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THE PROCESSING OF PLUTONIUM BY ION EXCHANGE
II. THE ANION-EXCHANGE SEPARATION OF PLUTONIUM AND THORIUM

Pu Th

by

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A method has been developed for the separation of plutonium and thorium on a plant scale using anion exchange from hydrochloric acid solution. The use of anion exchange from strong nitrate solutions for the processing of plutonium has been quite successful,[1,2,3] largely because the method is highly specific for plutonium. The method fails for plutonium-thorium mixtures, since both elements are strongly sorbed. Plutonium(IV), however, is strongly sorbed by anion exchange resin (Dowex 1X4, 50-100, Cl^-) from strong hydrochloric acid solutions, while thorium(IV) is not.[4,5]

The behavior of a small column was studied in order to optimize the process variables. A flow rate of 25 mg. Pu/min./cm.^{2} was chosen because this is as slow as would allow one run to be made during an eight-hour period, and a single experiment showed that twice this was not practical. Figure 1 shows the effect of the concentration of plutonium and hydrochloric acid on the breakthrough capacity of a 1.07-cm. (I.D.) column. This capacity was calculated from the amount of plutonium fed to the column at breakthrough, less the amount of plutonium in the space around the beads, divided by the volume of the resin bed.

Pretreatment of the feed solution with one equivalent of sodium nitrite per equivalent of plutonium was necessary to remove plutonium(III), which will pass through the column with the thorium and may prohibit the discarding of the raffinate. It was also necessary to make the feed and wash solutions 0.1M in nitric acid to prevent reduction during the process. It was found that three to four column volumes of wash solution (7M HCl - 0.1M HNO_3) were sufficient to give thorium concentrations in the product of < 10 ppm. The

volume of wash required is not a function of the initial concentration of thorium in the feed solution. Since this process will be used as a tail-end treatment for the nitric acid anion exchange system, it was ascertained that concentrations of < 3M nitric acid in the feed solution will not affect the process. The plutonium was stripped from the resin with 0.5M hydrochloric acid.

The process was tested with two pilot-plant columns (6 cm. I.D., 22 cm. of resin bed), with 60 grams of plutonium per run. It was difficult to keep the leading edge of the sorbing plutonium from developing long "spikes" which greatly reduced the apparent pre-breakthrough capacity of the column. With two columns, connected in series, the "spikes" were allowed to break through the first column and were leveled at the top of the second column. The first column was loaded to the degree expected from the earlier experiments, while only a few centimeters at the top of the second column were loaded. This amounted to less than one percent of the initial charge. After passing a sufficient amount of wash solution through the connected columns to free the first column of thorium, the first column was stripped separately. The small amount of plutonium at the top of the second column became the leading edge of the next run when the columns were connected in series in the reverse order. In several runs made with this test apparatus, concentrations of thorium in the feed of 2,000 to 200,000 ppm, based on Pu, always were reduced to < 10 ppm in the product. The coffee-colored band of plutonium(IV) was easily followed throughout the process.

The flowsheet in Fig. 2 had been successfully tested with one plant scale run at the time this summary was prepared.

REFERENCES

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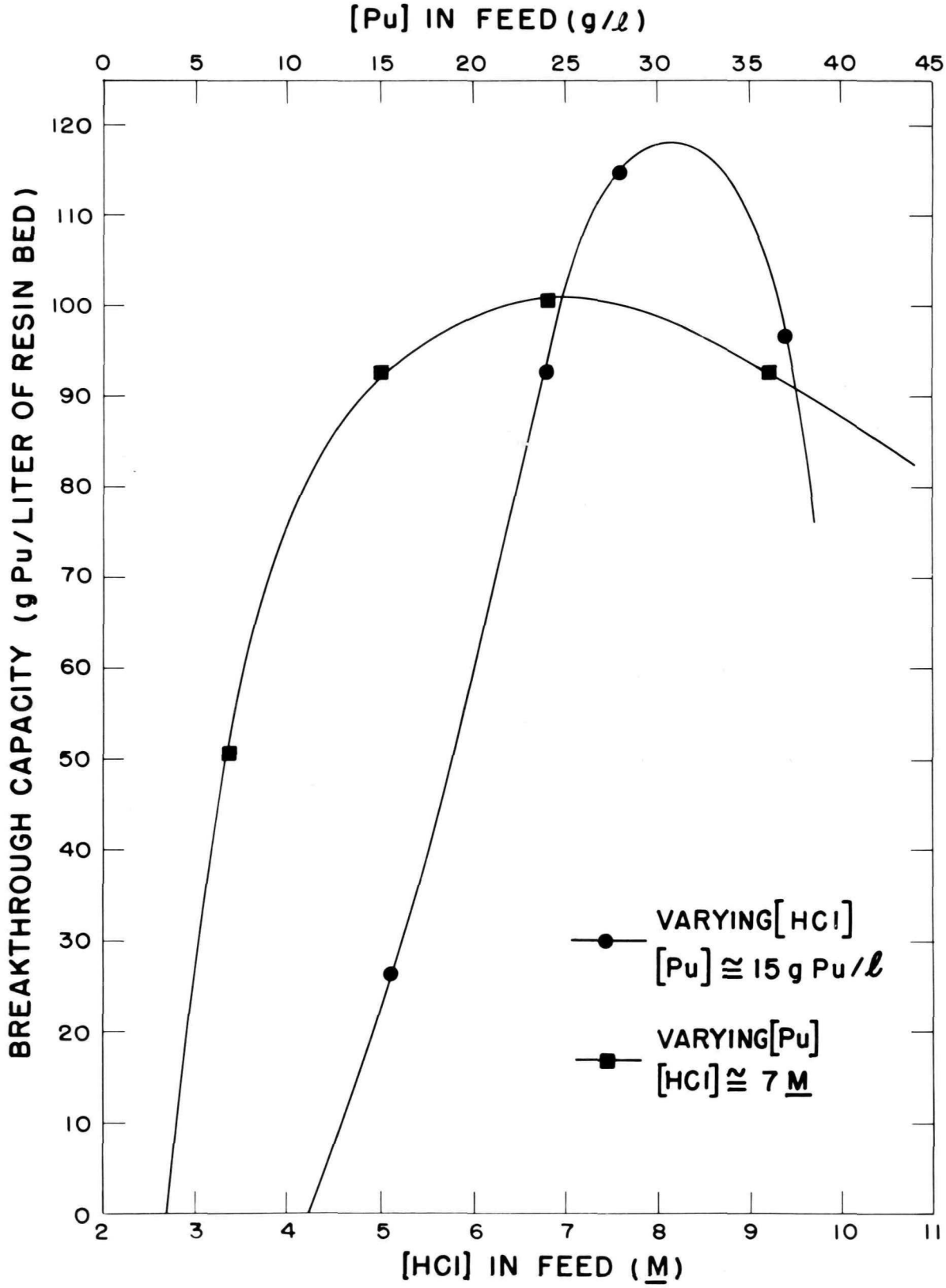
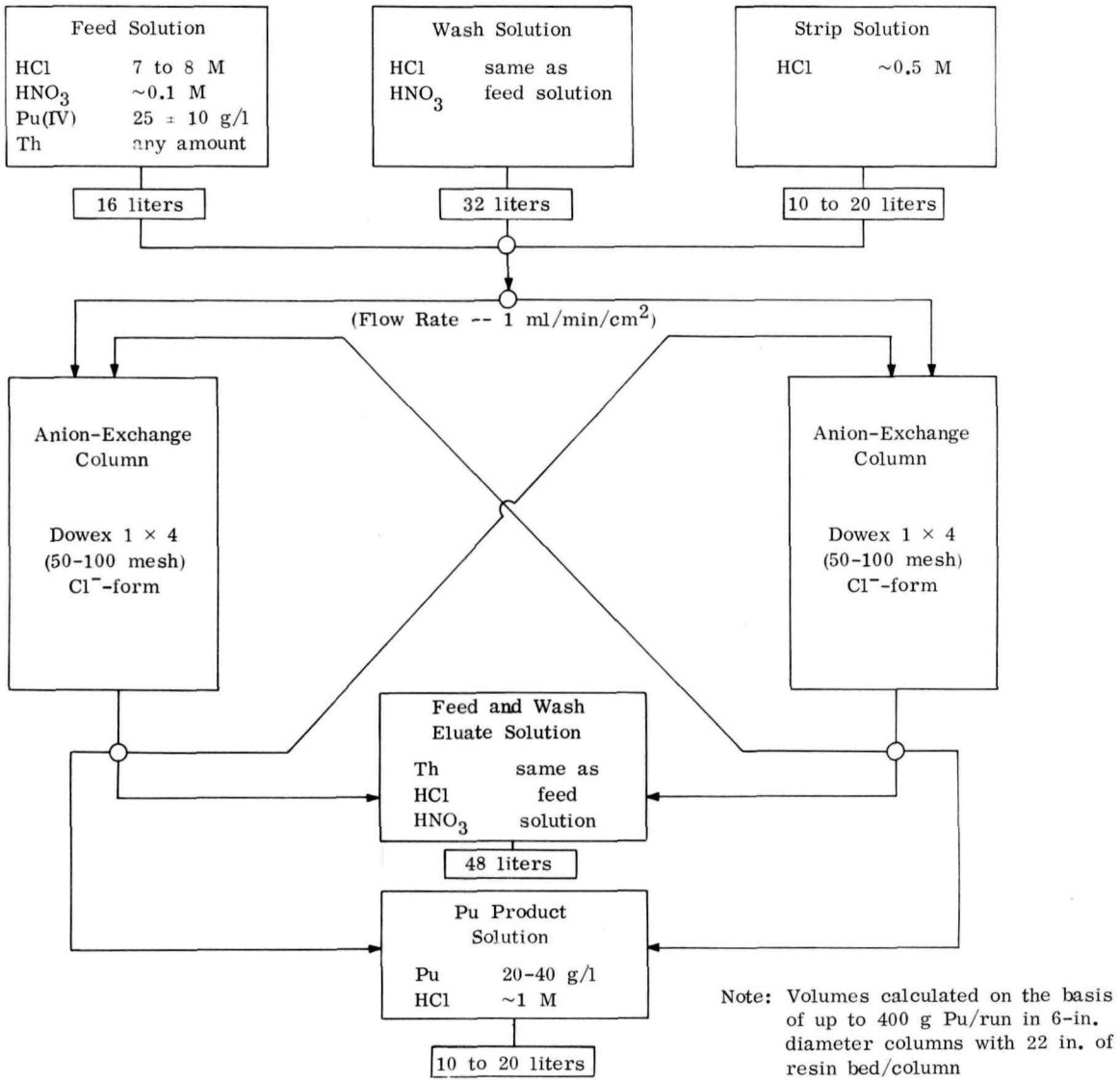


James and Christensen, Figure 1. "Effect of the Concentrations of Pu and HCl in the Feed on the Breakthrough Capacity"



Note: Volumes calculated on the basis of up to 400 g Pu/run in 6-in. diameter columns with 22 in. of resin bed/column

James and Christensen, Figure 2. "Flow Sheet for Plant-Scale, Anion-Exchange Separation of Plutonium and Thorium"