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Recovery of Plutonium from Plutonium-Beryllium Neutron Sources

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by

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ABSTRACT

At the Los Alamos National Laboratory, plutonium-beryllium neutron sources have traditionally been processed for plutonium recovery by precipitating the plutonium as plutonium oxalate, calcining to plutonium dioxide, redissolving the oxide and then precipitating as oxalate again. In most cases three dissolutions of the oxide are required to reduce beryllium content enough to meet specifications.

We first introduced an ion-exchange process to separate the beryllium from the plutonium. This process requires only one run, rather than three, to produce an acceptable oxide. We then experimented with ways of making the process more efficient. We found that increasing the volume of the wash solution and reversing the direction of loading and washing greatly improved the purity of the oxide.

I. BACKGROUND AND INTRODUCTION

Since the mid 1950s, plutonium-beryllium(13) alloy has been used as a neutron source in the nuclear industry and in experimental laboratories. It has been manufactured by a variety of methods, such as:

1) reduction of plutonium fluoride with an excess of beryllium;[1]
2) reduction of plutonium dioxide with an excess of beryllium;[2,3,4]
3) heating the two metals together.[5]

The method most commonly used in the United States was developed at Mound Laboratory by John Richmond and Charles Wells.[6] It involves placing a specified amount of plutonium metal in the drilled-out center of a weighed piece of beryllium rod, which is then inserted into a tantalum capsule. The beryllium rod has been coated with a thin layer of beryllium oxide-nitride to prevent any alloying of the beryllium with the tantalum capsule. The capsule and its contents are heated to a temperature of about 1150°C, producing an exothermic reaction that results in the formation of the $PuBe_{13}$ alloy.

When a source is no longer needed, it must be decommissioned and the plutonium recovered. Los Alamos National Laboratory has been charged with this responsibility.

Some of the problems posed by processing of $PuBe_{13}$ are:

1) the sources are high radiation emitters;
2) plutonium recovery uses hydrochloric acid, which requires special processing equipment;

3) beryllium processing requires a separate area, isolated from the normal plutonium processing areas.

In the past, neutron sources have been processed at Los Alamos by dissolution of the alloy in HCl. The acidity of the solution was reduced with water, and a crude plutonium oxalate was formed by addition of 1M oxalic acid. Although most of the beryllium was kept in solution, some precipitated with the oxalate cake and therefore contaminated the product oxide. The impure PuO_2 was redissolved in an HCl and HF mixture with heat, and the resulting solution was again precipitated as an oxalate. A total of three passes was usually needed to bring the plutonium oxide into product specifications. This method is not only very time-consuming, but the HCl/HF mixture does not efficiently dissolve the plutonium oxide.

II. RADIATION PROTECTION

Our efforts were directed not only at overcoming the disadvantages of the traditional methodology but also at improving the work environment. Because neutron levels are much higher during plutonium recovery than during other plutonium processing stages, radiation exposure of workers in the recovery areas is a major concern.

Most of the exposure takes place while the sources are being decanned and dissolved (the high neutron generation occurs only while the plutonium and beryllium are alloyed). During this part of the process, the neutron readings at the exterior of the gloveboxes are typically between 75 and 100 mR/h. The $3\frac{1}{2}$-inch-thick lucite shields in front of the gloveboxes reduce these readings to approximately 5 mR/h, which represents a 25-fold decrease in radiation in the working area.

To contain neutrons, a bisco[7] pig was placed inside the glovebox. Bisco is a high hydrogen-shielding product that contains boron carbide, lead, aluminum, nitrogen, carbon, and oxygen. The pig is 6 inches in diameter and has a 2-inch hole drilled in the center to store the sources before dissolution.

Another exposure problem was occurring during the aqueous recovery procedure. Because most of the sources were manufactured in the 1950s and early 1960s, the plutonium has undergone radioactive decay to americium(241), which is a much stronger gamma-emitter than plutonium. Gamma readings, especially during hydroxide precipitation, were much higher than originally expected. Using lead shielding for both the storage tanks and the filter vessel brought the radiation readings into a much more acceptable range.

III. EQUIPMENT

Because neutron source processing uses hydrochloric acid, the materials used for the gloveboxes and the processing equipment are important. The stainless steel traditionally used for the gloveboxes at Los Alamos was not a good choice because stainless steel corrodes in an HCl environment. The corrosion, moreover, adds unwanted impurities, especially iron, which does not separate from plutonium in chloride anion exchange. The new gloveboxes were constructed as stainless steel shells lined with polyvinylidene fluoride (PVDF). All equipment inside the gloveboxes was made of nonmetallic materials. All pipes and glass column endplates were constructed of either chlorinated polyvinyl chloride (CPVC) or PVDF.

The glovebox line itself is isolated from the rest of the aqueous recovery area. It is connected to the normal processing areas by a conveyor system, and a door permits passage to and from those areas. The glovebox line has its own introductory hood for bringing in items and sources needed in the $PuBe_{13}$ process.

Most of the solution stored in the gloveboxes is in slab tanks made of PVDF. This design allows for greater storage volumes without impinging on available work space. The precipitations are carried out in glass tanks equipped with CPVC endplates. The oxalate precipitation tank is equipped, in addition, with a tantalum stirrer for thorough mixing of the eluate and the oxalic acid.

Each of the aqueous processing gloveboxes is equipped with an off-gas scrubber. The scrubbers are placed in line with the vacuum system, so that any time the vacuum is used (to filter, transfer solution into a tank, sparge a tank, or pull fumes off during dissolution), any chloride gases generated are pulled through the scrubber before entering the house vacuum system. The fumes are passed through a layer of calcium carbonate (marble chips) to remove the chloride.

The plutonium oxalate is calcined in a platinum-lined furnace can. Platinum was selected because of its chloride resistance, and because it does not oxidize and therefore does not add any impurities to the product oxide.

The ion-exchange columns are 3- or 4-inch-by-24-inch glass pipe with CPVC clamp-type endplates. Other equipment is standard laboratory equipment that can be purchased from any laboratory supply company.

IV. METHODOLOGY

The plutonium recovery process uses the following reagents:

- Lewatit MP-500, 40-70 mesh, anion exchange resin
- Hydrochloric acid
- 8M HCl and 0.01M $NaNO_2$
- 0.5M $NH_2OH \cdot HCl$ and 0.1M HCl
- Oxalic acid ($H_2C_2O_4 \cdot 2H_2O$)
- Hydroxylamine hydrochloride ($NH_2OH \cdot HCl$)
- 9M KOH
- Sodium nitrite ($NaNO_2$)
- Calcium carbonate ($CaCO_3$).

Figure 1 is a flow diagram of our process for recovering plutonium from neutron sources.

Before aqueous recovery of the plutonium can begin, the $PuBe_{13}$ alloy must be exposed to the atmosphere. The source is placed in the chuck and a capsule-cutting saw (see Fig. 2) is used to cut through one end of the stainless steel jacket. The cap is removed, the source is turned around in the chuck, and the other end of the stainless steel jacket is removed. If the jacket slides off easily, the same procedure is then used on the tantalum capsule, except only one end needs to be cut. If the stainless jacket cannot be removed, it is placed in a vise attached to a stepping motor. This allows a crosscut to be made the length of the source. The jacket can then be removed with ease (Fig. 3 shows the source with the $PuBe_{13}$ alloy exposed).

After the $PuBe_{13}$ alloy has been exposed to the atmosphere, it is placed in the dissolution tank with 500 ml of water. A condenser attached to a KOH trap to remove chloride fumes is connected to the tank and a slow argon sparge is started. The amount of 12M hydrochloric acid required for dissolution is calculated as follows:

$$(\text{grams of Pu}) \times 11 = \text{ml of 12M HCl needed.}$$

The acid is added in 100-ml increments at 10-minute intervals to control the rate of hydrogen evolution.

When all of the HCl has been added, the solution is allowed to cool and then is filtered. The tantalum capsules are rinsed with 12M HCl to remove any residual $PuBe_{13}$ that remains attached to the walls of the Ta cylinder. The undissolved residues are also washed with 6M HCl and all solutions are combined. The residues and capsules are combined with those from previous runs, to be discarded when enough bulk is accumulated.

The feed solution is treated to change the valence of the plutonium from +3 to +4 and to increase the chloride concentration to 8 M. The amount of sodium nitrite needed is calculated by the following formula:

$$(\text{grams of Pu in feed}) \times (0.5) = \text{grams of } NaNO_2 \text{ needed.}$$

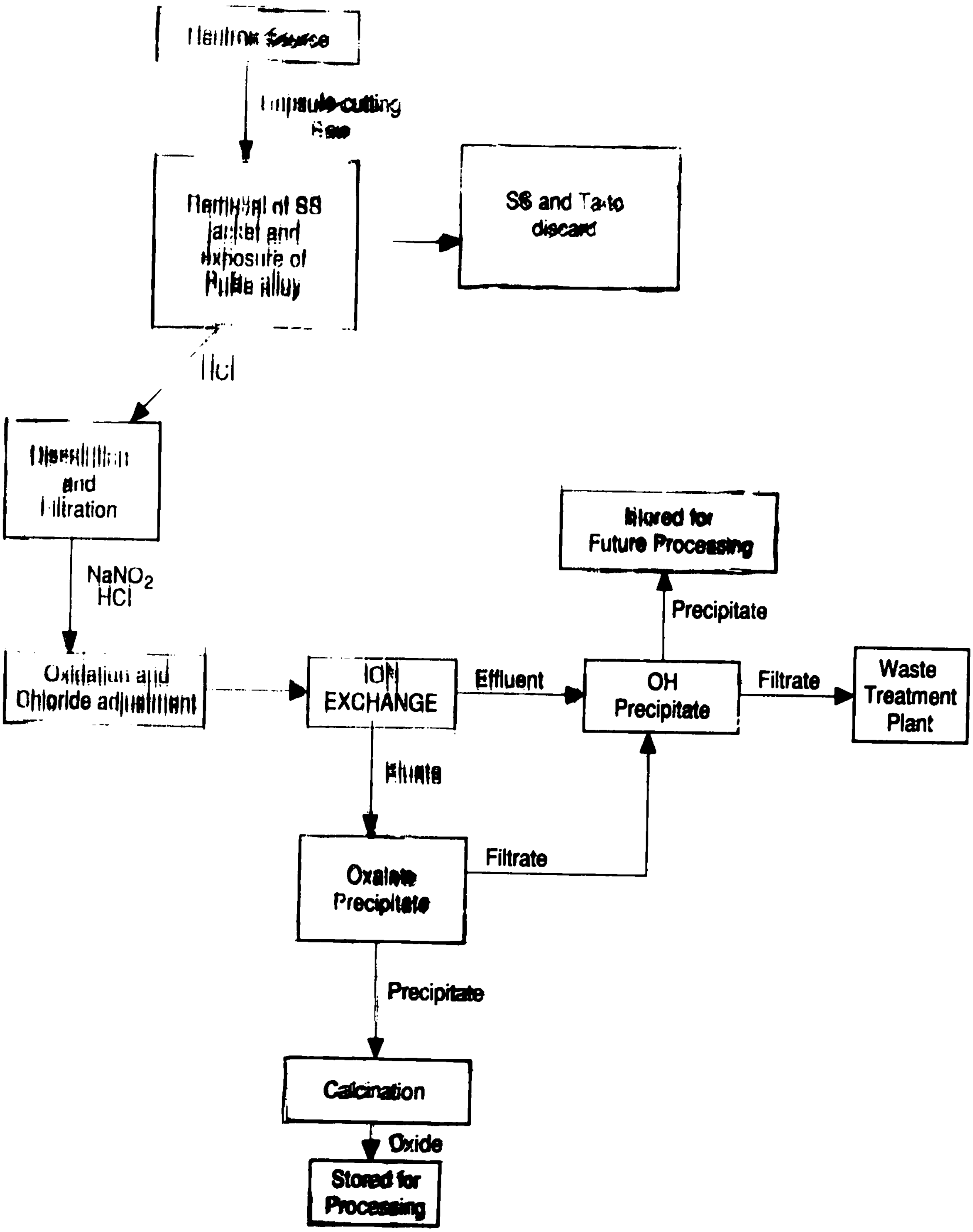

Fig. 1. Flow Diagram For Processing of PuBe Neutron Sources

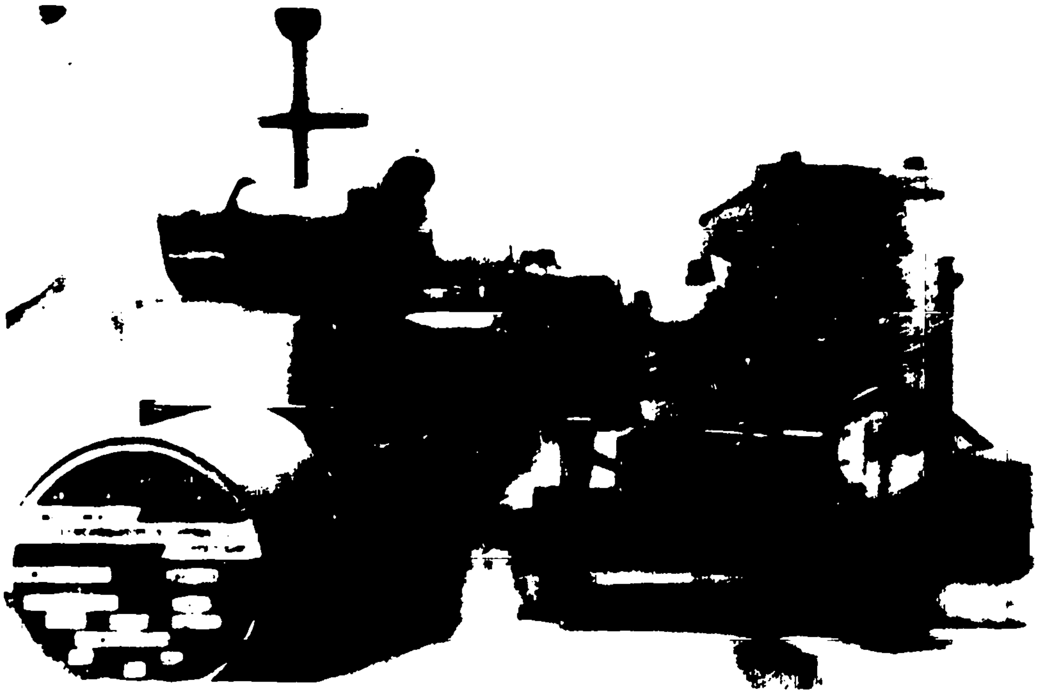

Fig. 2. Capsule-cutting Saw

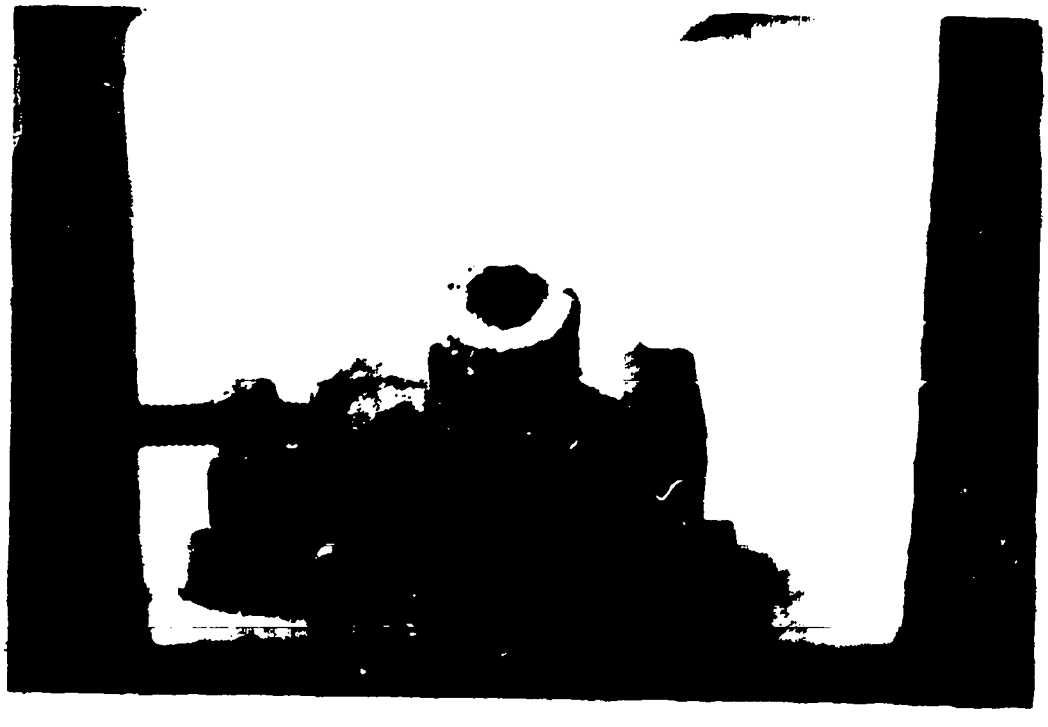

Fig. 3. Source with PuBe Alloy Exposed

The $NaNO_2$ is made into a slurry with a minimum amount of water. A vacuum is pulled on the treating tank and the slurry is introduced slowly into the feed through the bottom drain valve. The solution is allowed to mix for 30 minutes. After the reaction is complete, a sample of the feed is taken and titrated for the chloride ion by the silver chromate method[8] using 0.2M silver nitrate. If the chloride concentration of the feed is below 8M, enough 12M HCl is added to bring it up to 8M; if the concentration is 8M or greater, no adjustments are made. After oxidation and chloride adjustment, the solution is filtered to remove any chloride salts that may have precipitated out during the treatment process. The salts are washed with a small amount of 12M HCl to remove the last traces of plutonium. The feed is now ready to be loaded onto the ion-exchange column.

Before being loaded, the column is subjected to a vacuum to remove the 0.1M HCl the resin has been moved in. The resin is then reconditioned with two void column volumes of 0.01M $NaNO_2$ in 8M HCl solution. The feed solution is run through the column, from bottom to top, at a rate of 5 to 10 liters per hour. A slight vacuum is pulled on the column to remove the rest of the solution. It is followed by three void column volumes of 0.01M $NaNO_2$ in 8M HCl wash at a rate of 10 to 15 liters per hour. All the solutions (reconditioner, feed, and wash) are combined and sampled. The effluent is transferred to the hydroxide precipitation tank.

The column is now ready to be eluted. It is filled with a solution of 0.5M $NH_2OH{\cdot}HCl$ in 0.1M HCl and allowed to stand, vented, for 15 minutes. Then the flow direction is reversed: the elutriant is introduced at the top and run through to the bottom. The remainder of the hydroxylamine hydrochloride is run through the column, followed by 8 liters of 0.1M HCl to remove the remaining $PuCl_3$ solution. The two solutions are combined and sampled.

The eluate is transferred to the oxalate precipitation tank. The Ta stirrer is turned on and hydroxylamine hydrochloride is added to the solution to ensure that all the plutonium is in the +3 state (0.5 g of $NH_2OH{\cdot}HCl$ is added per gram of plutonium and the solution is allowed to react for a minimum of 30 minutes). Next, 0.9 g of oxalic acid is added per gram of plutonium, and this is allowed to digest for 30 minutes. The plutonium oxalate is allowed to settle and is then filtered. The tank and the precipitate are washed with 0.1M $H_2C_2O_4$ + 0.2M HCl. The filtrate and wash are combined, sampled, and transferred to the hydroxide precipitation tank.

The precipitate is allowed to air-dry, then the oxalate cake is placed in a platinum furnace can and transferred to a calcination furnace. The temperature is slowly raised to 600°C and is held at that temperature for 6 hours. After cooling, the oxide is weighed and a 4.5-g sample is taken for analysis. The balance of the oxide is stored.

The hydroxide precipitation is performed on both the effluents and the oxalate filtrate. Because the effluents contain large amounts of beryllium, an excess of KOH is added to redissolve the $BeOH_2$—unlike our usual hydroxide precipitations, in which the pH is adjusted to approximately 7. Five liters of water are then added to the precipitating tank, to prevent potassium chloride salts from precipitating out of the solution. Calculations show that we are operating slightly above the solubility level of KCl with the concentrations of potassium and chloride ions in the solution. The hydroxide precipitate is allowed to settle below the decanting tube. The clear liquid is run through the filterboat to catch any particles of plutonium hydroxide that have not settled. The remaining 4 liters of solution, containing the bulk of the precipitate, are then filtered. The process is repeated on the oxalate filtrate, but without an excess of KOH since no beryllium is present in the filtrate. The hydroxide filtrates are then sampled. If the alpha count is below 5×10^9 disintegrations per minute per liter, the filtrates are sent on to waste treatment for further processing. If the filtrates are not below discard limits, they are reprocessed.

V. EXPERIMENTAL MODIFICATIONS

After completing some preliminary runs using the above-described procedure, we made some modifications with the intent of increasing its efficiency. The parameters we studied were:

- feed treatment
- solution used for wash
- direction in which the column is loaded and washed
- amount of wash solution used
- flow rates.

The modifications were as follows:

1) The feed was adjusted to 0.1M fluoride using HF, and HF was also added to the wash (solution of 8M HCl, 0.01M NaNO$_2$, and 0.007M HF).
2) The feed was treated in the normal fashion (no HF added), but HF was added to the wash as above (8M HCl, 0.01M NaNO$_2$, and 0.007M HF).
3) The ion-exchange column was loaded and washed from top to bottom, instead of in the traditional bottom-to-top direction.
4) Thirty liters of wash, instead of fifteen to eighteen, were used.

Runs were made to test the effect of each modification. For each, samples were taken of the treated feed, effluent, eluate, oxide, and—every 2 liters—the wash. For comparison, the same samples were taken during a run without any of the above modifications.

In the process of experimenting with these variables, we also discovered that we could reduce the volume of effluent sent to hydroxide precipitation. We therefore added this parameter to our list of modifications.

VI RESULTS

A. Feed Treatment

It had been suggested that there might be some adsorption of beryllium on the resin. If the feed were adjusted to 0.1M HF, the beryllium would be complexed and not adsorbed on the column. Table 1 shows a comparison of plutonium in the effluents and beryllium in the eluates with changes in various parameters.

TABLE 1. Pu and Be Concentrations as a Function of Changing Processing Parameters

Run#	Feed Treatment	HF in Wash Solution	Loading Direction	Wash Volume (l)	Total Pu in Effluent (g)	Pu per liter in Effluent (g/l)	Be in Eluate (mg/l)
9	NaNO$_2$ and HCl	none	top-down	18	0.41	0.016	1
13	NaNO$_2$, HCl, and 0.1M HF	0.007M	top-down	18	0.776	0.035	5
14	NaNO$_2$ and HCl	0.007M	top-down	16	0.133	0.006	5
15	NaNO$_2$ and HCl	none	bottom-up	18	0.135	0.006	5
16	NaNO$_2$ and HCl	none	bottom-up	30	0.24	0.014	0.67

As the table shows, adding HF to the feed increases the amount of plutonium in the effluent by a factor of 5 without decreasing the beryllium in the eluate. Figure 4 shows clearly how one of these runs (#13), for which 0.1M HF was added to the feed, contrasts with another (#15), for which no HF was added to the feed solution. The pronounced effect of the HF is shown by the fact that the plutonium concentration remains higher throughout the wash cycle. In addition, if we look at the concentration of plutonium (in g/l) in the effluent, which includes all the solution leaving the column, we see that the only run with a significant increase of plutonium is the run for which fluoride was added to the feed. Further, as shown in Table 1, the concentration of beryllium in the eluate was the same (5 mg/l) for both runs, indicating that the addition of HF to the feed stream does not further purify the eluate.

Tables 2, 3, and 4 show the concentrations of Pu, Be, and Am, measured by sampling the effluent every 2 liters. These tables show that there was no increase in the plutonium removed from the column during the wash phase, except for the run for which HF had been added to the feed. The

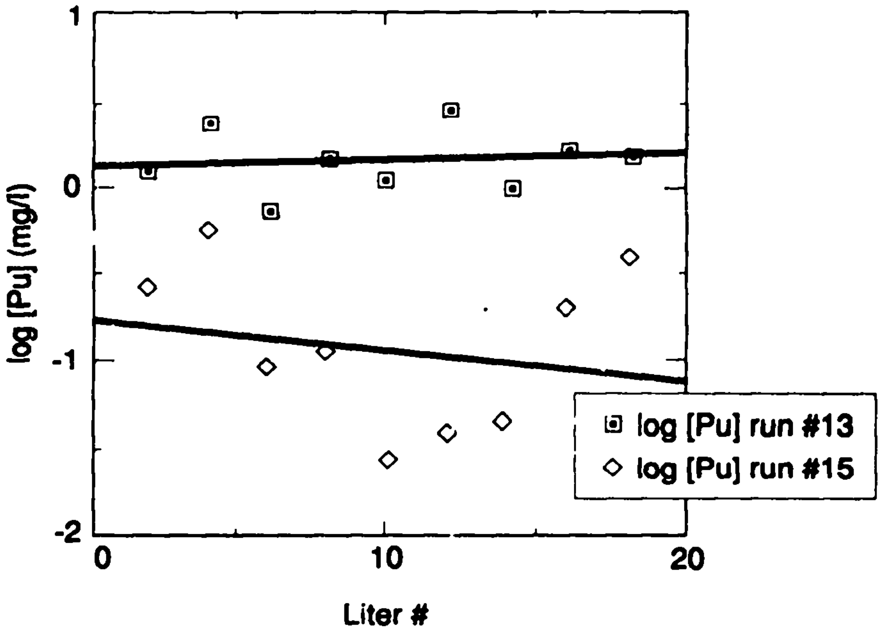

Fig. 4. Log [Pu] in Effluents vs Number of Liters of Wash

higher levels of plutonium seen in the first 2 liters (see Table 2) can be attributed to the plutonium either not totally oxidizing to the +4 state, oxidizing to the +6 state, or reducing back to the +3 state before contact with the column.

TABLE 2. Comparison of Pu Concentrations in the Wash Solution (mg/l)

	Run #09	Run #13	Run #14	Run #15	Run #16
Feed	No HF	0.1M HF	No HF	No HF	No HF
Wash	8M HCl, 0.01M $NaNO_2$	8M HCl, 0.01M $NaNO_2$, 0.007M HF	8M HCl, 0.01M $NaNO_2$, 0.007M HF	8M HCl, 0.01M $NaNO_2$	8M HCl, 0.01M $NaNO_2$
Direction	Bottom-up	Bottom-up	Bottom-up	Top-down	Top-down
Liter #					
0				1.52	13.8
2	11.1	1.27	3.73	0.273	0.249
4	6.43	2.45	0.756	0.602	0.055
6	0.608	0.709	0.654	0.0907	0.244
8	0.661	1.54	2.08	0.115	0.449
10	0.589	1.1	0.413	0.027	0.34
12	0.272	3.24	0.2	0.0388	0.365
14	0.981	1.01	0.264	0.0436	0.133
16	0.975	1.77	0.577	0.219	0.0765
18		1.67		0.432	0.0986
20					0.108
22					0.082
24					0.115
26					0.393
28					0.0615
30					0.0765

TABLE 3. Comparison of Be Concentrations in the Wash Solution (mg/l)

	Run #09	Run #13	Run #14	Run #15	Run #16
Feed	No HF	0.1M HF	No HF	No HF	No HF
Wash	8M HCl, 0.01M $NaNO_2$	8M HCl, 0.01M $NaNO_2$, 0.007M HF	8M HCl, 0.01M $NaNO_2$, 0.007M HF	8M HCl, 0.01M $NaNO_2$	8M HCl 0.01M $NaNO_2$
Direction	Bottom-up	Bottom-up	Bottom-up	Top-down	Top-down
Liter #					
0				20000	35700
2	17000	18000	20000	18000	32400
4	5100	8800	6500	3000	6500
6	280	740	810	90	175
8	85	155	180	8	27
10	14	34	25	2	11
12	7	12	3.3	1.5	17
14	1.5	7	0.8	<1	3
16	1.7	3	0.7	<1	4
18		12		<1	2
20					2
22					1
24					2
26					6
28					2
30					3

TABLE 4. Comparison of Am Concentrations in the Wash Solution (mg/l)

	Run #09	Run #13	Run #14	Run #15	Run #16
Feed	No HF	0.1M HF	No HF	No HF	No HF
Wash	8M HCl, 0.01M $NaNO_2$	8M HCl, 0.01M $NaNO_2$, 0.007M HF	8M HCl, 0.01M $NaNO_2$, 0.007M HF	8M HCl, 0.01M $NaNO_2$	8M HCl, 0.01M $NaNO_2$
Direction	Bottom-up	Bottom-up	Bottom-up	Top-down	Top-down
Liter #					
0				163	284
2	173	273	0.199	137	242
4	34.9	94	42	15.5	46.3
6	2.15	9.74	6.35	0.577	2.13
8	0.657	2.07	1.46	0.0596	0.18
10	0.113	0.461	0.198	0.0185	0.0559
12	0.0671	0.175	0.0293	0.0129	0.0382
14	0.0217	0.1	0.00954	0.00763	0.018
16	0.0266	0.042	0.00974	0.00545	0.0105
18		0.208		0.00588	0.00729
20					0.0106
22					0.00718
24					0.0058
26					0.0243
28					0.0111
30					0.0136

B. Solution Used for Wash

Hydrofluoric acid (0.007M) was added to the wash solution to test whether beryllium might have some very slight adsorption on the resin. From a comparison of runs 9 and 14 (see Table 3), it is clear that no more beryllium is removed in the wash solution with the HF than in the wash solution that contains no HF. Also, as seen in Figs. 5 and 6, the slope of the beryllium line is the same as that of the americium line. Since americium has no adsorption on the resin, beryllium must not have any adsorption either.

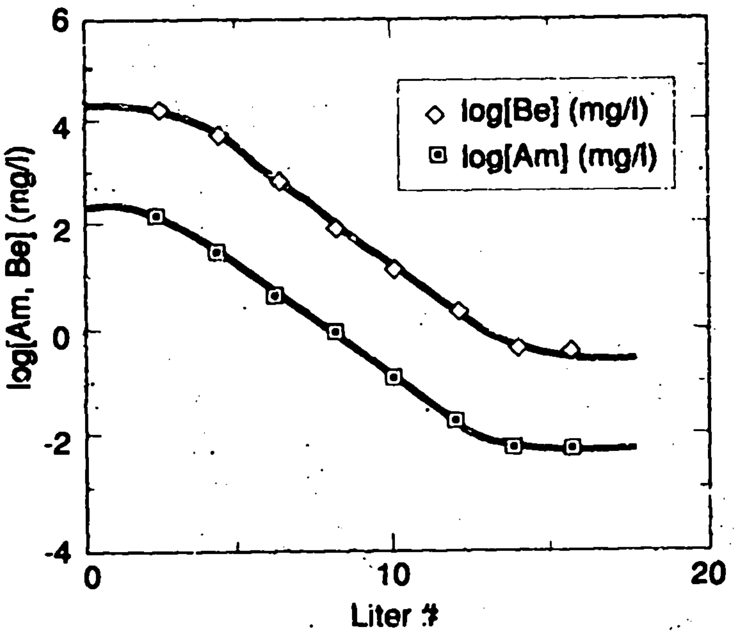

Fig. 5. Log [Am and Be] vs Volume (liters)—Run #14 Loaded and Washed Bottom-up

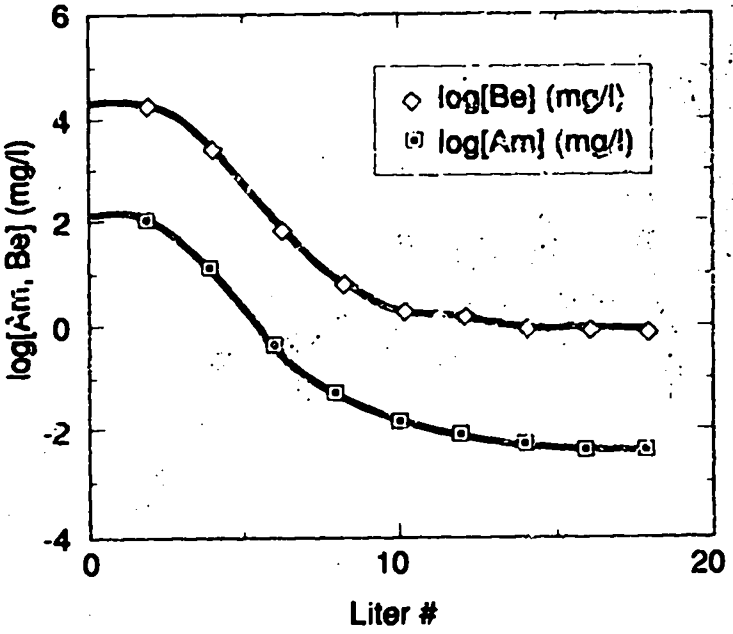

Fig. 6. Log [Am and Be] vs Volume (liters)—Run #15 Loaded and Washed Top-down

C. Direction of Loading/Washing of Column and Volume of Wash Solution

Neither the direction of the wash solution through the column nor the amount of wash used had any real effect on the amount of plutonium removed from the column. In Table 2, the comparison of runs 9 and 15 shows no increase in plutonium concentrations sampled at 2-liter intervals when the direction of the flow is reversed. Similarly, compare runs 15 and 16. The volume of wash was increased from 18 liters in run 15 to 30 liters in run 16, but the plutonium concentrations sampled every 2 liters from the 18th through the 30th liter of the wash step show no significant increases. The very low plutonium levels in the wash solution are expected, since the distribution coefficient of plutonium in 8M HCl is >500.[9]

The flow direction of the solutions and the volume of the wash did, however, significantly affect the purity of the product. Figures 5 and 6 show the log of both the americium and the beryllium concentrations versus the volume of the wash with bottom-up and then with top-down flow Figure 5 shows that after 15-liters, the elution curves are just beginning to flatten out. Figure 6 shows that if the flow is changed to a downward direction, the curves flatten out at about the 10-liter mark. To test the combined effect of this variable and that of an increase in the amount of wash solution, we did run #16 with 30 liters of wash instead of 18. Figure 7 shows that when 30 liters of wash were used, the concentration of beryllium in the effluent remained steady from the 15-liter mark on. In addition, as seen in Table 1, at 30 liters of wash the concentration of beryllium in the eluate was decreased by 700%.

The final product of the process is plutonium dioxide. Table 5 shows the beryllium concentrations of the oxides obtained on different runs (the oxalates from runs 13 and 14 were combined for the calcination step). These concentrations confirm the conclusions reached from the wash and eluate data: loading and washing in a downward direction and increasing the volume of the wash will yield an oxide with lower beryllium concentration. The volume of the wash has the greater effect on the purity of the oxide. The oxide produced by run #16, using 30 liters of wash, was lower in beryllium by at least a factor of 3 than that of any other run using less wash solution.

TABLE 5. Be Concentration in PuO_2

Run#	9	13	15	16
[Be] (ppm)	18	32	12	4

Table 6 gives an analysis of the oxide from run #16. The results are predictable. The total amount of beryllium present in the plutonium was reduced from 130 g to 0.9 mg, representing a decon factor of >144,000. The iron and tantalum concentrations were high, but this was expected (and removal of Fe and Ta was not the aim of the project—see below). Depending on the product purity, the oxide can be sent to blending, to be prepared for reduction to the metal, or, if needed, it can be sent to nitrate processing for further purification.

TABLE 6. Analysis of PuO_2: Run #16

Pu	86.69%	Th	<20 ppm
Be	4 ppm	U	70 ppm
Am	<10 ppm	Al	<5 ppm
Ta	355 ppm	Cu	30 ppm
Fe	210 ppm	Na	<50 ppm
Ni	55 ppm	Mg	15 ppm
Cr	35 ppm	Mo	20 ppm

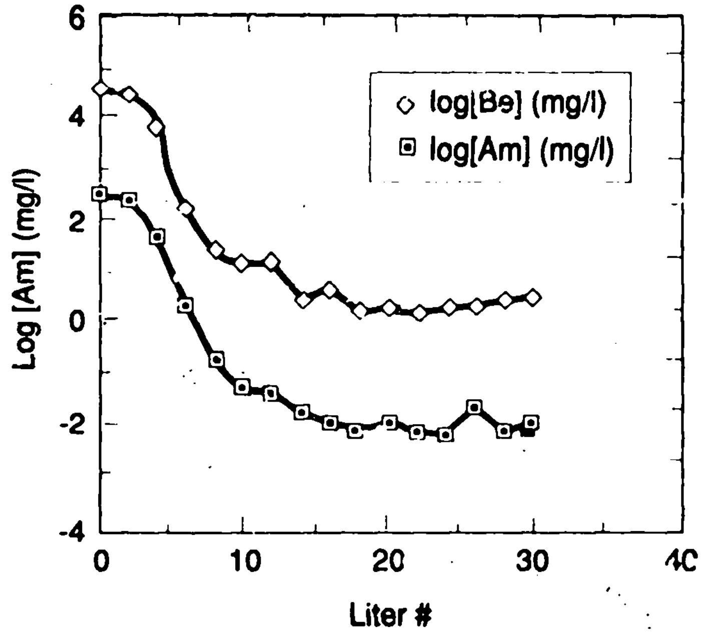

Fig. 7. Log [Am and Be] vs Volume (liters)—Run #16 Loaded and Washed Top-down

D. Flow Rates

The flow rates for the feed and the wash had little effect on the efficiency of the column. The flow rate for the feed was varied from 5 to 10 liters per hour and the flow rate for the wash was varied from 10 to 30 liters per hour, with little effect on the purity of the eluate.

E. Volume of Effluent Sent to Hydroxide Precipitation

The original process called for combining all of the effluents, then neutralizing and filtering them. However, after studying the plots of the log[Am] (Figs. 5, 6, and 7), we concluded that after 10 liters of wash, the Pu and Am concentrations in the remaining wash solution should be low enough that the solution could be sent to a separate cold tank and simply neutralized with potassium hydroxide. An alpha sample would be taken to ensure that the level was indeed below 5 × 10^9 disintegrations per minute per liter (approximately 31 mg of Pu/l or 0.67 mg of Am/l), and the neutralized solution would then be sent on to the waste treatment plant without filtering.

Each of the three runs made using this procedure produced a cold solution. Because the filtration of hydroxides is very slow, the time saved is considerable: the new procedure reduces by 67% the amount of solution to be filtered.

F. Removal of Other Elements

While running the experiments on americium and beryllium, we also studied the ion-exchange column's ability to remove nickel, iron, chromium, and tantalum. Table 7 compares the starting and finishing products and the amounts of these elements that they contain.

As expected, the nickel and the chromium were removed from the stream while the tantalum and the iron followed the plutonium into the eluate. However, the objective of the process is to remove the beryllium from the plutonium because we do not want to introduce excessive amounts of beryllium into the plutonium processing lines. After removal of the beryllium, the plutonium can be processed in the normal manner.

TABLE 7. Removal of Fe, Cr, Ni, and Ta

	Run #09			Run #15			Run #14		
	Feed	**Eluate**	**Decon Factor**	**Feed**	**Eluate**	**Decon Factor**	**Feed**	**Eluate**	**Decon Factor**
Volume (l)	5	12.4		5.8	9.5		5.8	10	
Fe (mg/l)	185	60		115	140		108	74	
Total Fe (mg)	925	744	1.2	667	1330	No Decon	626.4	740	No Decon
Cr (mg/l)	30	<2		25	<3		60	<5	
Total Cr (mg)	150	<24	>6	145	<28.5	>5	348	<50	>7
Ni (mg/l)	25	<1		55	<2		35	<2	
Total Ni (mg)	125	<12	>10	319	<19	>15	203	<20	>10
Ta (mg/l)	95	40		60	20		35	20	
Total Ta (mg)	475	496	No Decon	348	190	1.8	203	200	No Decon

VII. CONCLUSIONS

Our experiments led us to conclude that the following changes should be made to increase both the efficiency of the process and the purity of the product:

1) Load and wash the column in a downward direction.
2) Increase the volume of the wash solution to 25-30 liters.
3) Send only the first 10 liters of wash to hydroxide precipitation; pull the remainder into a separate cold tank to be neutralized and sent to waste treatment.

We feel confident that these modifications to the described process will allow consistent production of a low-beryllium product in a single run.

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