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Washing Behavior of Pu(IV) Charge on a Permutit SK, 40-73 Mesh Column

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F. Franssen  
M. Demonie

Date: September 13, 1961

Translated at ORNL.

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## SUMMARY

This report gives the results of tests on washing at 60°C of a Permutit SK resin column, 40-70 mesh, loaded with Pu(IV). Also given is an empirical formula with which may be calculated the maximum amount of plutonium that must be loaded on a Permutit SK resin column to prevent plutonium loss in the column effluent during washing.

### 1. INTRODUCTION

Report ETR-120 (1) gives the results for plutonium loading on Permutit SK 40-70 mesh resin at 60°C for initial plutonium concentrations of 0.8-5 g/liter and nitric acid concentration of 7.2 *M*.

If the objective of the loading operation is decontamination of the plutonium in a nitric acid solution of its fission products, mainly zirconium and niobium, this decontamination may be improved after the plutonium is loaded on the resin by washing with 7.2 *M* nitric acid (the same molarity as that of the loading solution) at 60°C (2). This possibility is made use of in existing installations.

The different steps in a plutonium purification installation are: (1) plutonium loading on the resin; (2) column washing at 60°C with 7.2 *M* nitric acid (the amount is 20-40 times the volume of the resin bed (2)); (3) elution of plutonium from the resin.

During the washing there is some danger of plutonium loss in the column effluent if the column is too highly loaded with plutonium. During the washing, the plutonium is displaced according to the equilibrium Pu concentration on resin  $\rightleftharpoons$  solution concentration. The plutonium is displaced from the upper part of the column toward the lower part. If the column is highly loaded, plutonium desorbed from the upper part cannot be sorbed by the lower part, so that plutonium is lost in the wash water leaving the column.

In order to prevent this loss, the different parameters that affect plutonium displacement during washing should be studied and the optimum operating conditions determined.

The objective of our study was to determine (a) the parameters that affect plutonium loss in washing, and (b) the optimum plutonium loading on the column to prevent this loss.

## 2. METHOD OF OPERATION

### 2.1 Preparation of Reagents

Nitric acid of 7.2 *M* concentration was prepared by diluting nitric acid (Merck p.a.) with distilled water.

### 2.2 Apparatus

Since the washing is carried out after the plutonium has been loaded on the resin, the apparatus used is the same as that used for the loading tests.

The characteristics of this apparatus are given in Ref. 1. The column was 31 cm high, 0.125 cm<sup>2</sup> in cross section, 3.88 cm<sup>3</sup> in useful volume. Figure 1 is a schematic view of the apparatus.

### 2.3 Resin Washing

When the loading operation is completed (Sect. 3.3 of Ref. 1), receiver A (Fig. 1) for the wash solution (7.2 *M* nitric acid) is put in place and passage of this solution through the resin at the determined washing rate is continued.

### 2.4 Sampling

The same sampling criteria were followed as given in Sect. 3.4 of Ref. 1.

### 2.5 Analyses

The analyses were given in Sect. 3.5.2 of Ref. 1.

### 2.6 Analytical Method

The analytical methods were given in detail in Sect. 3.6 of Ref. 1.

## 3. EXPERIMENTAL RESULTS

A series of tests was made on washing a Permutit SK column, 40-70 mesh, loaded with plutonium. Table 1 gives the conditions.

Table 1

Test No.	Wash Rate, cc/min/cm <sup>3</sup> of resin	Vol of Wash H <sub>2</sub> O per Vol of Resin	Pu Conc in Solution at Loading of g/liter	Pu Conc on Resin, g/liter
1	0.274	30	0.4302	95.2
2	0.253	30	0.02080	91.8
3	0.262	30	0.44	83.5
5	0.131	20	0.6380	80.4
6	1.27	26	0.1266	88.4

As may be seen from Table 1, tests were made under different conditions:

- a. Wash rates between 0.131 and 1.27 cc/min cm<sup>3</sup>
- b. Wash solution volumes between 20 and 30 bed volumes
- c. Plutonium concentrations in column effluent after loading between 0.0208 and 0.6380 g/liter
- d. Plutonium concentration on the resin almost constant, 80.4-95.2 g/liter

Figure 2 gives the results. On the abscissa is the volume of the wash solution passed through the column. On the ordinate is the plutonium concentration in the column effluent.

#### 4. DISCUSSION OF RESULTS

##### 4.1 Introduction

As seen from Fig. 2, all the washing curves, regardless of the original concentration (corresponding to the concentration in the solution at the end of the loading), approach an asymptotic value between 190 and 270 mg/liter. This value may be considered the concentration in the aqueous phase in equilibrium with the resin loaded to a plutonium concentration between 80 and 95 g/liter (Table 1).

##### 4.2 Effect of Washing Rate

The washing rate does not appear to affect the plutonium loss in washing.

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APPARATUS FOR PROCESSING Pu

- ① Compressed Air
  - ② Entrance for Air Bubbles
  - ③ Resin Charging
  - ④ Resin Discharging
  - ⑤ Heating Jacket (Water at 60°C)
  - ⑥ Resin
  - ⑦ Filter
  - ⑧ Needle Valve
  - ⑨ Flow Meter, Greiner 9142B
- 
- Ⓐ Solution Receiver
  - ⓧ 2-Way Valve
  - Ⓣ 3-Way Valve
  - Ⓑ Loading Exit, Low Concentration (Pu 0.8-2 g/liter)
  - Ⓒ Loading Exit, High Concentration and Elution

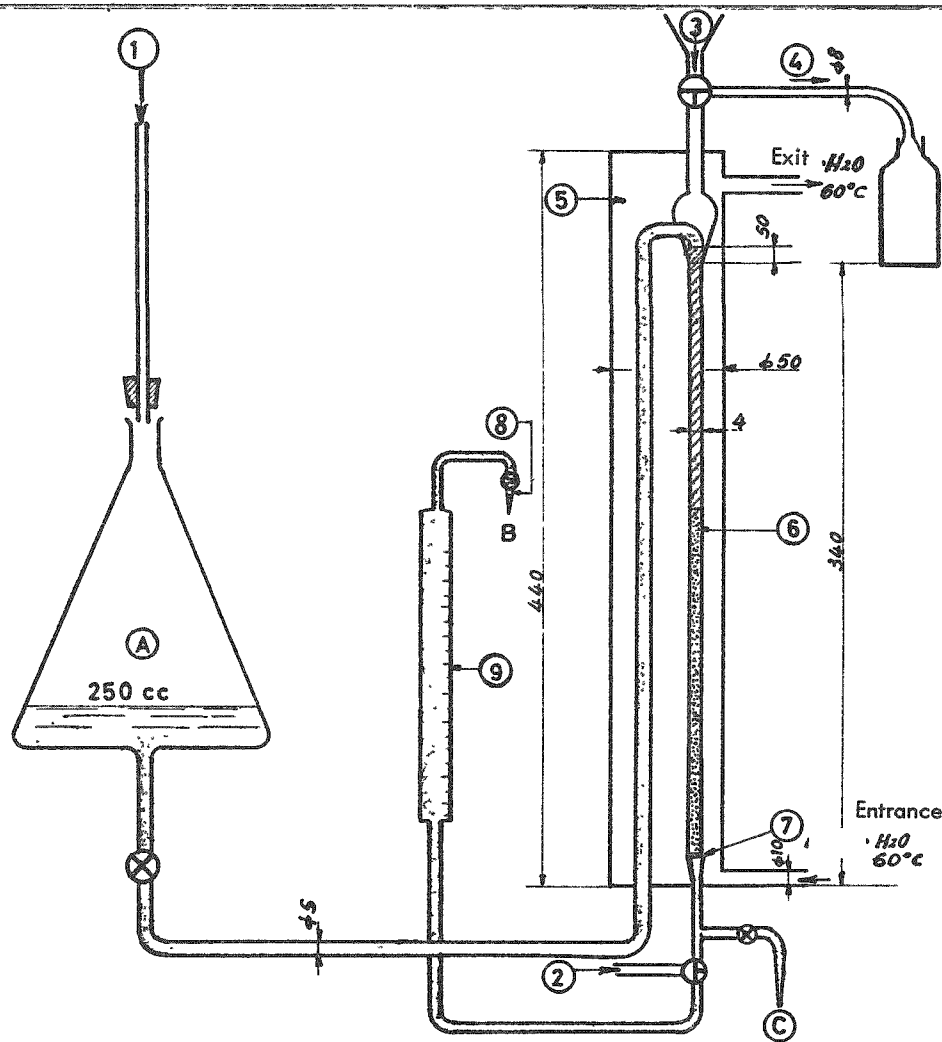
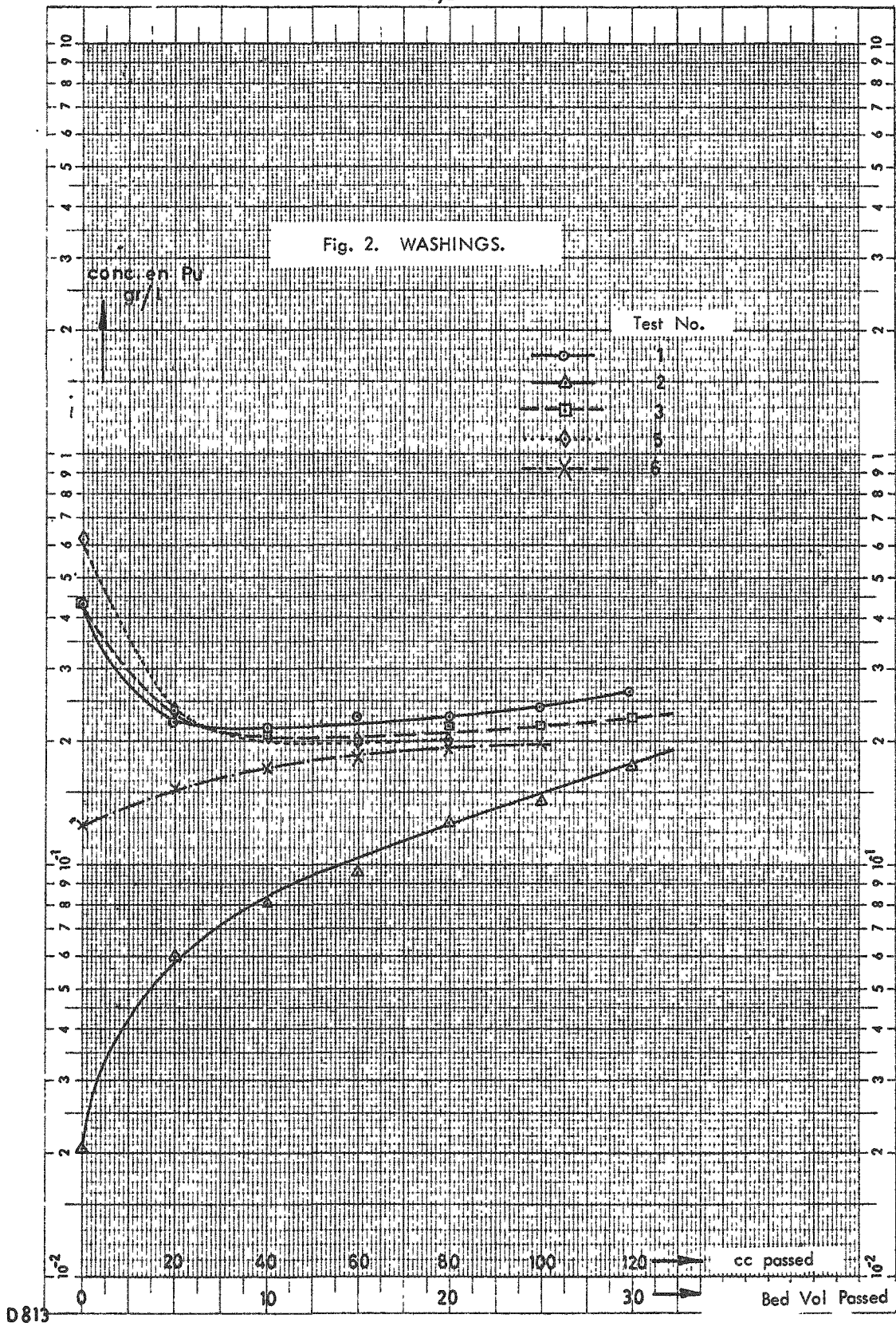


Fig 1





Eine Achse logar. geteilt von 1 bis 1000, Einheit 90 mm, die andere in mm

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