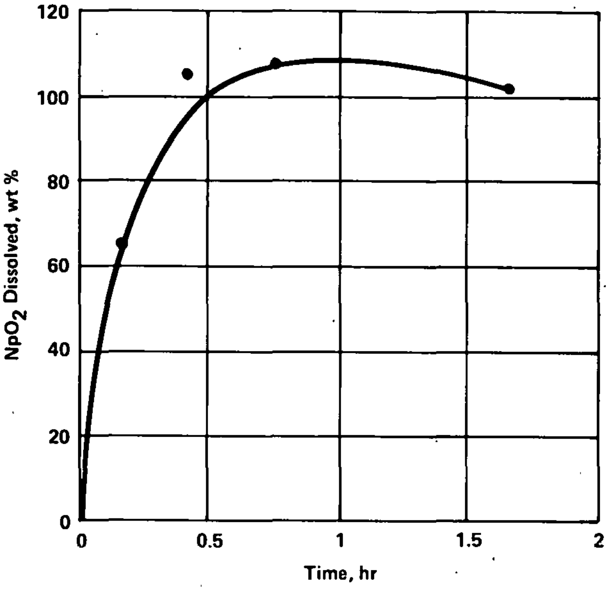

FIGURE 3 - Dissolution Rate of Np-237 Oxide in 12N HNO_3-0.05N HF-0.01N H_2SO_4.

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*This was a powder which was approximately 94% Cm-244, 4% Cm-246, and the remainder Cm-245, Cm-247, and Cm-248.

Table 5 - Cm-244 OXIDE DISSOLUTION TESTS

Acid Test Number	Reagent Composition	Time Heated (min)	CmO_2 Dissolved (wt%)
Cm-2A	12N HNO_3-0.05N HF	10	103.3
Cm-2B	"	25	100.8
Cm-2C	"	60	105.0
Cm-2D	"	120	99.1
Cm-2E	"	180	104.5
Cm-2F	"	540	93.3
Cm-2G	"	540	99.0
Cm-3A	4N HNO_3-0.1M CAN[a]	10	112.1
Cm-3B	"	25	110.0
Cm-3C	"	60	109.0
Cm-3D	"	120	107.6
Cm-3E	"	330	110.5

[a] CAN is an acronym for ceric ammonium nitrate.

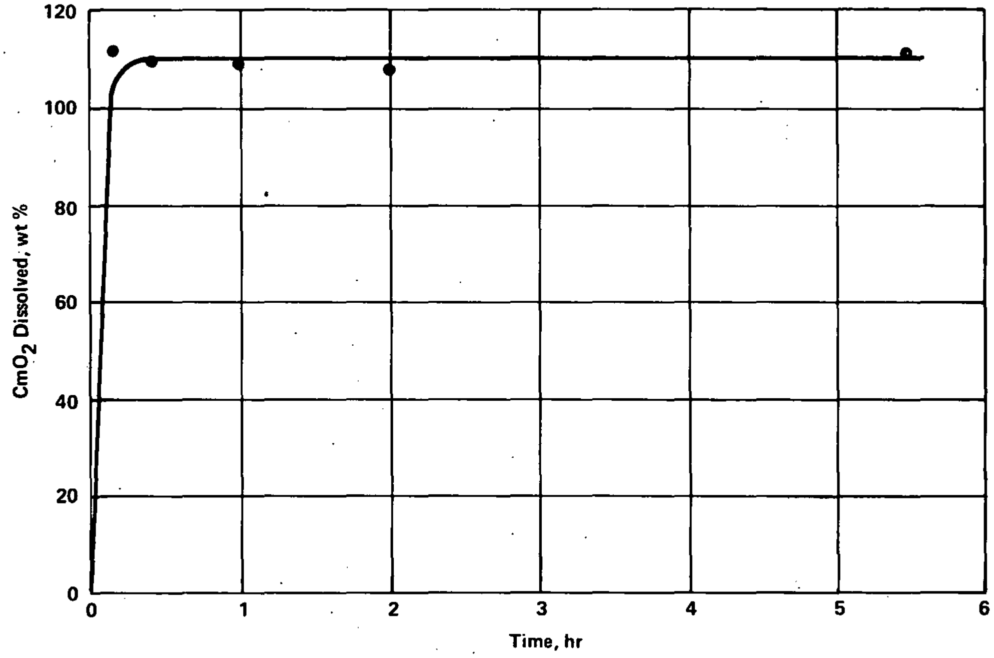

FIGURE 4 - Dissolution rate of CmO_2 in 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$.

Table 6 - ThO_2 DISSOLUTION DATA

Acid Test Number	Reagent Composition	Time Heated (min)	ThO_2 Dissolved (wt%)
Th-1	4N HNO_3-0.1M CAN[a]	5	0.6
Th-2	"	10	0.4
Th-3	"	20	1.4
Th-4	"	35	2.4
Th-5	"	60	5.1
Th-6	"	180	4.7
Th-7	"	300	12.5
Th-8	"	440	31.7
Th-9	"	440	29.3
Th-10	"	470	42.1
Th-11	"	545	82.3
Th-12	"	905	100.5
Th-13	"	1205	104.3

[a] CAN is an acronym for ceric ammonium nitrate.

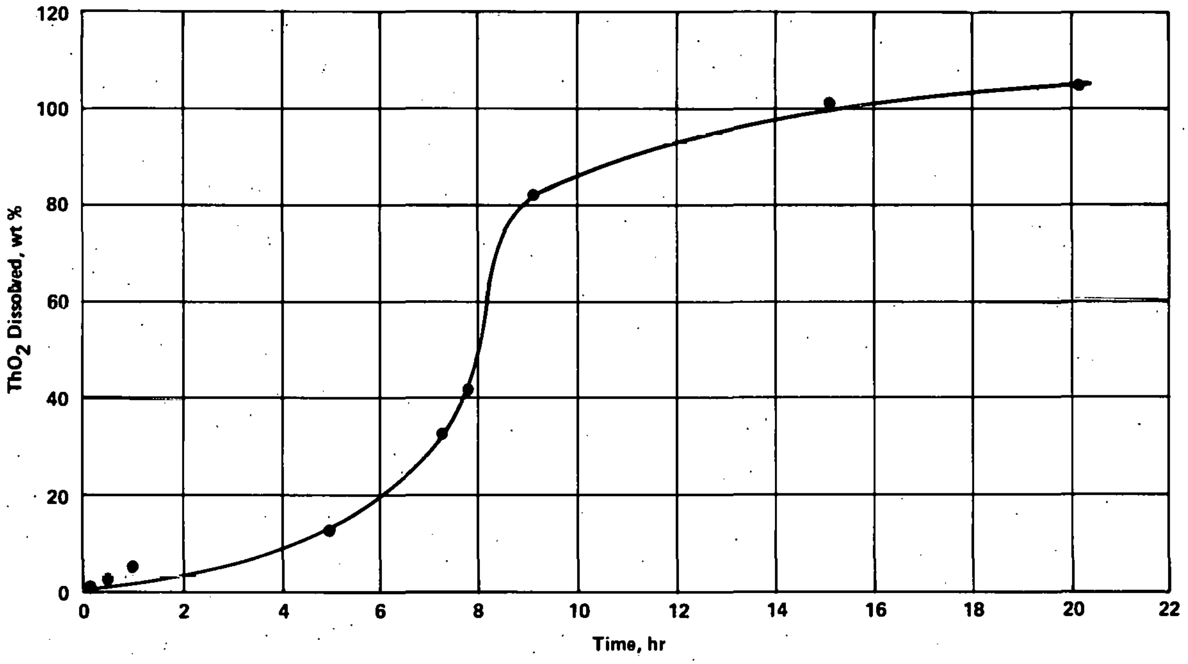

FIGURE 5 - Wt % ThO_2 dissolved in 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$.

VII. Dissolution of PuO_2 from actual glovebox filters

These tests were conducted using samples of filter media removed from actual spent glovebox filters. The PuO_2 on the filters was the same type as that used in the fusion experiments (Section III).

EXPERIMENT A

A 9.5 g sample of filter media containing 0.18 g of PuO_2 was dissolved in a six-stage dissolution procedure. The leaching solution volume was based on the flowsheet submitted last quarter [1], namely, 18 ml of solution per gram of filter media. The ceric ammonium nitrate required was based on the initial moles of PuO_2 present in the filter media (60 moles Ce^{+4} per mole PuO_2). Using these criteria, the leaching reagent in the first five stages was 4N HNO_3-0.23M $(NH_4)_2Ce(NO_3)_6$, and the sixth stage contained 12N HNO_3-0.1N HF. The 12N HNO_3-0.1N HF was used because the 4N HNO_3-0.23M $(NH_4)_2Ce(NO_3)_6$ had proved ineffective in the fifth stage of dissolution. The volume of 12N HNO_3-0.1N HF used was 250 ml, and all studies were done at boiling temperature. The solution was sampled periodically for plutonium concentration, and all samples were filtered through 4-5 μm glass filters. When the dissolution rate for a particular stage had become very slow (as indicated by α analysis of solution), the filter media were separated from the leaching solution by filtration and placed in the next stage of dissolution. Results for this experiment are given in Table 7. Figures 6 through 8 show the dissolution rate for each stage, and Figure 9 shows the cumulative PuO_2 dissolution percentage for the six-stage process. It should be noted that final dissolution percentage was 99.88% (a contamination factor of 833) This was determined by gamma ray spectrometry of the residual filter media and agrees well with the final value as determined by alpha counting of the solution samples (99.80%). It should also be noted that the 12N HNO_3-0.1N HF solution used in the sixth stage was more than twice as effective as the 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$ used in the fifth stage (0.079% dissolved versus 0.031%). The reason for this is not understood at this time. More studies will be done to investigate this phenomenon.

EXPERIMENT B

A 25.6 g sample of filter media containing 0.31 g of PuO_2 was dissolved in a four-stage dissolution process. The leaching reagent used in the test was 4N HNO_3-0.1M $(NH_4)_2$ $Ce(NO_3)_6$. The solution volume was again based on the flowsheet (18 ml of solution per gram of filter media), and the amount of ceric ammonium nitrate was increased to 120 times the initial moles PuO_2 present in the filter media. The experimental procedure was identical to that used in Experiment A of this section. The results for this experiment are given in Table 8; Figure 10 shows the cumulative PuO_2 dissolution percentage for the four stage process. The dissolution percentages in Table 8 were determined by alpha counting, but the cumulative percentages used in Figure 10 were based on a final gamma spectrometry value of 98.24%. As one can see, this value compares well with the value of 97.62% obtained by alpha counting. The gamma value is, however, considered more accurate than the total obtained by simple addition of the individual alpha count values. The decontamination factor obtained using this gamma value is 55.8. The decontamination factor obtained after four stages in Experiment A of this section

Table 7 - PuO_2 DISSOLUTION DATA (9.5-g sample)

Acid Test Number	Reagent Composition	Time Heated (min)	PuO_2 Dissolved (wt%)	Process Stage
256-1 A	4N HNO_3-0.23M CAN[a]	10	82.0	1
256-2 A	"	20	79.5	1
256-3 A	"	40	87.3	1
256-4 A	"	60	80.9	1
256-5 A	"	120	85.8	1
256-6 A	"	210	90.6	1
256-1 B	"	10	0.41	2
256-2 B	"	20	1.17	2
256-3 B	"	45	2.50	2
256-4 B	"	65	2.97	2
256-5 B	"	120	4.75	2
256-6 B	"	360	7.30	2
256-7 B	"	510	7.47	2
256-1 C	"	30	0.29	3
256-2 C	"	75	0.55	3
256-3 C	"	135	0.87	3
256-4 C	"	315	1.06	3
256-5 C	"	435	1.09	3
256-6 C	"	675	1.31	3
256-7 C	"	675	1.39	3
256-1 D	"	20	0.029	4
256-2 D	"	45	0.063	4
256-3 D	"	60	0.079	4
256-4 D	"	135	0.139	4
256-5 D	"	330	0.213	4
256-6 D	"	645	0.222	4
256-7 D	"	645	0.228	4
256-1 E	"	15	0.011	5
256-2 E	"	30	0.012	5
256-3 E	"	60	0.015	5
256-4 E	"	135	0.023	5
256-5 E	"	195	0.025	5
256-6 E	"	465	0.029	5
256-7 E	"	600	0.031	5
256-8 E	"	780	0.032	5
256-9 E	"	780	0.025	5
256-1 F	12N HNO_3-0.1N HF	30	0.016	6
256-2 F	"	60	0.030	6
256-3 F	"	240	0.047	6
256-4 F	"	585	0.079	6
256-5 F	"	945	0.075	6

[a] CAN is an acronym for $(NH_4)_2Ce(NO_3)_6$.

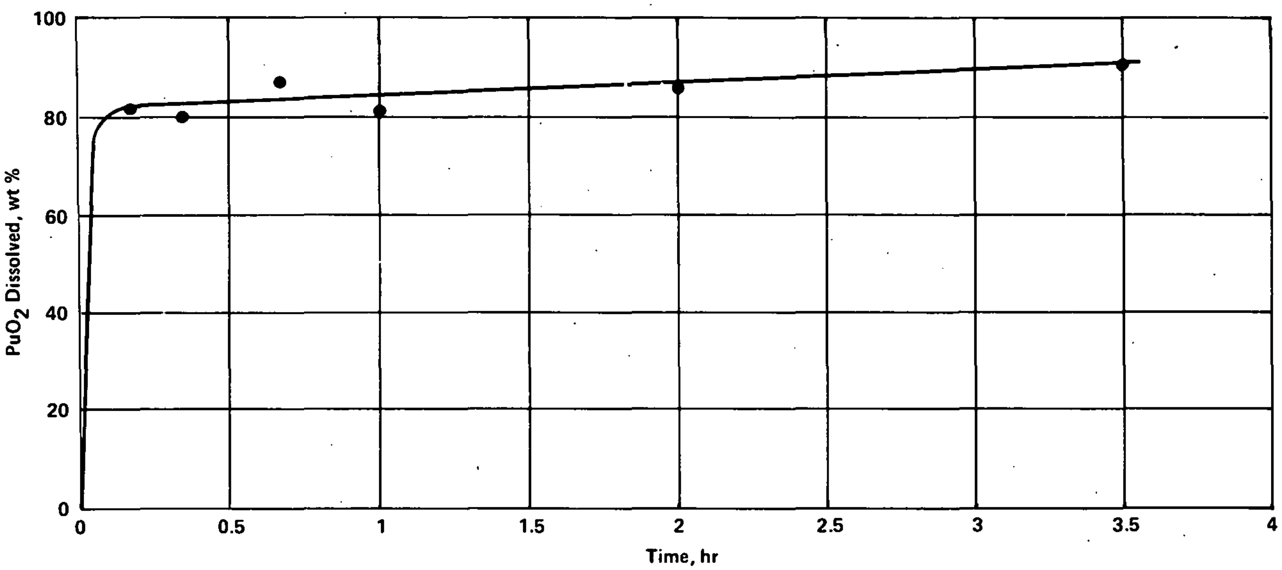

FIGURE 6 - Dissolution rate of PuO_2 from filter media (Ist stage).

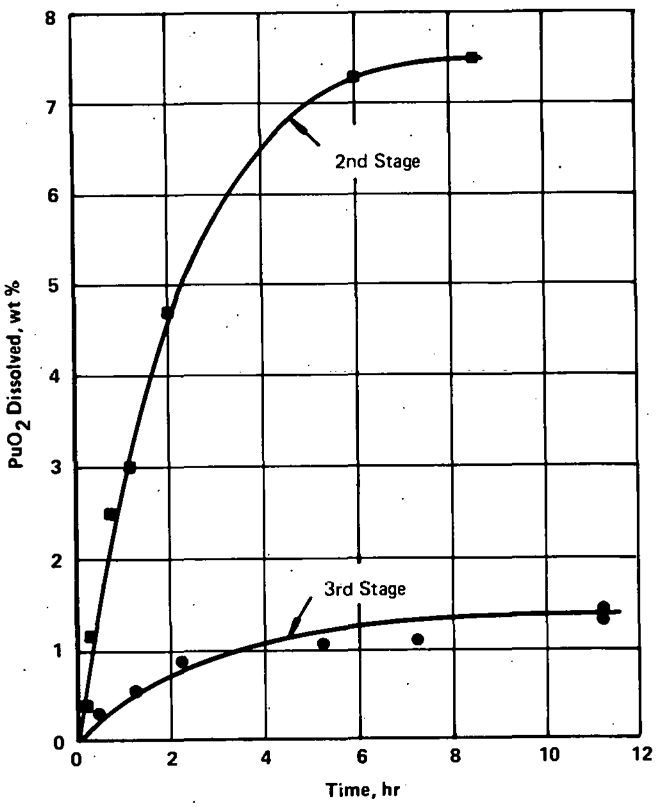

FIGURE 7 - Dissolution rate of PuO_2 from filter media (2nd and 3rd stage).

was 417. A logical question would be: Why was the decontamination factor less when more ceric ammonium nitrate per mole of PuO_2 was used? It is known that the 25.6-g sample of filter media contained a greater percentage of pack-to-frame sealant* than the 9.5-g sample. It is also known (from the color change of the leaching solution) that the Ce^{+4} ions were reduced much more quickly during the dissolution of PuO_2 from the 25.6-g filter media sample. Therefore, it is surmised that the reason for the smaller decontamination factor (smaller percent dissolved) is that the Ce^{+4} ions were mainly used to oxidize the sealant thus leaving

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*This is the material that is used to seal the asbestos-glass filter pack to the wooden frame. It is a fire retardant polyurethane foam and a rubber base adhesive.

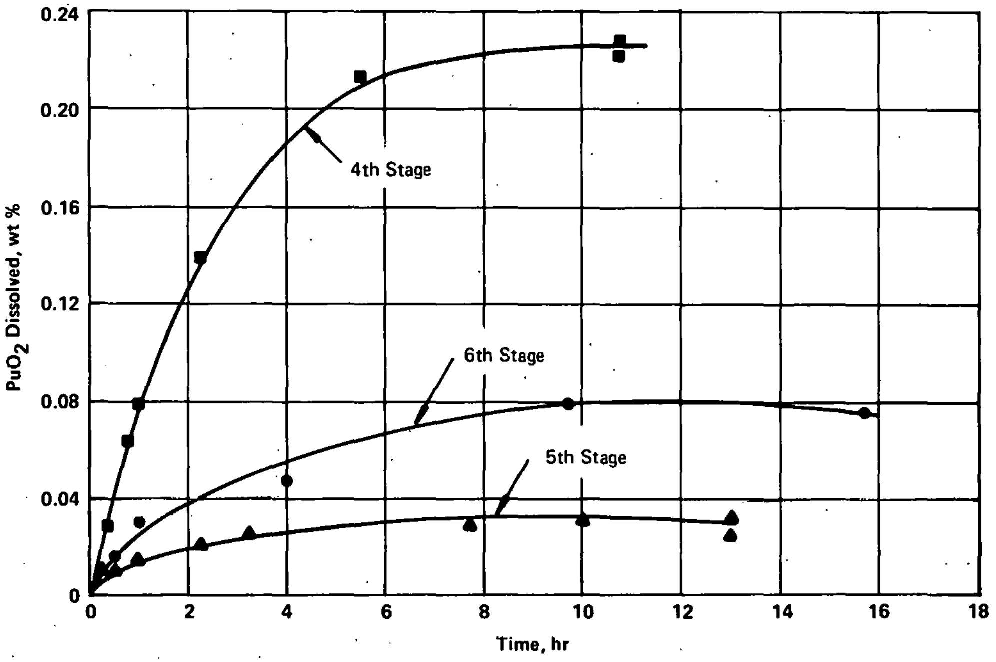

FIGURE 8 - Dissolution of PuO_2 from filter media (4th, 5th, and 6th stages).

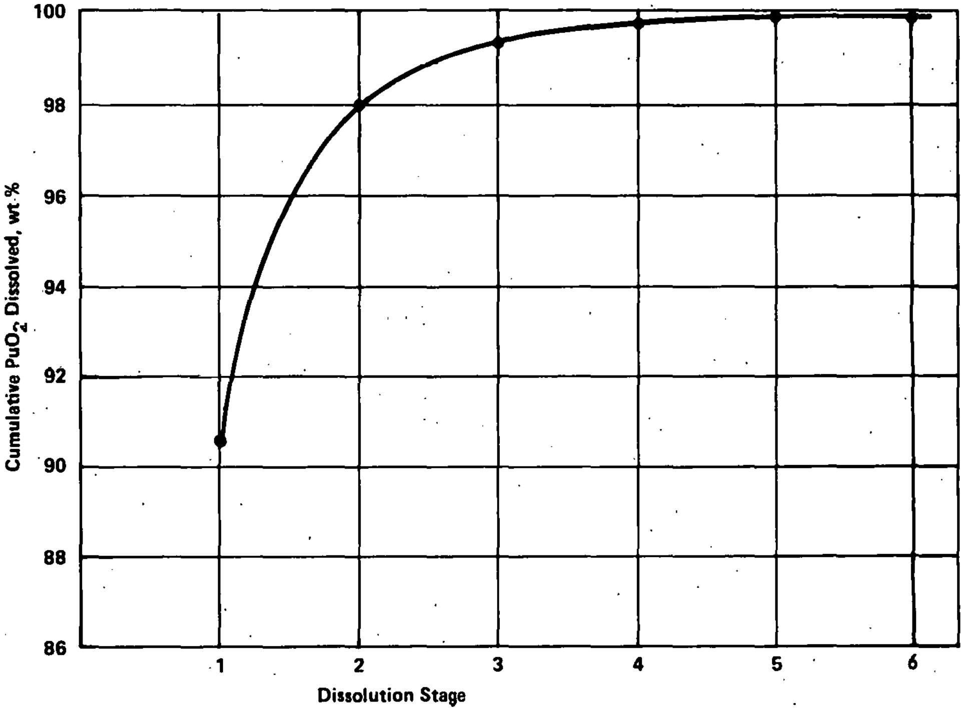

FIGURE 9 - Cumulative weight % PuO_2 dissolved during six stage process.

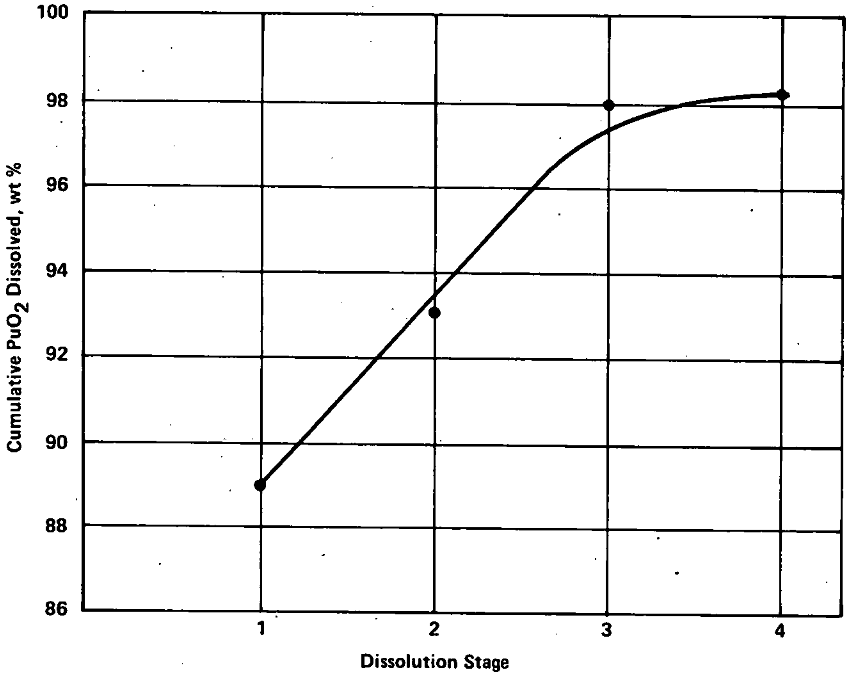

FIGURE 10 - Cumulative weight % PuO_2 dissolved during four stage process.

Table 8 - PuO_2 DISSOLUTION DATA (25.6-g sample)

Acid Test Number	Reagent Composition	Stage	Time Heated (hr)	PuO_2 Dissolved (wt%)	Cumulative PuO_2 Dissolved (wt%)
12-1	4N HNO_3-0.3M CAN[a]	1	6 1/2	88.40	88.40
12-2	"	2	15	4.04	92.44
12-3	"	3	7 1/4	4.91	97.35
12-4	"	4	7	0.27	97.62[b]

[a] CAN is an acronym for ceric ammonium nitrate.

[b] Gamma ray analysis of remaining filter media indicated that 98.24% of the PuO_2 had been dissolved.

fewer Ce^{+4} ions to oxidize to Pu^{+4}. A simple solution to this problem might be to increase the amount of ceric ammonium nitrate when larger amounts of sealant are present with the filter media.

EXPERIMENT C

A 30-g sample of filter media containing 0.33 g PuO_2 was dissolved in a four-stage dissolution process as shown on the flowsheet submitted last quarter [1]. The amount of ceric ammonium nitrate required for each step was based on the assumption that 0.01 mole Ce^{+4} would oxidize 1 Ci (0.058 g) of plutonium-238 oxide. Verification of this assumption was the purpose of this experiment. A 90% dissolution of the PuO_2 present was to be achieved in each of the four steps. The results of this experiment can be seen in Figure 11. Only 93.96% of the PuO_2 was dissolved in the four-stage process (a decontamination factor of only 16.55). This experiment certainly showed that the desired decontamination factor of 10,000 cannot be achieved using the above mentioned basis for determining the amount of $(NH_4)_2Ce(NO_3)_6$ required. As found in Experiment B, the amount of ceric ammonium nitrate must be based on the total amount of material (PuO_2, sealant, and other organics) present. This must be determined for each dissolution since the amount to be reacted varies with each batch.

Summary

Filter media which were contaminated with americium-241 and plutonium-239 oxides were treated with three different leaching reagents to determine the effectiveness of each reagent in dissolving the actinide oxides. The three reagents used were 8N HNO_3, 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$, and 12N HNO_3-0.05N HF-0.01N H_2SO_4. It was

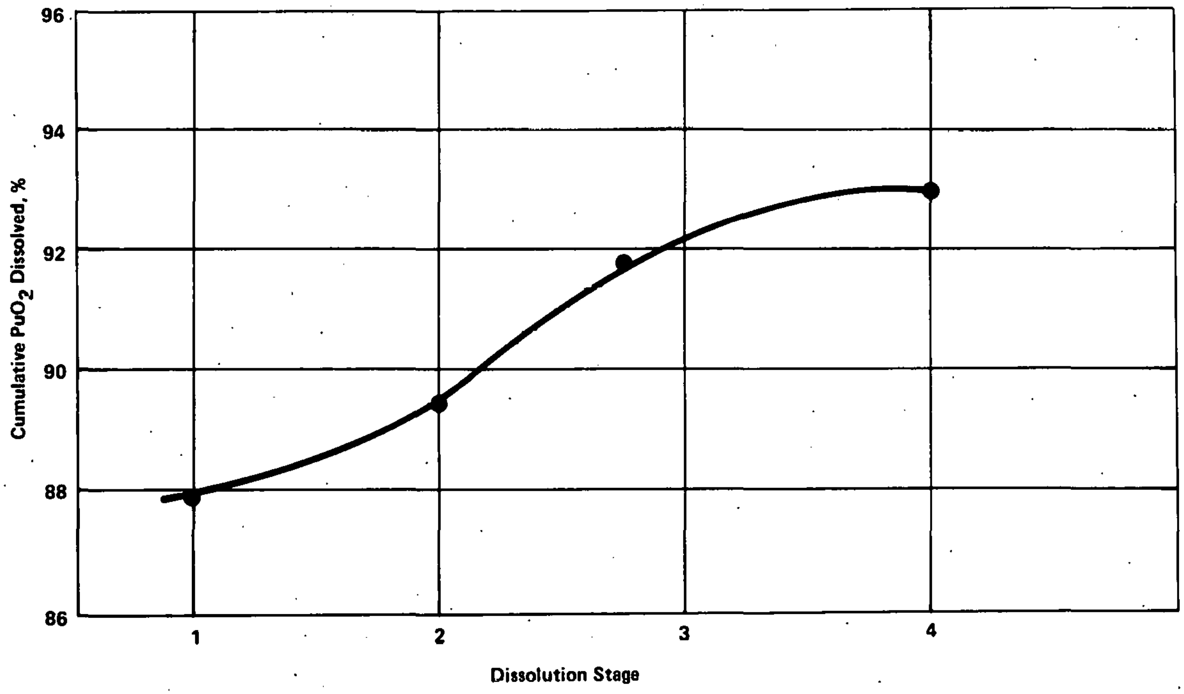

FIGURE 11 - Cumulative weight % PuO_2 dissolved during four stage process.

found that the best leaching agent was the 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$ which dissolved greater than 90% of the AmO_2 and the PuO_2.

Another series of tests was conducted to determine the best leaching agent for uranium-233 oxide (U_3O_8). Simulated samples of contaminated filter media were prepared by mixing 0.2 g U_3O_8 powder with 3 g of shredded filter media. The reagents used were the same three as used for the AmO_2-PuO_2 studies. It was found that all three reagents were successful in dissolving greater than 90% of the U_3O_8 in a very short time (15 min).

Fusion studies were conducted using Na_2CO_3 and PuO_2. The purpose of these experiments was to determine the effect of temperature on fusion efficiency. It was found that there was not a significant difference in percent PuO_2 solubilized (4.6%) within the temperature range under investigation (1200-1300°C).

Experiments were conducted using filter media contaminated with neptunium-237 oxide. The purpose of the tests was to determine which of the three reagents would be successful in dissolving the NpO_2. Results indicate that both the 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$ and the 12N HNO_3-0.05N HF-0.01N H_2SO_4 were successful in dissolving greater than 90% of the NpO_2 in 0.5 hr. The 8N HNO_3 on the other hand solubilized only 32% of the NpO_2 in 8 hr and therefore would not be recommended as a leaching agent for NpO_2.

In other series of experiments, filter media contaminated with curium-244 oxide were dissolved in leaching tests using both 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$ and 12N HNO_3-0.05N HF. Both reagents dissolved essentially 100% of the CmO_2 in approximate 10 min and therefore would be recommended for dissolving CmO_2 from filter media.

Filter media contaminated with thorium-232 oxide were leached with a 4N HNO_3-0.1M $(NH_4)_2Ce(NO_3)_6$ solution in an attempt to dissolve the ThO_2. The rate of dissolution was slow at the beginning (0-7 hr), rapid from 7 to 9 hr, and slow again at the end (9-20 hr). Complete dissolution was achieved in 20 hr, considerably longer than for the other actinide oxides previously tested. A point of interest is that in a similar experiment using only 4N HNO_3-0.1M ceric ammonium nitrate and ThO_2 (no filter media), the amount dissolved was only 25% after 20 hr. The reason for this difference is not understood at this time. Therefore, it is concluded that although the dissolution time was much longer for the ThO_2, it is still an acceptable leaching reagent.

Several experiments were completed using PuO_2 contaminated filter media from spent glovebox filters. These tests were made in order to verify the flowsheet given last quarter [1] and to determine how much $(NH_4)_2Ce(NO_3)_6$ is required for dissolution of PuO_2 from such filters. In one experiment, a 9.5-g sample of filter media was processed through a six-stage dissolution process. In the initial five stages, 4N HNO_3-0.23M $(NH_4)_2Ce(NO_3)_6$ was used and in the sixth stage 12N HNO_3-0.1N HF was used. The amount of $(NH_4)_2Ce(NO_3)_6$ used in each stage was 60 times the initial moles of PuO_2 present in the filter media. The overall decontamination factor obtained for the six-stage process was 833. In a second experiment, a 25.6-g sample of filter media was processed through a four-stage dissolution procedure. The leaching solution was 4N HNO_3-0.30M $(NH_4)_2Ce(NO_3)_6$. The amount of ceric ammonium nitrate used in each stage was 120 times the initial moles of PuO_2 present in the filter media. The overall decontamination factor achieved in the four-stage process was 55.8. In a third experiment, a 30-g sample of filter media was dissolved in a four-stage dissolution process. The amount of $(NH_4)_2Ce(NO_3)_6$ used in each stage of this process was based upon the assumption that 0.01 mole of Ce^{+4} would oxidize 1 Ci (0.058 g) of plutonium-238. The overall decontamination factor achieved for the four-stage procedure was 16.55. The flowsheet given last quarter [1] was based on the above assumption (0.01 mole Ce^{+4}/curie Pu-238) and an overall decontamination factor of 10,000. This degree of decontamination was certainly not achieved and hence the flowsheet is not valid.

The amount of ceric ammonium nitrate required cannot be based exclusively upon the amount of actinide present in the filter media. The reason for this is that other reactants such as pack-to-frame sealant* and other organic materials are present in the spent filter media. A high percentage of sealant was present in the 25.6-g sample and this is perhaps why less plutonium was dissolved. In other words, even though more ceric ammonium nitrate was used per mole of plutonium present, a lower percentage of the plutonium dissolved because a large percentage of the Ce^{+4} ions were depleted by reaction with organics present. A formula for calculating the $(NH_4)_2Ce(NO_3)_6$ required per gram of filter media has not been developed. More studies must be done before the relationship can be determined.

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*This is a fire retardant polyurethane and a rubber base adhesive.

Reference

1. D. F. Luthy and E. L. Lewis, Decontamination of HEPA Filters: April-June 1977, MLM-2445 (September 22, 1977), Figure 6, p. 12.

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