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SEPARATION OF ^{238}Pu FROM ^{237}Np BY PRESSURIZED ANION EXCHANGE

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by

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ABSTRACT

Pressurized anion exchange with small particle (∿200 mesh) *Dowex** MSA-1 macroporous ion resin gave excellent separation of ^{238}Pu from ^{237}Np. Separation was satisfactory with ∿90 mesh *Dowex* 1-X4 gel-type resin at 3 to 4 times the flow rates normally used in the Savannah River Plant with 40 to 60 mesh *Dowex* 1 resin. Results with small-particle *Dowex* 1-X2 in the pressurized system were not satisfactory because the resin beads deformed due to low structural stability, with resulting excessive pressure drop and poor separation. Small-particle *Dowex* 1-X8 was very ineffective because of slow ionic diffusion in the resin matrix. The excellent performance of the macroporous resin is attributed to rapid ionic diffusion resulting from high resin porosity.

The pressurized system was operated safely at temperatures up to 55°C and pressures up to 350 psig.

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*Trademark of Dow Chemical Co., Midland, Michigan

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INTRODUCTION

At the Savannah River Plant, many kilograms of ^{238}Pu and ^{237}Np have been processed in the past decade using anion exchange from nitrate solutions.[1] The ^{238}Pu, which is produced from ^{237}Np by neutron capture and subsequent beta decay of the ^{238}Np, has been used mainly in thermoelectric power and heat source applications. Anion exchange has been used to separate the neptunium and plutonium from radioactive fission products and from nonradioactive ionic impurities, and to partition neptunium from plutonium.[2,3] Problem areas in this process have been: (1) obtaining an anion exchange resin that consistently yields good separation of plutonium and neptunium in a single exchange cycle, and (2) the limited stability of the resin to the large dose of alpha radiation that results when the ^{238}Pu is sorbed on the resin.

Presently *Dowex* 1, a gel-type resin with low surface area and no discrete pores, is used in gravity-flow columns. The major problem with this resin has been variable separation efficiency from batch to batch. Conventional resin property measurements such as moisture content and capacity have failed to determine the reason for variation. In general, gel-type resins have small pore openings, and thus slow mass transfer, which contributes to the inefficient separation and slow elution. Therefore, the use of a smaller-particle resin should minimize the resistance to mass transfer. Generally, the lower the degree of crosslinking, the greater the permeability and effective pore size of the resin. However, low crosslinked resins have low structural stability and deform badly at high column pressure, requiring a compromise.

This report describes testing of pressurized anion exchange to establish: (1) whether the processing rate could be increased using small particles of resin, e.g., 170 mesh or smaller, thus reducing the radiolytic damage to the resin per unit of ^{238}Pu processed, and (2) whether using small particles of resin (to reduce diffusion distance both in the resin particle and in the aqueous film)[4] would reduce the considerable variation in efficiency of partitioning neptunium from plutonium shown by different batches of larger-particle resin.

ANION EXCHANGE PROCESS DESCRIPTION

In the present anion exchange process,[2] Np-Pu-Al cermet targets are dissolved in nitric acid, and the resulting concentrated nitrate solution is processed through three anion exchange cycles at atmospheric pressure under gravity flow:

1. Plutonium and neptunium are adjusted to the tetravalent states using ferrous sulfamate followed by heating at ∿50°C. In concentrated nitrate solution, $Pu(NO_3)_6^{2-}$ and $Np(NO_3)_6^{2-}$ result, and these hexanitrato complex ions are cosorbed on the anion exchange resin. After washing with ∿8M HNO_3 to remove fission products, Np^{4+} and Pu^{4+} are coeluted with dilute nitric acid.

2. The eluate from Step 1 is adjusted with concentrated nitric acid, and the hexanitrato anionic complexes are produced by the same valence adjustment used in Step 1. After cosorption, Pu^{3+} is selectively eluted with 5.4M HNO_3-0.05M $Fe(NH_2SO_3)_2$- 0.05M N_2H_4. Np^{4+} is then eluted with dilute nitric acid.

3. The plutonium solution from Step 2 is adjusted with strong nitric acid and heated to yield $Pu(NO_3)_6^{2-}$. The plutonium nitrate complex anion is sorbed and eluted as described in Step 1.

The tests of pressurized anion exchange described in the following sections simulated on laboratory scale the second cycle of the process.

EQUIPMENT

In recognition of literature reports of accidents resulting from chemical reaction of anion resin with strong nitric acid, the experimental equipment included a number of safety features. A safety analysis is included as Appendix A.

A 160-cc stainless steel jacketed column, ∿0.8-in. ID x 16 in. long, was equipped with a rupture disc assembly with a burst range at 22°C of 700 ±25 psig. The assembly was vented to an 8-liter stainless steel container to catch resin and solution should rupture occur. The system was installed in a glove box designed for handling high-level alpha activity. A schematic flow diagram of the system is shown in Figure 1.

A positive displacement pump, external to the glove box, was used to feed the column. The system was designed so that

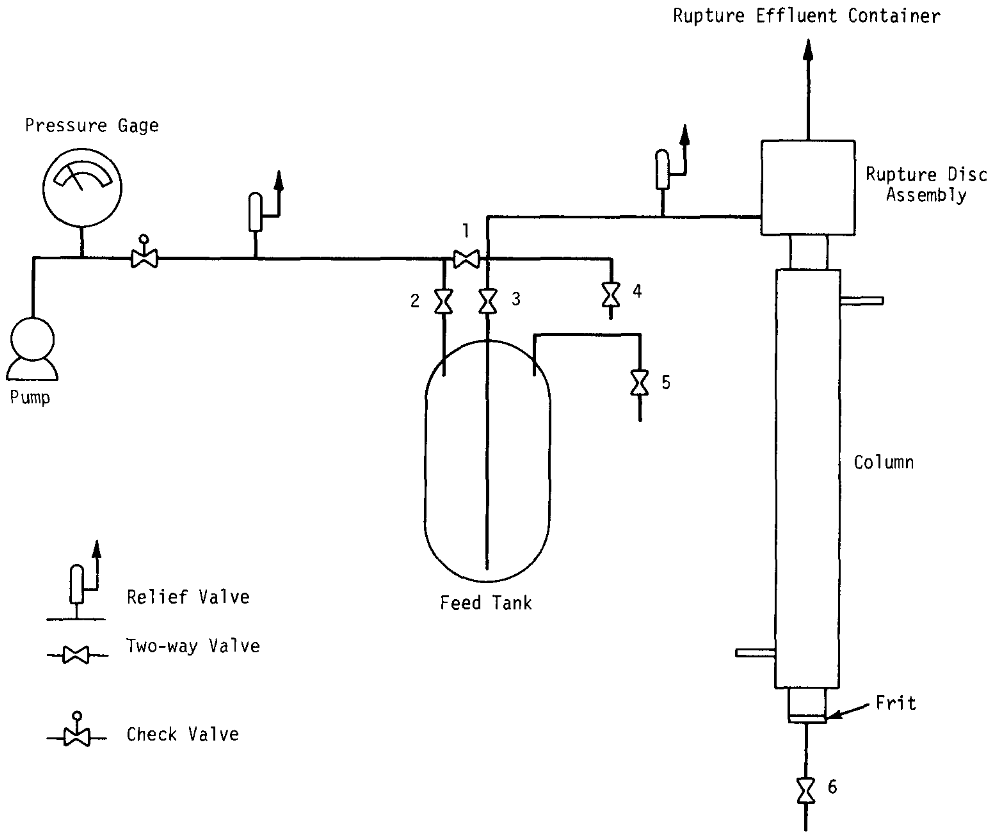


FIGURE 1. SCHEMATIC DIAGRAM OF PRESSURIZED ANION EXCHANGE SYSTEM

nonradioactive solutions for column wash and elution were pumped directly to the column. Radioactive feed was displaced with three tank volumes of $\sim$8M HNO_3 from a pressurized tank. The line from the pump was equipped with a pressure relief valve to prevent pressure buildup ("dead-heading"). A second pressure relief valve was located between the feed tank and column to further reduce the chance of system overpressurization. Both relief valves were set at 500 psig. All associated lines and valves were stainless steel.

The resin was retained in the column by a G-porosity (10 μm) frit of stainless steel. The resin was introduced by vacuum transferring a water slurry of the resin into the top of the column. Used resin was removed by back flushing the resin into a polyethylene bottle. The resin bed was kept at <55°C in the jacketed column with a constant temperature circulating water-bath inside the glove box. Solutions were pumped through the column at a maximum flow of 80 ml/min and a maximum pressure of 350 psig.

No safety problems were encountered in operating the pressurized anion exchange system. However, it is strongly recommended that the precautions detailed in Appendix A be followed whenever nitrated anion exchange resin is used in a pressurized system.

PROCEDURE

Partitioning tests using pressurized anion exchange were conducted only of the second anion exchange cycle in the process. Graded (see Appendix B) nitrate-form resin was loaded into the column to the desired depth, usually ∿15 inches. As an added safety measure, the resin was not conditioned with ∿8M HNO_3 until immediately before the sorption of actinides. The feed, ∿5 g ^{237}Np and ∿1 g ^{238}Pu (or ^{239}Pu), was adjusted to produce Np^{4+} and Pu^{4+} using a conventional method (ferrous sulfamate followed by heating, or adding sodium nitrite) and after adjustment was transferred by vacuum into the feed tank. The feed was displaced from the feed tank by pumping ∿8M HNO_3 into the tank at a flow of up to 80 ml/min. After sorption, the resin was washed with 2 liters of ∿8M HNO_3 at up to 80 ml/min. Plutonium was separated with 5.4M HNO_3-0.05M N_2H_4-0.05M $Fe(NH_2SO_3)_2$ at a flow of up to 50 ml/min. Then neptunium was eluted with 0.25M HNO_3 at ∿10 ml/min.

The initial three tests in the pressurized system were made with ^{239}Pu to verify system response at relatively low rates of radiolysis and gassing. The feed solutions contained ∿3.3 g ^{237}Np and ∿0.9 g ^{239}Pu. All other tests (Table I) were with ^{238}Pu present.

Analyses for neptunium and plutonium were done by TTA extraction and alpha pulse height techniques. Acid analyses were by titration with standard base.

TABLE I. ^{237}Np-^{238}Pu PARTITION TEST RESULTS

	Resin			Flow, ml(min-cm^2)				Impurity in Product, wt %	
	Dowex		Pressure Drop Across			Elution of			
Test	Type	Mesh Size	Column, psig	Sorption	Wash	Pu	Np	Np in Pu	Pu in Np
1[a]	1-X4	∿190	55	15	15	6	2.5	<0.01	0.12
2[a]	1-X4	∿190	120	15-25	25	9	2.5	0.02	0.24
3[a,b]	1-X4	∿190	130	15-25	25	6	2.5	0.01	0.94
4	1-X4	∿190	30-150	15-25	25	6	2.5	0.19	0.52
5	1-X4	∿240	30-150	15-25	--[c]	6	2.5	0.08	0.91
6	1-X4	∿240	10-55	6	--[c]	2.5	2	0.20	0.64
7	1-X4	∿280	100-350	15-25	--[c]	6	2	0.13	1.29
8	1-X2	∿170	10-80	15-25	25	6	2.5	0.81	6.7
9	1-X2	∿150	0-30	15-25	25	6	2.5	13.1	10.3
10	1-X2	∿140	<5	7	--[c]	2.5	2	0.07	0.28
11	1-X2	∿140	50	15-25	--[c]	6	2.7	1.36	0.88
12	1-X8	∿260	10-20	15-25	--[c]	2.4	2	0.02	12.36
13[d]	MSA-1	∿200	10-90	15-25	--[c]	6	2	0.09	0.12
14[d]	MSA-1	∿260	20-110	15-25	--[c]	6	2.5	0.12	0.05

---

a. ^{239}Pu.
b. 55°C; all other runs ∿23°C.
c. Wash omitted.
d. Macroporous resin; all others gel-type.

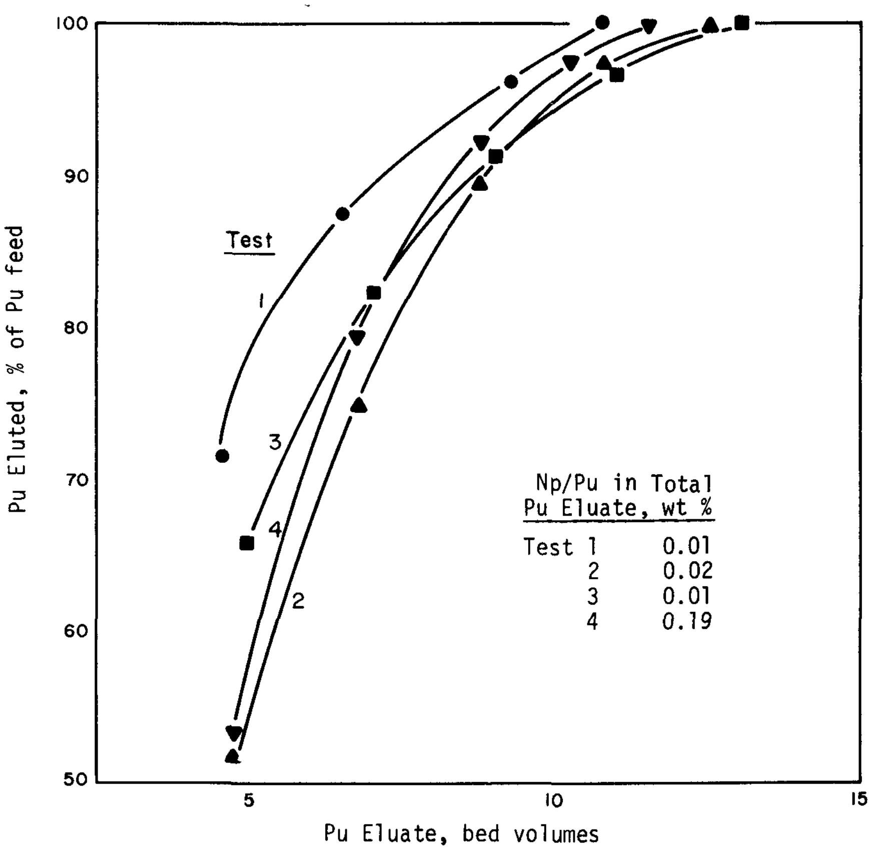
RESULTS

Prior demonstrations of plutonium-neptunium partitioning using *Dowex* 1 resins in gravity-fed columns showed that separation of these radioisotopes was much more effective with 2 or 3 percent crosslinked resins, e.g., *Dowex* 1-X2, than with resins containing a higher percent divinylbenzene.[2] However, flow tests with 8M HNO_3 in the pressurized system showed that the structural stability of *Dowex* 1-X2 limited the minimum particle size and/or flow rate because the resin deformed and caused excessive pressure drops. *Dowex* 1-X4, ∿190 mesh, which is more resistant to deformation under pressure, did not cause excessive pressure drops, and separation characteristics were very good in the pressurized system. *Dowex* 1-X8, a hard, highly cross linked resin was the strongest resin tested but has the slowest ionic diffusion. *Dowex* MSA-1, a macroporous resin, was found to have outstanding plutonium and neptunium sorption and elution properties. This resin is copolymerized under special conditions to yield small, tough beads with discrete pores that facilitate diffusion of large ions (in contrast, *Dowex* 1 resins have a relatively nonporous gel structure).

Test results are summarized in Table I and Figures 2 and 3. The figures show the percent of plutonium eluted from the resin as a function of the volume of plutonium elutriant [5.4M HNO_3-0.05M N_2H_4-0.05M $Fe(NH_2SO_3)_2$].

The pressurized system permitted actinide loading rates of 3 to 5 times those used for current plant operations with 40 to 60 mesh *Dowex* 1-X2 resin. Excellent separation of plutonium was attained at flows ∿3 times those presently used in the gravity-fed plant columns. Separation of ^{238}Pu and neptunium was somewhat less effective than separation of ^{239}Pu and neptunium. This has been noted previously and is attributed to solution radiolysis and the consequent effects on the oxidation states of neptunium and plutonium.[2]

Effective separation of actinides was obtained with three of the four *Dowex* resins tested, although several deficiencies were noted. *Dowex* MSA-1 resin was most effective, followed by *Dowex* 1-X4 and *Dowex* 1-X2. *Dowex* 1-X8 was least effective.



Resin: 125 ml *Dowex* 1-X4, 200 to 400 mesh

Pu Elutriant: 5.4M HNO_3-0.05M N_2H_4-0.05M $Fe(NH_2SO_3)_2$

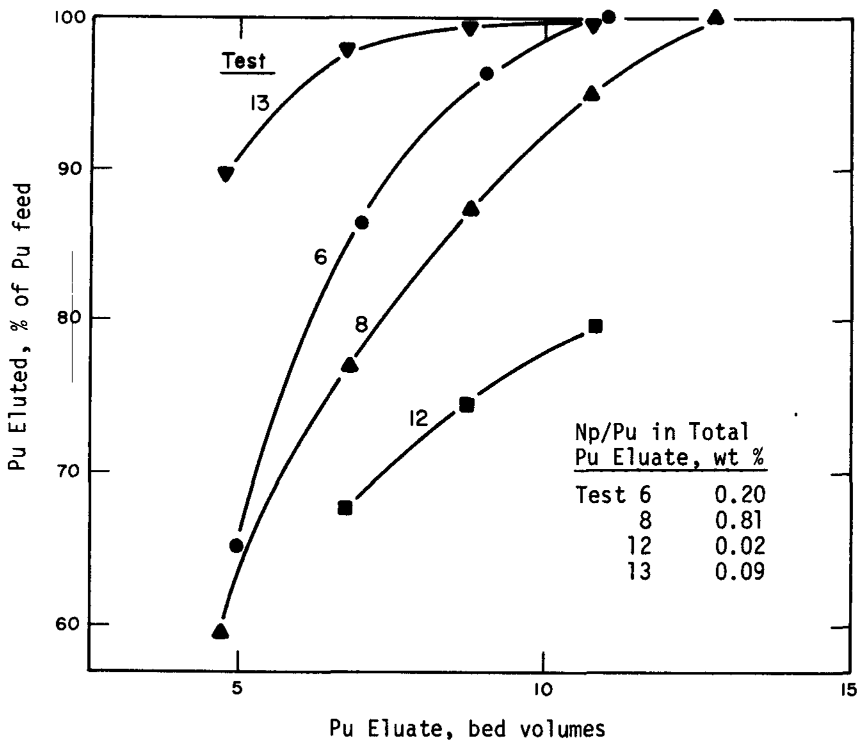
Test 1 2.5 g ^{237}Np, 0.5 g ^{238}Pu; 23°C; 55 psig
Feed 15 ml/(cm^2-min)
Elution 6 ml/(cm^2-min)

Test 2 3.3 g ^{237}Np, 0.9 g ^{239}Pu; 23°C; 120 psig
Feed 15-25 ml/(cm^2-min)
Elution 9 ml/(cm^2-min)

Test 3 3.3 g ^{237}Np, 0.9 g ^{239}Pu; 55°C; 130 psig
Feed 15-25 ml/(cm^2-min)
Elution 6 ml/(cm^2-min)

Test 4 4.3 g ^{237}Np, 1.2 g ^{238}Pu; 23°C; 30-150 psig
Feed 15-25 ml/(cm^2-min)
Elution 6 ml/(cm^2-min)

FIGURE 2. SEPARATION OF $^{238,239}Pu$ FROM ^{237}Np WITH *DOWEX* 1-X4 RESIN IN A PRESSURIZED ANION EXCHANGE COLUMN



Pu Elutriant:	$5.4M\ HNO_3-0.05M\ N_2H_4-0.05M\ Fe(NH_2SO_3)_2$	
Test 6	4.1 g ^{237}Np, 1.1 g ^{238}Pu; 23°C; 10-55 psig	
Dowex 1-X4	Feed	6 ml/(cm^2-min)
	Elution	2.5 ml/(cm^2-min)
Test 8	3.4 g ^{237}Np, 1.3 g ^{238}Pu; 23°C; 10-80 psig	
Dowex 1-X2	Feed	15-25 ml/(cm^2-min)
	Elution	6 ml/(cm^2-min)
Test 12	4.0 g ^{237}Np, 1.2 g ^{238}Pu; 23°C; 10-20 psig	
Dowex 1-X8	Feed	15-25 ml/(cm^2-min)
	Elution	2.4 ml/(cm^2-min)
Test 13	4.2 g ^{237}Np, 1.3 g ^{238}Pu; 23°C; 10-90 psig	
Dowex MSA-1	Feed	15-25 ml/(cm^2-min)
	Elution	6 ml/(cm^2-min)

FIGURE 3. COMPARISON OF *DOWEX* RESINS FOR SEPARATING ^{238}Pu FROM ^{237}Np IN A PRESSURIZED ANION EXCHANGE COLUMN

Dowex MSA-1 (Macroporous) Resin

Tests with *Dowex* MSA-1 resin gave excellent separation of plutonium from neptunium,* yielding contamination values of ∿0.1 wt % of one element in the other. Performance was much better with macroporous resin than with the gel-type resins. Rapid flows for sorption, plutonium elution, and neptunium elution were maintained (Table I and Figure 3) without excessive pressure drop or other deleterious effects.

Dowex 1-X4 (Gel) Resin

This resin was quite effective, separating neptunium and plutonium well within the specified upper limit of 1 wt % of each product in the other. Performance over a range of operating conditions was determined, as shown in Table I and Figure 2. As expected, the smaller the particle size, the greater the system pressure necessary to achieve adequate flow. A side effect, less effective separation, was found as the system pressure was increased. This effect of increasing system pressure, seen also with *Dowex* 1-X2, indicated that deformation of the resin particles was occurring.

Dowex 1-X2 (Gel) Resin

Separation of neptunium and plutonium using *Dowex* 1-X2 resins was not adequate at flow rates requiring >50 psig pressure. Much better separation was usually obtained with 2% crosslinked resin than with 4% (*Dowex* 1-X4) at low pressure with larger particles. The reduced effectiveness is believed to be caused by the deformation of the lower crosslinked resin under pressure.

The initial charge of *Dowex* 1-X2 for testing pressurized column operating characteristics was ∿280 mesh, and even at 5 cc/(min-cm^2) flow with 8M HNO_3, the resin deformed and caused unacceptably high pressure. Separation of plutonium and neptunium was thus not attempted. At flow rates of ∿25 cc/(min-cm^2) for feed sorption and washing, the minimum useable size of *Dowex* 1-X2 resin was ∿170 mesh. Smaller particles became packed in the column as solution was pumped, causing the pressure to increase. Once the packing (and deformation) occurred, decreasing the flow

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* Because the small-particle macroporous resin gave such outstanding results in the pressurized system, larger particle macroporous resin was tested in a low pressure column and results were excellent. The results with larger particle macroporous resin are the subject of another report.[5]

rate did not cause reduction of pressure, and the tests were terminated.

Dowex 1-X8 (Gel) Resin

Dowex 1-X8 resin (∿260 mesh) gave very poor separation of plutonium from neptunium even though the partition wash was done at the low rate of 2.5 ml/(min-cm^2). This resin is mechanically stronger than X2 or X4 resin; however, the ionic diffusion is decreased. This confirms the very ineffective separation demonstrated previously in low pressure systems with 40 to 60 mesh *Dowex* 1-X8 resin.[2] The pressure necessary during feed sorption was 5 to 10 times lower than with X4 resin, confirming the structural stability of this resin which is attributed to the higher degree of crosslinking.

APPENDIX A. SAFETY ANALYSIS

ABSTRACT

Potential hazards that exist with nitric acid anion exchange systems are discussed. Safeguards are defined for a system that was operated at the Savannah River Laboratory. In addition, three maximum credible accidents in the proposed system are analyzed.

POTENTIAL HAZARDS

Seven failures of anion exchange resin systems have been reported in nuclear work since 1962, and five studies have been made of the thermal stability of nitrate-form resins. The system failures and results of the resin investigations have been summarized, and the conclusions which follow were abstracted from this summary.[6]

Four factors were present in all the reported failures: (1) resin in the nitrate form, (2) $\geqslant$7M nitrate ion concentration, (3) some source of heat, and (4) ion exchange resin of low thermal conductivity. The three sources of heat identified were the heat of reaction between nitric acid and the ion exchange resin, radioactive decay, and heat of dilution of nitric acid. In two cases, the temperatures of the columns were thermostatically controlled: one at 80°C, another at 85°C; the remaining cases concerned columns operated at ambient temperature. In six cases, the resin bed was static for more than 1 hour with the nitric acid concentration $\geqslant$7M. It was postulated that during these long contact periods the beds were heated to ignition by the various heat sources. The maximum safe operating temperature has not been defined, but it may be as low at 80 to 90°C.

Dry nitrate-form resin seemed to be a factor in only one occurrence, a resin spill in a remote-control operation. Tests on vented resin, i.e., not resin in columns, showed that the minimum ignition temperature was 135°C for plutonium-loaded resin and higher for nitrate-form resin.

The hazards outlined above can be avoided by design and operating procedures. In the study, resin was placed in the column in the nitrate form, so that regeneration would not be required with its attendant heat evolution. Flow was interrupted only to change solutions. The resin bed was not permitted to stand in the 8M HNO_3 condition. Another pump was available should pump failure occur. About 10 minutes was required to change pumps, during which time the average temperature increase would be 2°C. The column diameter was small, with <1°C centerline-to-wall ΔT. The maximum operating temperature was 55°C.

Cold tests with nitric acid were run before the neptunium-plutonium partitioning tests were begun. The first partitioning tests were done using ^{239}Pu. When these were accomplished without difficulty, tests with ^{238}Pu were performed.

Spent resin was converted to the hydroxide form before disposal to eliminate the possibility of a fire should the resin dry out. Conversion was done outside the column with 0.25M NaOH. Resin spills were promptly cleaned up, and the resin bed itself was not permitted to run dry. The bed was vented when not in use to prevent pressure buildup from any source.

The planned maximum operating pressure was 350 psig. If a small pressure excursion had occurred, the relief valves set at 500 psig would have opened (see Figure 1). If a large pressure excursion (e.g., a column deflagration) had occurred, the 500 psig relief valves would have been inadequate; in this case the rupture disc would have relieved at 700 psig.

CREDIBLE ACCIDENTS

The possibility of system failure and the spread of radioactivity into unshielded areas was analyzed. There are three credible accidents:

1. Anion Exchange Resin Fire

The most severe accident is a column deflagration, causing expulsion and ignition of actinide-loaded resin. This could result from the heat of reaction of 8M HNO_3 and anion exchange resin. Because of the relatively large volume of the glove box and the small column volume, rupture of the absolute filter in the box by a deflagation and fire was not considered credible. Although new filters are guaranteed by the vendor to retain 99.97% of particulate matter, 99.9% efficiency was used in this analysis as a retention value for the filtration system (two filters in series).

The maximum test batch size was 2 g ^{238}Pu and 5 g ^{237}Np. The low activity ^{237}Np was neglected in the following analysis. Ten percent of the ^{238}Pu was assumed to be released to the glove box during an anion exchange fire, and the filters were assumed to retain at least 99.9% of the material,[7] thereby releasing 0.2 mg ^{238}Pu from the stack. Using generally the same method for estimating atmospheric diffusion as reported by Singer, et al.[8] and Allen and Courtney,[9] the following maximum doses to onsite and offsite population would result:

	Dose, rem			
Distance from Stack →	100 m	700 m	4000 m	8000 m
Dose to bone (70 years)	0.75	0.25	0.09	0.04
Dose to lung (7 days)	2.8×10^{-4}	1.5×10^{-4}	0.6×10^{-4}	0.4×10^{-4}

The anion exchange system was designed to prevent airborne contamination from an accident such as that described. Column temperature was controlled by a constant-temperature, circulating-water bath. The rupture disc on the column relieved into a 1-inch stainless steel tube leading to an 8-liter container; several inches of water were placed in the container to quench expelled burning resin. *Halon* fluorocarbon extinguishers* were installed on this glove box.

Written operating procedures were followed carefully. The column was not permitted to stand in the actinide-loaded and/or concentrated acid condition. The glove box filtering system was subjected to a penetrometer test, and the system filters retained the required 99.97% of particulate matter.

A fire originating with a dry nitrate-form resin was not considered to be a credible accident. Any spill of resin was cleaned up. Also, reconditioning the resin with hydroxide before discarding ensured that nitrate-form resin would not dry and ignite in waste containers.

2. Backup of Radioactive Solution into Operating Area

Backup of radioactive solution into the operating area could occur from failure of the pump check valve while the system was pressurized. Feed solution, $\sim 10^{11}$ dpm/ml, could contaminate the pump, and radiation exposure in the area would be <100 mR/hr from the low energy ^{238}Pu gamma.

This occurence was prevented by a check valve in the line leading to the pump. Similar check valves were used previously in similar facilities and were found reliable for this purpose.

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*Product of E. I. du Pont de Nemours and Co., Wilmington, Del.

In addition, a gamma monitor with an audible alarm was located on the cold feed line which entered the glove box. Although the amount of gamma radiation penetrating the stainless steel tubing would be small, it should be adequate to activate the alarm.

3. Rupture of Pressurized System

Failure of the system could be caused by overpressurization or by failure of column components. This accident is similar to 1 above, except in this case a fire is not postulated.

Although the system is essentially liquid, gas could be present in the column due to liquid degassing or the nitric acid-resin reaction. Radiolytic gassing is discounted because of the small amounts and moderate activity of the actinides being used. Rupture of the system and gas expansion could expel liquid and resin from the column. This would not significantly increase the air pressure in the box but could cause airborne activity, some of which might penetrate the filters.

Such an accident could result in an aerosol containing perhaps 10% of the actinides in a test batch. Again neglecting the low level ^{237}Np, the glove box air would contain 0.2 g ^{238}Pu. Assuming 20% of the ^{238}Pu reaches the filtering system (postulated to be 99.9% efficient), then 0.04 mg ^{238}Pu would be released. The resulting exposures would be one-fifth of those calculated for accident 1.

The test system contained several design features to prevent an overpressurization accident. One relief valve was installed on the line between the feed tank and column, and another between the pump and the first valve to prevent pressure buildup. The rupture disc assembly on the column would vent the column should a deflagration occur. The only credible missile in such an occurrence would be the rupture disc which could be blown into and through the 1-inch-diameter tube leading to the 8-liter steel container.

The system was hydrostatically tested to 1000 psig, without the rupture disc assembly and relief valves, to qualify for 700 psig use. In the extremely remote possibility that some part would fail and become a missile, 1/2-inch-thick polymethylmethacrylate shielding was placed around the system. Movable shields were employed to permit access to the equipment.

APPENDIX B. RESIN GRADING

Decreasing the size of ion exchange resin particles results in an increase of operating pressure. This effect can be partially reduced by using particles in a narrow size range, which ensures a maximum void volume in the packed resin.

Wire sieves do not adequately size resin particles smaller than ∿350 mesh.[10] Such particles can be separated into size fractions by taking advantage of their settling velocity in fluid media. A solid particle settles in a fluid at a velocity that is dependent on its size and density as well as on fluid properties. A particle released into a fluid having a density less than that of the particle will approach a constant velocity that is referred to as the Stokes settling velocity, or the terminal velocity. The terminal velocity of a sphere is defined as[11]

$$V_t = ga^2(\rho_p - \rho_f)/18\mu$$

where: V_t = terminal velocity

g = acceleration due to gravity

a = particle diameter

ρ_p, ρ_f = densities of the particle and fluid, respectively

μ = viscosity

In hydraulic grading, the solid particle is subjected to an upward fluid velocity. If the fluid velocity is greater than the terminal velocity of the particle, the solid particle will be carried along with the fluid stream. Sized fractions can be obtained by varying the flow rate. This type of hydraulic grading was employed to grade resins used in this study. The ease of grading varies with the percent crosslinkage with MSA-1≈8>4>>2. The 8% material graded easily (∿1 week required). The time required for grading the 4% material varied form 1 to 3 weeks. Grading of 2% material was ineffective, with only very fine or very large particles removed after five weeks.

The grading system comprised an interconnected series of five glass columns with 1.5-, 2-, 2.5-, 3-, and 4-in. diameters, 40-in.-long and with conical sections 7-, 8-, 9-, 11-, and 16-in.-long, respectively. A positive displacement pump delivered 385 ml/min at full stroke. A surge tank reduced flow variations from pumping. *Teflon** stopcocks at the top and bottom of the columns permitted resin loading and unloading. The stopcocks on the column side arms permitted individual column isolation during loading and unloading. A schematic of the grading system is shown in Figure 4.

Small quantities of resin were placed in the 1.5-in.-diameter column. Larger amounts (1 lb max) were placed in the surge tank and displaced to the columns. The flow rate was varied to obtain an adequate size distribution in the columns. Grading efficiency was determined by sampling the resin and measuring the particle size and distribution with an optical comparator.

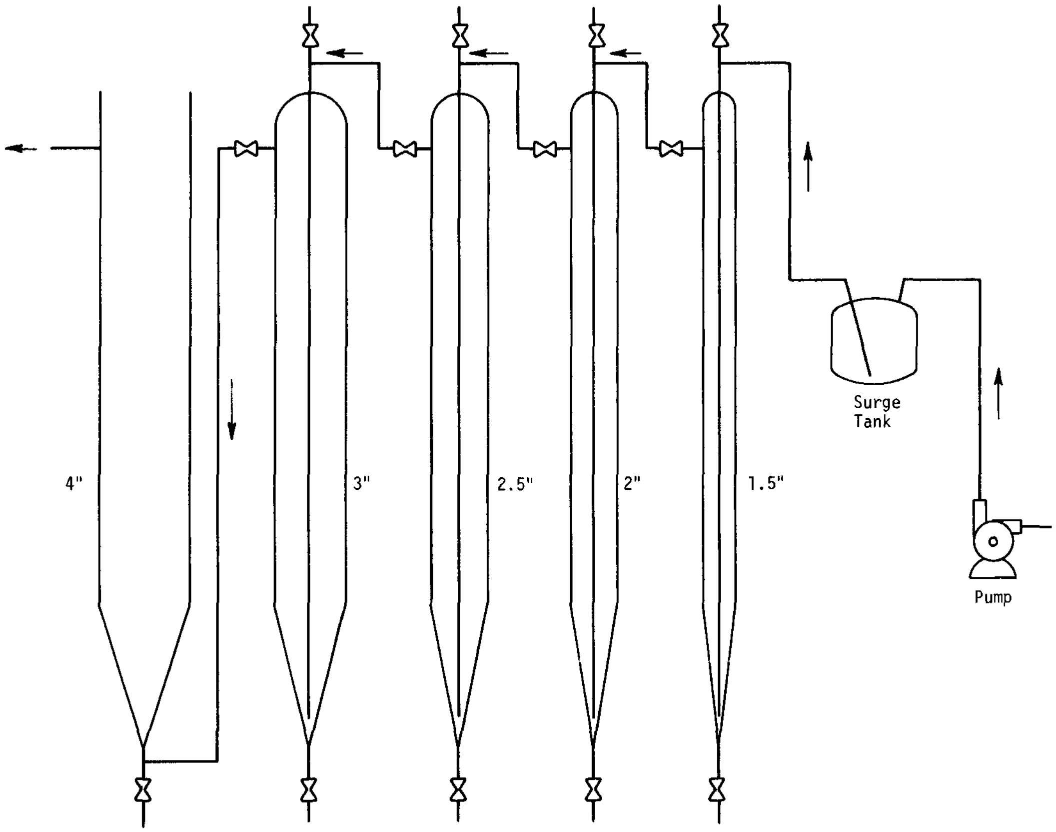

FIGURE 4. SCHEMATIC DIAGRAM OF RESIN GRADING COLUMN SYSTEM

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* Trademark, E. I. du Pont de Nemours and Co., Wilmington, Del.

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