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**MARTIN MARIETTA**

**Recovery, Concentration, and  
Solidification of Europium  
by Ion Exchange from a  
Dilute Process Stream**

D. O. Campbell

OPERATED BY  
MARTIN MARIETTA ENERGY SYSTEMS, INC.  
FOR THE UNITED STATES  
DEPARTMENT OF ENERGY

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Chemical Technology Division

RECOVERY, CONCENTRATION, AND SOLIDIFICATION OF EUROPIUM  
BY ION EXCHANGE FROM A DILUTE PROCESS STREAM\*

D. O. Campbell

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RECOVERY, CONCENTRATION, AND SOLIDIFICATION OF EUROPIUM  
BY ION EXCHANGE FROM A DILUTE PROCESS SOLUTION

D. O. Campbell

ABSTRACT

Experimental studies were carried out to develop a process to recover americium from a very dilute waste solution and convert it to a stable solid suitable for waste disposal. A promising process is to load the americium onto organic ion-exchange resins and burn the resins to yield a granular solid that can be disposed of directly or formed into a monolith using known solidification processes.

Experiments were carried out in which europium, a stand-in for americium, was loaded onto small ion exchange resin columns from dilute aqueous solutions containing nitric acid and, in some cases, iron. Breakthrough curves were obtained for conditions appropriate for a process. The effluent solution was decontaminated sufficiently to be a non-TRU waste in the plant application. The volumetric distribution coefficient under anticipated conditions was in the range of 18,000, indicating that a relatively very small volume of solid, americium-loaded, ion-exchange resin would be produced (probably less than 1 L/d). Burning the loaded resin in air yields a stable, solid oxysulfate with a further volume reduction of about a factor of six.

Iron, and possibly other impurities, can interfere with the americium recovery; thus, additional work is required to resolve such problems and to devise a reasonably optimized process. The engineering of the resin-burning equipment also requires further attention.

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1. INTRODUCTION

The Plutonium Finishing Plant (PFP) at Hanford generates significant volumes of waste containing  $\alpha$ -active transuranium (TRU) elements. Consideration is being given to applying Transuranium Extraction (TRUEX) technology to remove the TRU elements from acidic wastes so that they can be managed more efficiently. The dominant  $\alpha$ -active element, aside from plutonium, is americium; and it is this

element which typically causes liquid wastes to be TRU-wastes. The present study was directed to developing a method to recover and concentrate the americium into a small volume and isolate it as a solid product suitable for final waste management. This work was initially based on an evaluation of process options<sup>1</sup> which concluded that cation exchange was most promising.

The TRUEX technology,<sup>2</sup> developed at Argonne National Laboratory, is based on liquid-liquid extraction from an acidified nitrate salt solution into a bidentate organophosphorous reagent. The reference extractant for this application is phenyl(octyl)-N,N-diisobutylmethyl-carbamoylphosphine oxide (CMPO), dissolved in a solution of tri-n-butyl phosphate in  $CCl_4$ . The americium product of this process is a dilute  $HNO_3$  stream containing  $^{241}Am$ , which is the dominant transplutonium  $\alpha$ -emitter in the PFP waste, and the only one requiring special attention.

In the initially proposed flowsheet, the americium product stream was projected to be ~150 L/h with a composition of ~0.01 M  $HNO_3$  and 3.5 mg/L of americium ( $1.4 \times 10^{-5}$  M). The quantity of americium is about 12 g/d. This stream would also contain small amounts of plutonium (about a factor of 100 lower concentration), some other elements such as zinc, and a significant amount of iron. The iron concentration will depend on the way the TRUEX process is operated, so is not defined at this time; but the effect of iron was one of the variables to be studied. There are flowsheet variations that could decrease the volume and increase the concentrations in the product stream by significant factors.

Two requirements that greatly influence any process are (1) the specifications of the final americium product from the recovery operation, and (2) the americium and  $\alpha$ -content of the raffinate solution after treatment by the process. At this time neither has been defined. The required properties of the final americium product have not been specified, because the disposition of the TRU-waste is not yet determined. It is assumed that a stable solid of reasonably low volume will be satisfactory, since, if necessary, it can be further processed fairly easily (i.e., converted to concrete, glass, cermet, or some other solid).

The extent to which americium must be removed from the aqueous waste stream is also not defined. It is assumed that a TRU  $\alpha$ -limit for the raffinate from the americium removal process will be required to be in the range of 10 to 100 nCi/g, but it is not clear what the gram unit refers to. Depending on circumstances, it could be the solution, some solid waste (such as concrete) produced by solidification of the solution, or only the dissolved solids left after evaporation of the water; these options differ by large factors. For comparison, 100 nCi of Am per mL of solution corresponds to  $2.9 \times 10^{-5}$  g/L or  $1.2 \times 10^{-7}$  M americium; this is approximately 1% of the feed concentration. An analytical sensitivity of 0.1% of the feed concentration was specified to obtain useful data in the required range.

## 2. EXPERIMENTAL METHODS

In all cases except one, the ion-exchange columns were made from 1-cm-diam Pyrex glass tubing containing a glass frit (70- to 100- $\mu$ m porosity) to support a resin bed 2 cm long, giving a bed volume of 1.57 mL. In the first test, the column consisted of a 1.5-cm-long resin bed of 1-mL volume, supported between Ottawa sand layers in a 25-mL buret. Dowex 50, X-8 resin, H<sup>+</sup>-form, 50 to 100 mesh (300 to 150  $\mu$ m), was used for all the experiments except one, in which 20- to 50-mesh (840- to 300- $\mu$ m) HCR-S resin was used. The res. . bed volume decreased during the loading cycle. The resin was washed with 3 M nitric acid and then water before starting a loading experiment.

The feed solutions were pumped to the open tops of the columns at the specified flow rate, and the solution found its own level to maintain that flow rate. The columns were jack-legged to assure that the resin bed was always flooded. The raffinate was collected in successive bottles, and the volumes were measured. Samples were taken for analysis either from the bottles or from the flowing raffinate at known throughput volumes.

Feed solutions contained ~3 mg/L of stable europium in either 0.1 or 0.15 M HNO<sub>3</sub>. Tracer europium was also used in nearly all the

experiments, and iron was added in one set. An attempt was made to analyze the solutions with atomic absorption (AA), but this proved unreliable at the low concentrations of the raffinate solutions before substantial breakthrough. The sensitivity of the AA method was inadequate to reach the required  $\mu\text{g/L}$  range. Accordingly, tracer europium was added in an amount designed to give the required sensitivity (0.1% of feed).

The europium activity of each sample was determined by counting known volumes (usually 4 mL) of effluent solution with a 3-in. x 3-in., well-type, NaI(Tl) crystal scintillation detector and an LKB Wallac 1282 Compugamma Universal Gamma Counter controlled with a Televideo 912C computer terminal. The largest gamma band for the particular mixture of europium isotopes was used. The concentrations relative to the feed concentration were plotted against the average volumetric throughput of the sample, in apparent bed volumes (the total geometric volume of the resin bed), using log-normal graph paper; this sort of plot causes the breakthrough curves to be nearer to linear. The volumetric distribution coefficient ( $K_d'$ ) was approximated by interpolating or extrapolating the graph to the 50% breakthrough point; this differs from the conventional  $K_d$  (which is based on the mass of resin) by a factor which is the resin density. This  $K_d'$  value is the limiting volume of solution that can be processed, per unit volume of resin, under ideal conditions. Under practical conditions, with columns in series and reasonably long residence times, one can hope to achieve significantly above 50 % of this throughput before excessive breakthrough occurs.

Some experiments were also carried out in which resin, loaded to varying extents with europium, was burned in air to produce a stable solid waste. In all cases, 10 mL of resin was batch-contacted with a specified amount of europium; the resin was washed, dried, weighed, and burned for several hours in air at 700°C. Finally, the weight and volume were determined. Microscopic photographs were taken of several of these preparations.

## 3. ION EXCHANGE RECOVERY

## 3.1 EXPERIMENTAL RESULTS

The ion-exchange column experiments yielded typical S-shaped break-through curves. The main experimental parameters and  $K_d'$  values are summarized in Table 1, and graphs of the breakthrough curves are given in Figs. 1-5. The three runs with 0.15 M acid and no iron gave consistent distribution coefficients of about 18,000, in good agreement with each other. The breakthrough curves were favorably steep in all cases; but, as the residence time decreased (Fig. 3 vs Fig. 1), the initial breakthrough occurred earlier. Unfortunately, the experiment with the highest flow rate (Fig. 2) used AA for analysis, and the early part of the curve could not be measured.

Table 1. Summary of ion exchange loading tests

Run No.	Concentrations			Flow rate (mL/min)	Resin type <sup>a</sup>	Resin volume (mL)	$K_d'$	Note <sup>b</sup>
	Acid (M)	Eu <sup>3+</sup> (mg/mL)	Fe <sup>3+</sup> (M)					
1	0.15	3.0	0	2	50-X8	1.00	18,500	
2	0.15	3.0	0	10	50-X8	1.57	18,000	
3	0.15	3.0	0	4	50-X8	1.57	17,500	
4	0.10	2.94	0.001	4	HCR-S	1.57	1,500	
5	0.10	2.94	0.001	4	50-X8	1.57	2,700	
6	0.10	2.94	0.001	4	50-X8	1.57	3,500	HAN
7	0.10	2.94	0.001	4	50-X8	1.57	>5,000	oxalic

<sup>a</sup>Run 4 used 20-50 mesh HCR-S resin; all others used 50-100 mesh Dowex 50-X8.

<sup>b</sup>Run 6 contained 0.002 M hydroxylamine nitrate which partially reduced the ferric iron to ferrous. Run 7 contained 0.05 M oxalic acid.

As noted above, the effluent concentration probably should be held in the range of 0.1 to 1% or lower if the effluent is to be a non-TRU waste, although this depends on the ultimate fate of that effluent. It is characteristic for a small fraction of the feed activity to leak through a column, even at the beginning. In most cases this amounted to between 0.1 and 0.01% of the feed concentration, which is

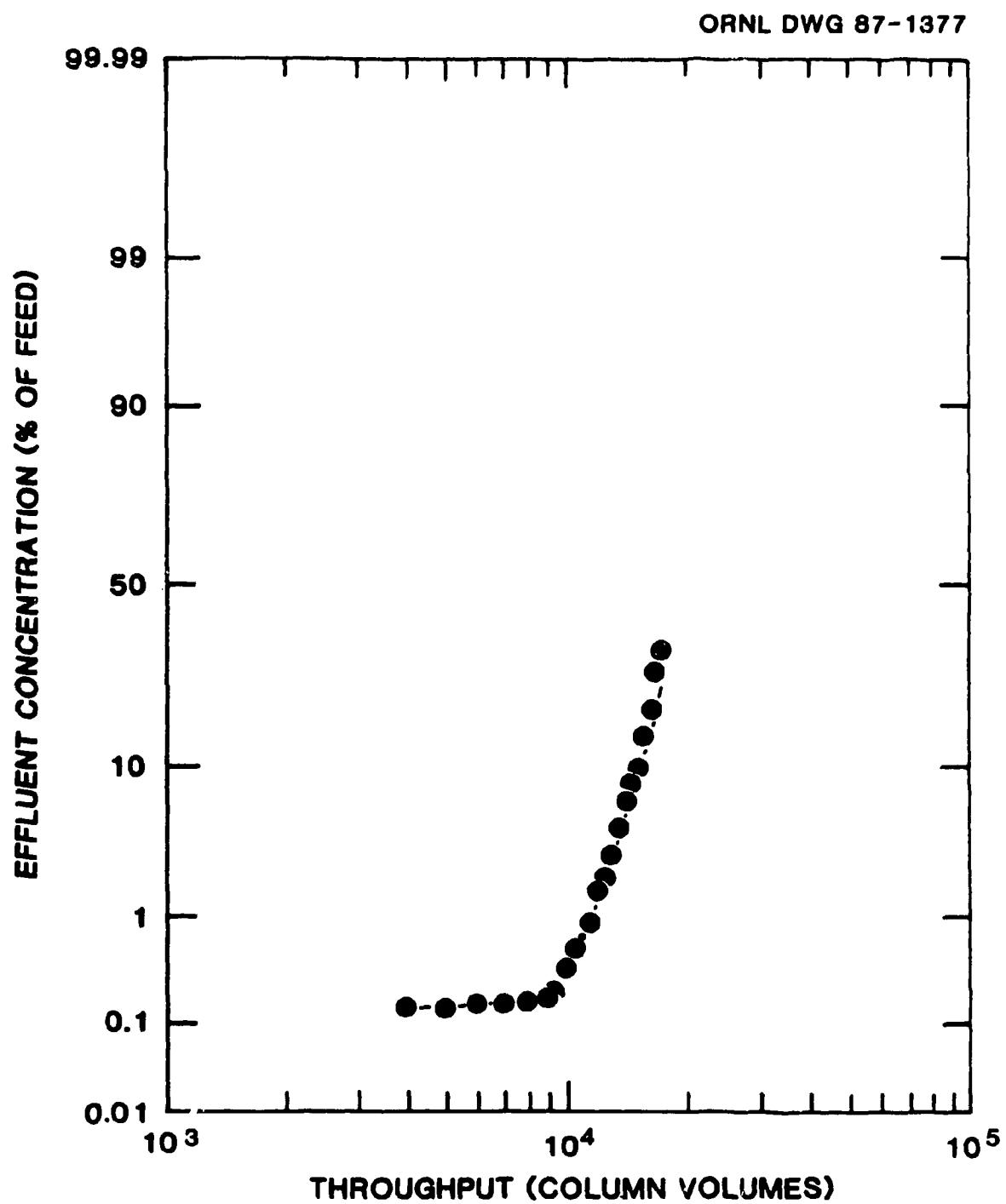


Fig. 1. Europium breakthrough curve for 30-s residence time.  
Feed: 0.15 M HNO<sub>3</sub>, 3 mg/L Eu; Resin: 50-100 mesh Dowex 50-X8.

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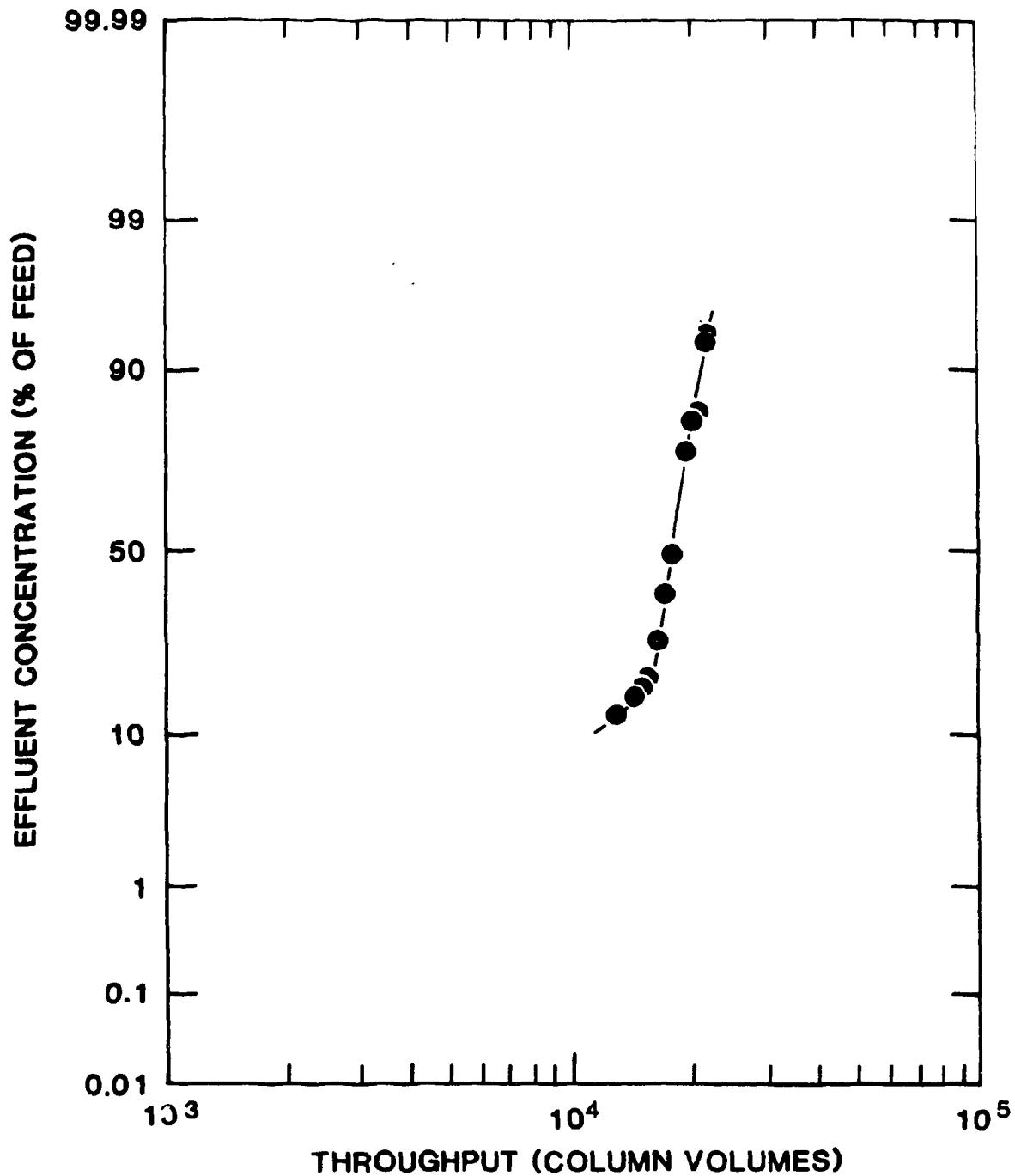


Fig. 2. Europium breakthrough curve for 9.4-s residence time.  
Feed: 0.15 M HNO<sub>3</sub>, 3 mg/L Eu; Resin: 50-100 mesh Dowex 50-X8.

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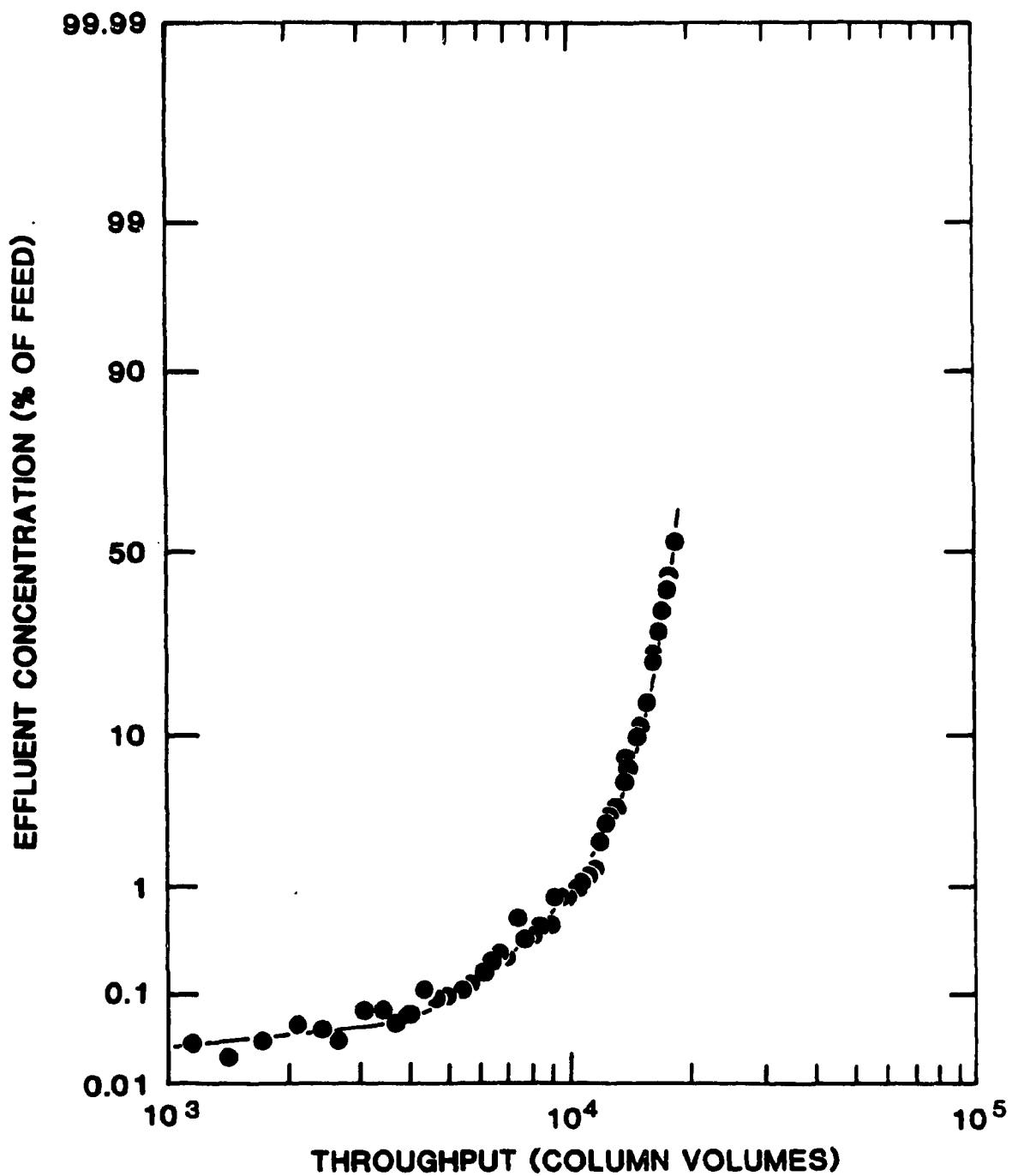


Fig. 3. Europium breakthrough curve for 23.6-s residence time.  
Feed: 0.15 M HNO<sub>3</sub>, 3 mg/L Eu; Resin: 50-100 mesh Dowex 50-X8.

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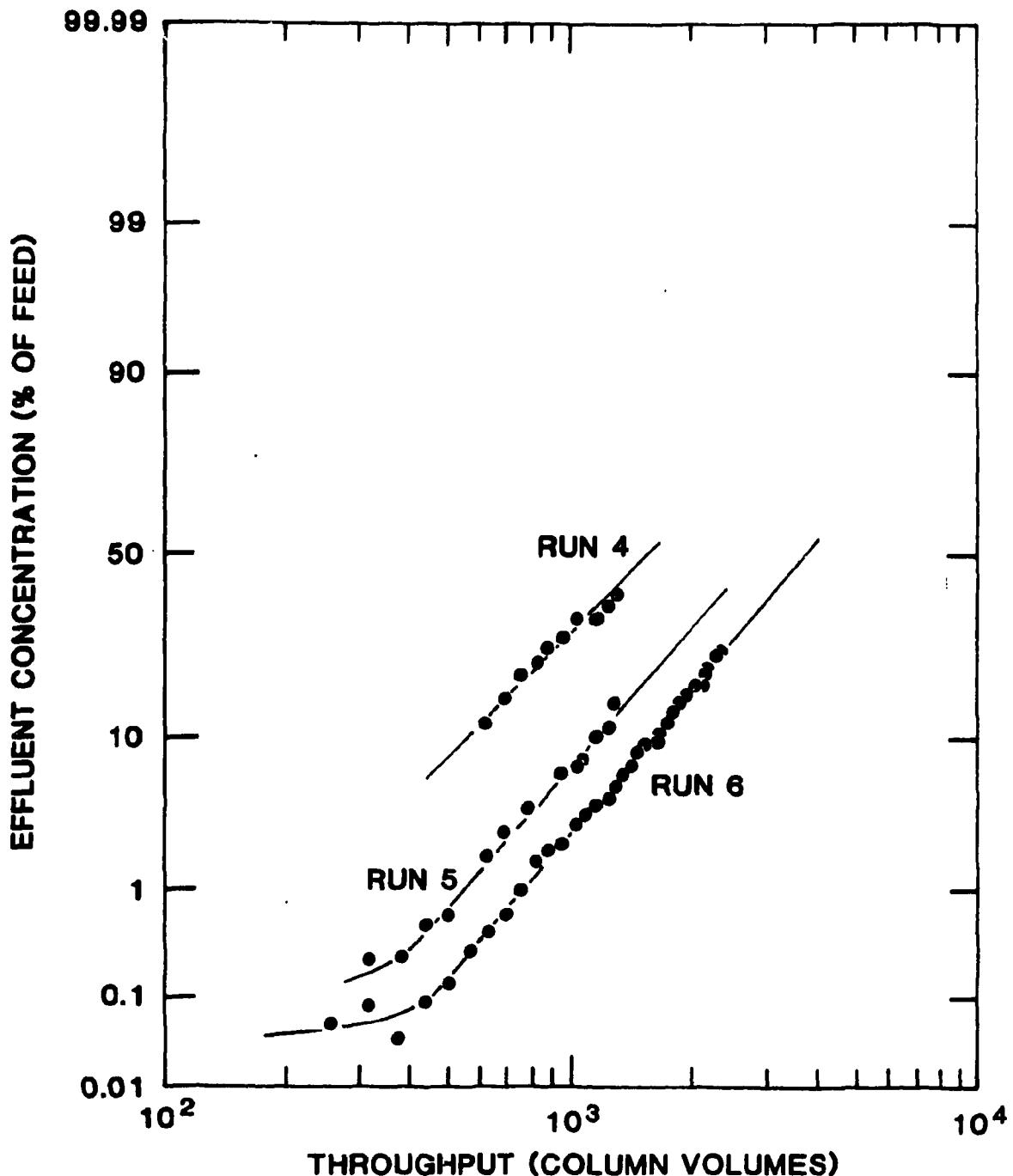


Fig. 4. Europium breakthrough curves with iron present.  
Residence time 23.6 s; feed: 0.10 M HNO<sub>3</sub>, 2.94 mg/L Eu, 0.001 M Fe<sup>3+</sup>,  
0.002 M hydroxylamine nitrate added for run 6; resin: 20-50 mesh HCR-S  
in run 4, 50-100 mesh Dowex 50-X8 in runs 5 and 6.

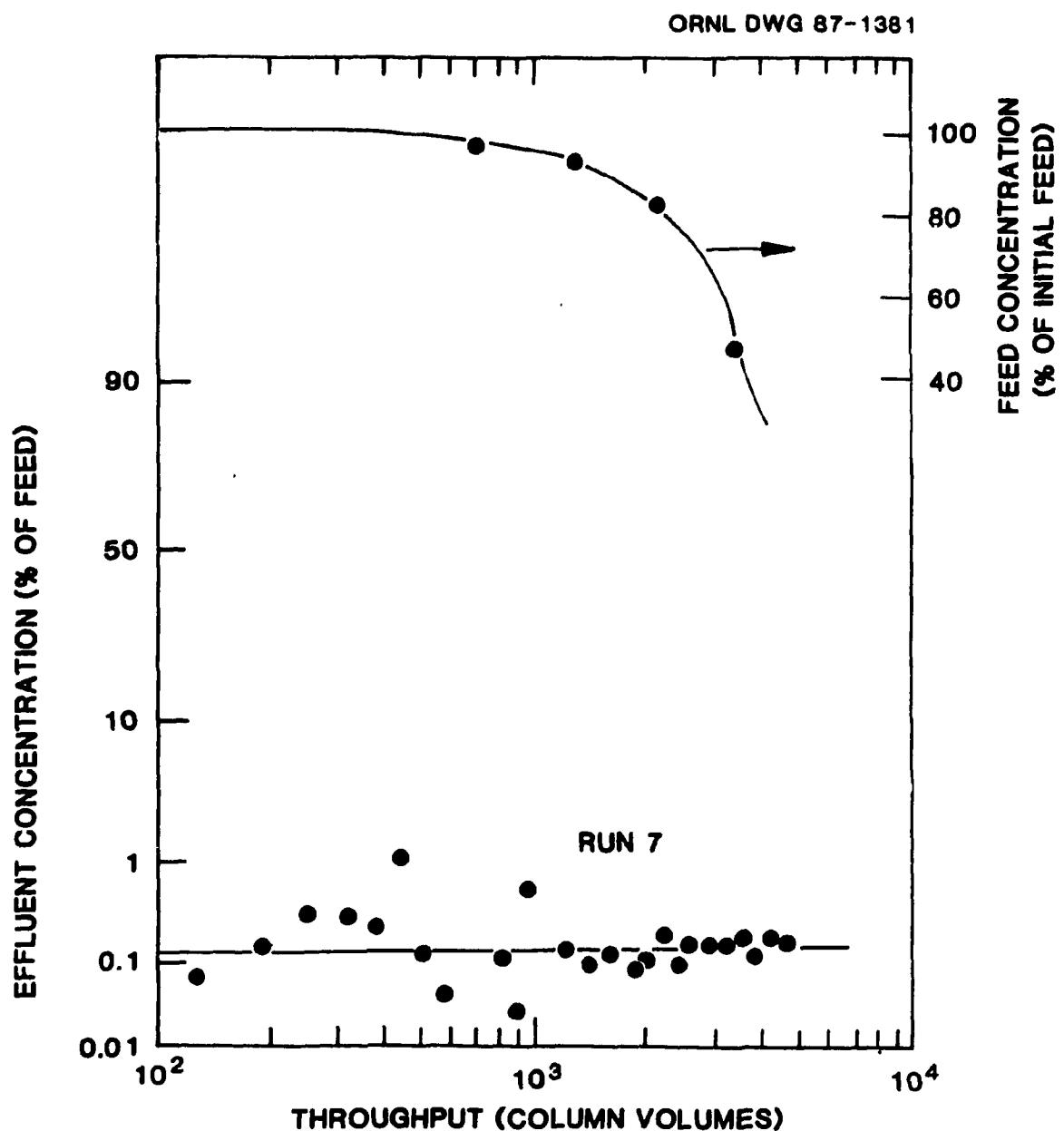


Fig. 5. Europium breakthrough curve with iron and oxalic acid present. Feed: 0.10 M HNO<sub>3</sub>, 2.94 mg/L Eu, 0.001 M Fe<sup>3+</sup>, 0.05 M oxalic acid; resin: 50-100 mesh Dowex 50-X8.

satisfactory. The relatively constant leakage continued for some time, and then an abrupt increase in slope occurred as the true breakthrough started. In practice, plant-scale equipment, operated in a careful and clean manner, often gives lower leakage prior to breakthrough than the small test columns.

During the course of this work the TRUEX flowsheet was tentatively revised, with the result that the americium product composition was changed. The acidity was reduced to 0.10 M, the americium concentration was increased, and iron was projected to be present at a concentration possibly as high as 0.001 M. Based on published related data,<sup>3</sup> a very rough estimate was made regarding iron interference. Ferric iron was expected to interfere with europium loading in a manner such that europium would break through to 50% of the feed concentration after passing less than 10 times the volume required to saturate the resin with iron. Since 0.001 M iron would saturate the resin after about 600 bed volumes, europium breakthrough might then be expected after treating a volume considerably less than 6000 bed volumes. Similarly, if the iron was reduced to ferrous, it would interfere less, by a factor of about five, which places the column capacity in the range observed with no iron present; thus, ferrous iron should have little effect on resin capacity.

The effect of iron was investigated with a series of experiments (Figs. 4 and 5). In the first two experiments with ferric iron, HCR-S (Run 4) and Dowex 50 (Run 5) resin were used. The Dowex 50 proved superior, but the breakthrough capacity was lower than estimated above, by about a factor of two, which is reasonable agreement for such a rough estimate as this. In one case (Run 6), hydroxylamine nitrate was added at a concentration of 0.002 M to reduce  $Fe^{3+}$  to  $Fe^{2+}$ ; and the solution was allowed to stand overnight before starting the experiment. The  $K_d'$  value increased, but only to 3600, less than expected. However, the resin took on the dark brown ferric color, indicating that much of the iron had not been reduced to ferrous. A more complete reduction of the iron would probably result in a useful resin capacity.

In the final test, oxalic acid was added to a concentration of 0.05 M to complex the ferric iron. This proved very successful, since 5000 bed volumes were processed with no increase in the breakthrough (Fig. 5); clearly,  $K_d'$  is much larger than 5000 in this case. There was another problem however, the oxalic acid concentration was sufficiently large that part of the europium was precipitated. Initially the solid formed a thin layer on top of the resin bed, and the feed concentration decreased slowly by about 10%. After several days, very small crystals appeared in the feed vessel, and the feed concentration finally decreased by about 50%. Periodic feed analyses were made, as indicated in Fig. 5. This problem could be avoided by using a smaller excess of oxalic acid, because the iron complex formation is so strong that iron interference should still be effectively controlled.

The modifications suggested above might have some impact on the TRUEX flowsheet if the effluent solution is recycled within the flowsheet or to the PFP, options that have been considered in some flowsheet variations. A reductant for iron would also probably reduce any plutonium to the (III) state, in which case plutonium would load on the ion exchange resin along with the americium. The reductant could easily be destroyed before recycling the effluent, if that is a desired option. The impact of oxalic acid is less clear, and it should be examined. For example, low concentrations of oxalic acid might improve iron removal in the scrub section of TRUEX, but its effect on plutonium behavior requires definition.

The three curves with iron interference (Fig. 4) show a nearly linear and parallel behavior, with a slope considerably less steep than for Fig. 1-3. This behavior is due to the increased competition from the iron, which causes a smaller europium distribution coefficient once the column is loaded with iron. The result is unfavorable, since a small extent of breakthrough occurs relatively early; so, the usable fraction of the ion-exchange capacity is much smaller (less than a thousand bed volumes) than for the other cases (greater than 10,000 bed volumes).

### 3.2 DATA TREATMENT

It is useful to model the system mathematically for the purpose of scaling performance of the ion-exchange process from the small test columns to larger columns operating under different conditions, and eventually to multiple columns operated in series with countercurrent column transfer. A limited study was done to fit the general Thomas equation<sup>4</sup> to the breakthrough curves of Figs. 1-3. The integrated form of the equation for the special case of reversible second-order kinetics (appropriate to ion-exchange), which was developed by Heister and Vermeulen,<sup>5,6</sup> was used.

The model uses three parameters: (1) a throughput parameter,  $T$ , which is related to  $K_d'$  as defined above; (2)  $N$ , the length of the column expressed as the number of transfer units; and (3) a separation factor,  $R$ , which is expected to be about unity for the process and conditions considered here. Empirically, however, the curves for  $R < 1$  give better fits to the data of Figs. 1-3. Values for  $N$  appear to be in the region of 50 for Runs 1-3, with no large effect from flow rate. In the runs with iron,  $N$  was much smaller, around 10.

These parameters can be converted to new values for specified operating conditions, and the breakthrough curves can then be calculated for those conditions. The parameter,  $N$ , is related to experimental conditions by the equation:

$$N = K_d' K_a (v/f),$$

where  $K_a$  is the mass transfer coefficient,  $v$  the resin bed volume, and  $f$  the feed flow rate. Thus,  $N$  is directly proportional to  $(v/f)$ , which is the residence time. Accordingly, as the ion exchange column size is increased at a given flow rate, or multiple columns are used to give a larger resin volume,  $N$  will increase in proportion to the residence time. With columns as small as the test columns, however, length and diameter can also influence  $N$ ; it is not unusual for  $N$  to be larger with a well-designed plant column than with a small test column with the same bed residence time.

The distribution coefficient,  $K_d'$ , depends on chemical conditions such as the type of resin and concentrations of acid, iron, etc., and is not a function of physical parameters such as flow rate or column dimensions. In this case, it is influenced primarily by acid concentration and, as noted above, is 18,000 for 0.15 M HNO<sub>3</sub>. It can be estimated for small changes in conditions, or it can be measured; but care must be taken that the resin is actually in its equilibrium composition. Several factors can affect  $K_d'$ , notably the resin particle size. For a given separation, the effect of changes in experimental conditions on  $R$  is presumed to be small.

For scaling purposes, it will be necessary to obtain data over a wider range of conditions and, preferably, with larger columns. The expected effect of flow rate on  $N$  was not observed in these tests, but that may be because of the small column sizes and the short residence times. In practice, the residence time may be considerably longer than those used here, but experiments under such conditions would require many days to complete.

### 3.3 APPLICATION

The nature of the process can be visualized by considering an example. The maximum flow rate of the americium product for the reference TRUEX flowsheet is 150 L/h. If an effective resin capacity of 10,000 bed volumes is assumed (which is conservative with a  $K_d'$  of 18,000), the resin requirement for americium recovery is 0.36 L/d. After resin burning (see next section), the reference volume of the solid americium waste would be about 40 to 50 mL/d.

If iron or other impurities are present the volumes might be significantly larger. On the other hand, there are possible modifications of the TRUEX flowsheet, involving reflux within the process, which would yield a several-fold smaller americium product volume, and therefore a smaller resin requirement. The minimum resin requirement, limited by resin capacity, is of the order of 90 mL/d, and the corresponding final waste volume about 15 mL/d.

An example of possible process parameters follows. If the process is operated for 120 consecutive hours (5 full days) per week at 150 L/h, a 2-L ion exchange resin volume should be adequate for the full week. To provide a safety margin, two such columns in series could be used, with the upstream column removed as product after each week of operation, the second column advanced to the upstream position, and a new column placed downstream from it. The resin bed residence time would be 48 s for each column, 96 s total. The column removed from service after each week of operation would be loaded somewhat short of the point at which significant breakthrough should occur, but if breakthrough did occur it would be caught on the second column. After drying and igniting the resin, the initial 2-L volume should be reduced to approximately 0.25 L of granular, solid waste.

#### 4. RESIN IGNITION TESTS

The loaded ion-exchange resin could be converted to a solid appropriate for disposal by a number of approaches, depending on what the final requirements are for the waste form. The resin beads themselves are a possible waste form. Direct modifications include (1) thermal decomposition in the absence of oxygen and (2) ignition with oxygen. Both of these processes have been investigated to varying extents. If a monolithic waste form is required, any of these three products could be incorporated in one or more of a variety of solids, such as concrete, cermets, or glass.

For many years, americium and curium have been prepared in this manner for irradiation in the High Flux Isotope Reactor (HFIR).<sup>7</sup> The americium and curium are loaded on the resin from dilute nitric acid; the resin is dried and burned by heating to 800°C in a quartz ion-exchange column with flowing air; the oxidized particles are treated with 4% H<sub>2</sub> in argon to remove part of the sulfur, and then reoxidized with air, all at 800°C. Finally, the particles are fired at 1050°C before they are mixed with aluminum powder and pressed into pellets for irradiation.

An earlier study,<sup>8</sup> which used neodymium as a stand-in for trivalent actinides, demonstrated that the product over a wide temperature range was  $\text{Nd}_2\text{O}_2\text{SO}_4$ , which decomposed to the sesquioxide ( $\text{Nd}_2\text{O}_3$ ) only above 1000°C. The weight change in the present study with europium is more consistent with a different oxysulfate composition,  $\text{Eu}_2\text{O}(\text{SO}_4)_2$ , but the actual composition was not determined.

Thermal decomposition of loaded resin beads in the absence of oxygen produces a black, free-flowing spherical product; but decomposition products, including copious sulfur-containing organic vapors, could cause some operational problems. The ultimate product is probably an oxysulfate interspersed in a carbon matrix.

Ignition of a layer of resin beads in air yields a grey-colored, free-flowing spherical material with considerably reduced volume; it is primarily an oxysulfate compound of the metals that had been loaded on the resin. During the process, gases, consisting of  $\text{H}_2\text{O}$ ,  $\text{H}_2\text{S}$ ,  $\text{CO}$ , and  $\text{CO}_2$  are evolved as the resin beads are charred black. As the temperature is increased to near 600°C, the beads glow (ignite) and burn slowly, leaving a light ash on the surface; this behavior greatly resembles that of charcoal briquets.

An experiment was carried out to estimate the weight and volume of the product from burning europium-loaded resin (Table 2). In each case

Table 2. Properties of europium-loaded resin after ignition

Exp. No.	Initial quantities		Dry resin weight	After ignition		Weight <sup>a</sup> as $\text{Eu}_2\text{O}(\text{SO}_4)_2$
	Total Eu (mM)	Total resin (mL)	(g)	Volume (mL)	Weight (g)	(g)
1	7.61	10	4.95	1.4	1.47	1.95
2	6.08	10	4.84	1.5	1.45	1.56
3	5.07	10	4.35	1.6	1.27	1.30
4	4.06	10	4.43	1.2	1.07	1.04
5	3.04	10	4.39	1.3	0.78	0.78
6	2.55	10	4.23	1.2	0.64	0.65

<sup>a</sup>Weight of the total initial quantity of europium as the oxysulfate.

10 mL of resin was used, and the europium loading was varied from near saturation of the resin by adding excess europium, to approximately 40% of capacity by adding insufficient europium. After drying at 105°C, the resin weighed between 4.2 and 4.9 g; its volume was not measured because the electrostatic charge made it difficult to handle. However, after ignition, handling presented no problem. The volume was ~15% of the original resin volume for resin loaded to near saturation, and ~12% of the original volume for resin loaded to between 40 and 75% of saturation. Resin in the H<sup>+</sup>-form, with no added europium, left essentially no ash.

The weight of the product corresponded well with the weight of the europium added, calculated as the oxysulfate, Eu<sub>2</sub>O(SO<sub>4</sub>)<sub>2</sub>, except for the first two cases in which excess europium was added. The maximum capacity of the resin, 0.57 mol/L (1.7 meq/mL), limited the europium content to 1.45 g of europium oxysulfate in the first two cases, again in good agreement with the quantity found.

The ignited beads were stable in air, showing almost no weight gain and no change in appearance after standing in water-saturated air at room temperature for several months. There have been reports of rare earth or actinide oxide microspheres decomposing slowly to a powder on standing in air, presumably due to conversion to a carbonate. There was no evidence for this with the europium product prepared here.

Photographs of typical products from highly loaded resin (Exp. No. 1, Table 2) are shown in Fig. 6, and comparable photographs for products from the resin loaded to 40% of saturation (Exp. No. 6) are shown in Fig. 7. A common appearance with the more highly loaded resin (Exps. No. 1 to 4) is a glassy shell with an opaque, ill-defined core. When the beads were crushed, fragments of both materials (thin transparent shell and irregular white core) were observed. The beads from Exps. No. 5 and 6 showed a rougher surface which appeared to be less glassy. They could be crushed very easily, yet they did not tend to break up or form powder during normal handling, pouring, etc.

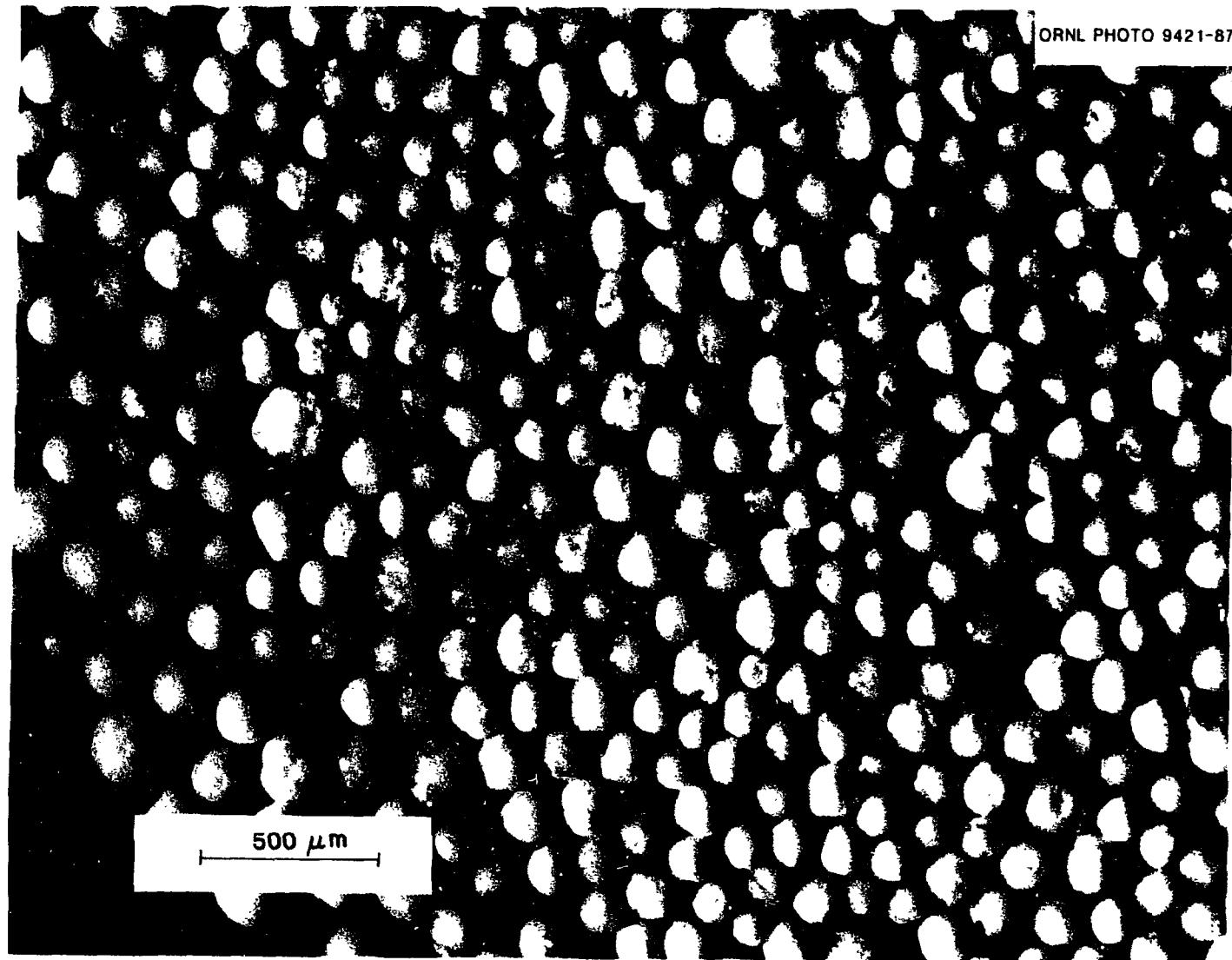


Fig. 6. Photographs of europium oxysulfate product from resin loading experiment No. 1.

ORNL PHOTO 9420-87

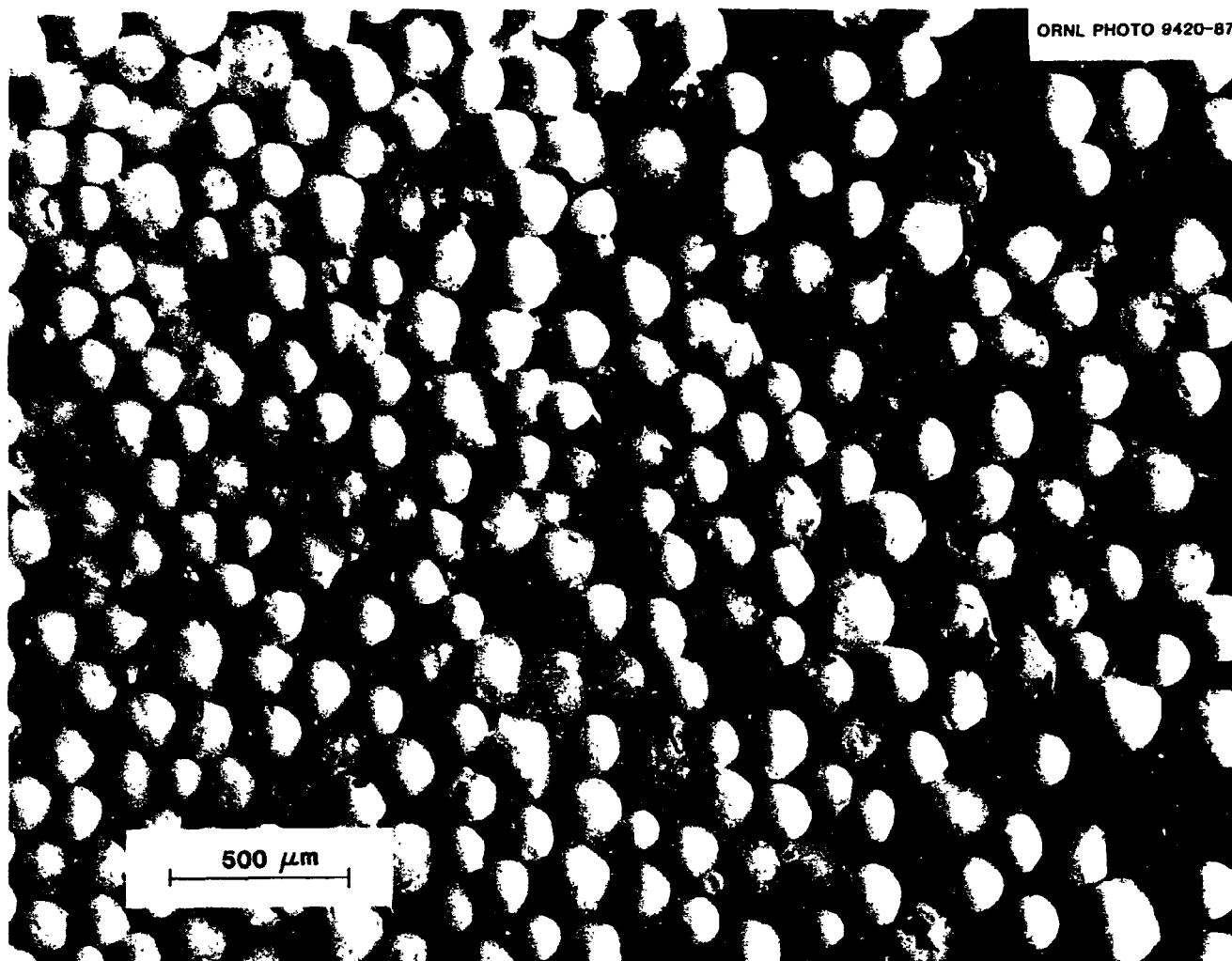


Fig. 7. Photographs of europium oxysulfate product from resin loading experiment No. 6.

There are several ways to ignite the resin beads. The simplest is to burn a reasonably thin layer or bed of resin beads in air in an appropriate container; as noted before, a temperature of 700°C for several hours was used, but a lower temperature may be adequate. Under these conditions, the burning front glows a dull red color as the black beads are converted to smaller spheres of light grey color. Alternatively, the resin can be burned in a column configuration with flowing air, as is done for the HFIR target material, but there is danger of overheating in the burning zone unless proper precautions are taken. In all cases, the off-gas will contain sulfur and other gases which must be handled properly.

##### 5. RECOMMENDED FURTHER WORK

The work was terminated because of funding limitations before it was completed. In general, the approach is promising and no unsolvable problems were found; but several potential problem areas were identified which require additional study. Scouting experiments suggested approaches for resolving these difficulties, but final verification and integration of this step into the TRUEX process remains to be done. Some subject areas needing more attention are listed below.

1. Better definition of the ion-exchange loading and breakthrough curves is needed with feeds (TRUEX americium products) more appropriate to the actual flowsheet that will be used, or covering a range of parameters that would include any reasonable flowsheet variation that may come up. This depends on better TRUEX flowsheet definition. The presence of iron and other impurities is particularly important.

2. The iron interference problem requires more work. Both reduction to ferrous and oxalate complexation are promising, particularly the latter. Additional experimental work, such as changing oxalate to iron ratios, is required to establish the proper conditions to minimize this interference. At the same time, the TRUEX process

must be examined to establish whether or not there is any complication introduced if these means are used to control iron.

3. Depending on additional information regarding iron scrubbing in TRUEX, a wider range of iron concentrations in the americium product may require investigation. On the other hand, it is possible that iron can be tolerated in the americium product so that poorer scrubbing might be acceptable in the TRUEX process, thereby relaxing the requirements in that step. This might be the case if (1) iron can be dealt with adequately in the ion-exchange process and (2) any other impurities do not increase the resin requirement and the final americium waste quantity to an undesired extent. It is possible that some dilution of the solid americium product might be advantageous (or at least not disadvantageous) for preparing the final waste form.

4. A definition of product specifications for the americium recovery process (both the aqueous effluent from the process and the solid americium product) is required so that the process can be properly designed. Since recycle between this step and the TRUEX process is certainly possible, the study must include integration within the overall operation.

5. To provide greater confidence in scaling the test results to plant operations, a few larger-scale experiments with europium tracer should be carried out after operating conditions are better defined. The ion-exchange process has often met with unusual success with respect to scaleup, but one should not push his luck unnecessarily.

6. Finally, to verify all the work, at least one test with americium is recommended for the final flowsheet that evolves from this work. A small matrix of tests to define the final conditions would be most advantageous.

#### 6. ACKNOWLEDGEMENT

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