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NP-12244
(ETR-125)

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E U R O C H E M I C

T E C H N I C A L R E P O R T N O . 125

- I. Study of the Kinetics of Pu(IV) Elution from Permutit SK Resin, 40-70 mesh, at 60° by 0.6 M Nitric Acid
- II. Study of Resin Regeneration by 7.2 M Nitric Acid
- III. Calculation for a Plutonium Purification Column of 40-70 mesh Permutit SK

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Date: September 27, 1961

Translated at ORNL.

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SUMMARY

This report gives the results of tests on elution, at 60°C, of a Permutit SK, 40-70 mesh, resin column loaded with Pu(IV).

The optimum elution rate to ensure the highest plutonium concentration in the eluate (or the minimum amount of eluant necessary) in a minimum elution time is also given.

1. INTRODUCTION

Report ETR No.120 (1) gave the results obtained for a plutonium load on 40-70 mesh Permutit SK resin at 60°C, for initial plutonium concentrations of 0.8-5 g/liter and for 7.2 M nitric acid.

Report ETR 123 (2) gave the results obtained for washing tests, at 60°C, of a Permutit SK, 40-70 mesh, resin column loaded with Pu(IV).

The study given below completes the two preceding studies, permitting the establishment of a final chemical purification scheme for plutonium by anionic resins.

2. CHOICE OF CONDITIONS

2.1 Choice of Eluant

A nitric acid molarity of 0.6 was selected as this concentration is considered the best according to the work of Ryan and Wheelwright (3).

2.2 Choice of Operating Temperature

Work on the kinetics of elution by the same authors (3) indicated that a temperature of 60°C is best to ensure a final high concentration of plutonium in the eluate.

3. METHOD OF OPERATION

3.1 Preparation of Reagents

The nitric acid, 0.6 M, was prepared by diluting Merck p.a.* nitric acid with distilled water; the solution thus prepared was standardized by titration with sodium hydroxide of known concentration.

3.2 Apparatus

The elution being made after the loading and washing of the resin, the apparatus used is the same as that used for the loading and washing tests. The characteristics of this apparatus are given in the report cited (1).

The column had the following dimensions: 31 cm height, 0.125 cm² cross-sectional area, 3.88 cm³ volume utilized.

Figure 1 is a schematic view of the apparatus.

*As on original.

3.3 Resin Elution

After completion of the loading and washing, the eluant (0.6 M HNO₃) is placed in vessel A (Fig. 1) and passage of this solution through the resin at a fixed rate is continued.

3.4 Sampling

After passage through the resin, the solution is collected in a capillary at c (Fig. 1).

Samples for determination of the plutonium concentration are taken at regular intervals.

3.5 Analytical Method

The analytical methods are given in detail in Sect. 3.6 of reference 1.

4. EXPERIMENTAL RESULTS

4.1 Aims of the Tests

The tests made had the aim of determining: (a) the relation between the elution rate and the volume of eluant necessary to elute the plutonium from the resin; (b) the relation between the plutonium load on the resin before the elution and the volumes of eluant necessary, the elution rate remaining constant.

4.2 Tests at Different Elution Rates

A series of tests was made at different elution rates; the operating conditions are given in Table 1.

Figure 2 shows the effect of the elution rate on the volumes of eluant necessary (expressed as resin bed volumes).

Preliminary Observations

1. Determination of the Amount of Eluant Needed Expressed as Bed Volumes (Column 3). The volume of eluant necessary was determined experimentally in the following way: From the total volume of solution needed for elution to the point of obtaining a plutonium concentration in the eluate of 1 g/liter was subtracted 2.5 cm³, the volume corresponding to the apparatus between the bottom of the column and the end of the capillary for taking samples. The volume thus obtained was divided by the resin bed volume.

2. Plutonium Loading on Resin before Elution (Column 4). The loading was obtained by dividing the number of milligrams of total plutonium loaded on the resin (see resin loading curve in ref. 1) by the volume of the resin bed.

The values followed by an asterisk (*) were calculated from the total amount of plutonium eluted (column 8) divided by the bed volume.

As may be seen (column 4), the tests were made at a plutonium concentration that varied between 51 and 97.5 g/liter.

This concentration should not affect the volume of the eluant (3).

Table 1

Test n°	Rate, cc/min/cm ³	Eluant Bed Vol	Pu Loading on resin		∫ Elution Curve	Average Elution Conc		Observation Errors, % (5) - (8)
			g/liter (4)	mg total (5)		g/liter (7)	mgr total (8)	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
1	0.0129	2.71	87,5	340	343	31	325	+ 4,4
3	0.0065	2,46	75,5	294	287	27.2	261	+ 11
5	0.0098	2,96	71	271	288	21.2	239	+ 11.8
6	0.0197	2,57	89	339	237	26.3	258	+ 24
7	0.0226	3.02	86.5	336	359	24	281	+ 16.4
8	0.0229	2.75	80	305	302	-	-	-
9	0.0194	2.63	59	229	217	19.5	197	+ 14
12	0.026	2.75	74 *	-	-	27	297	-
14	0.0329	3.45	86 *	-	405	25	334	-
15	0.0390	3.74	86 *	-	310	23	332	-
16	0.0454	3.45	86 *	-	390	25	334	-
17	0.0305	3.51	97.5 *	-	384	27.8	378	-
21	0.120	4.51	51 *	-	255	14.1	191	-

3. Percentage of Plutonium Not Eluted (Column 9). The difference in percent between the total amount of plutonium loaded on the column (column 5) and the total amount of plutonium found in the eluate (column 8) gave us the percentage of the plutonium that remained on the resin after the elution (column 9).

As may be seen, except in test 6 (24%, which may be considered due to experimental error), it may be concluded that about 10% of the plutonium remained on the column after the elution. This conclusion is in contradiction to the results of other authors (3), who considered that under the experimental conditions all the resin can be eluted. On the other hand, if an error of 5% in the determinations is assumed in our case, it seems difficult to always have an error that gives a lower elution yield.

Also, it must be taken into account that in our tests the elution was stopped when the plutonium concentration in the eluate became less than 0.5-0.8 g/liter, to eliminate a long tail on the elution curve.

On the other hand, as shown in the second part, some plutonium was recovered from the column wash at the end of the elution and from the column regeneration

In conclusion, it may be said that if some of the plutonium remains on the column after the elution, it is small enough not to affect the calculations for a resin cycle if a safety factor of 10% is used in calculating the column loading.

4.3 Tests at Constant Elution Rate for Different Plutonium Loadings on the Resin

Table 2 gives the conditions of the tests.

Table 2

Test No.	Rate, cc/min/cm ³	Pu Loading on Resin, g/liter	Vol of Eluant Necessary Bed Vol
4	0.0264	18.3	1.94
19	0.0255	42.4	2.79
20	0.0259	44.6	3.14
12	0.0260	74	2.75

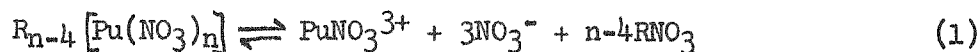
Figure 3 gives the volumes of eluant necessary as a function of the plutonium concentration on the resin after loading.

Preliminary Observations. As may be seen from Fig. 3, the plutonium loading on the resin has no effect for a loading of 40 g/liter or higher. For a lower loading, the necessary eluant volumes are lower.

5. ELUTION MECHANISM

5.1 Introduction

Assuming that the plutonium absorbed on the resin is a simple nitrate complex that does not contain a H⁺ radical, we may write the following relations for the desorption of plutonium with nitric acid (3):



The equilibrium constant for reaction 2, calculated by Hindman (4), is

$$K_{(2)} = (PuNO_3^{3+}) / (Pu^{4+})(NO_3^-) = 2.9 \pm 0.6 \quad (3)$$

On the other hand, if the plutonium loading on the resin is kept constant, the equilibrium constant of relation 1 may be written

$$K'_{(1)} = (PuNO_3^{3+}) \times (NO_3^-)^3 \quad (4)$$

To consider the electroneutrality of the solution, we may write

$$(NO_3^-) = (H^+) + 3(PuNO_3^{3+}) + 4(Pu^{4+}) \quad (5)$$

The overall total of these reactions has certain kinetics; if the elution rate is lower than the kinetics of the equilibriums mentioned above, the concentration at the peak of the elution corresponds to the plutonium concentration at equilibrium in these reactions.

Elution tests under equilibrium conditions at 60°C on Permutit SK of 20-50 mesh under conditions identical to our tests, made by Ryan and Wheelwright (3), gave an elution peak at 59 g of plutonium per liter.

The following system is proposed:

$$(PuNO_3^{3+}) / (Pu^{4+}) \cdot (NO_3^-) = 2.9 \quad (3)$$

$$(NO_3^-) = (H^+) + 3(PuNO_3^{3+}) + 4(Pu^{4+}) \quad (5)$$

or $(H^+) = 0.6$

$$K'_{(1)} = (PuNO_3^{3+}) \cdot (NO_3^-)^3 \quad (4)$$

$$(PuNO_3^{3+}) + (Pu^{4+}) = 59/239 = 0.246 \quad (6)$$

This system of four equations in four unknowns ($PuNO_3^{3+}$; Pu^{4+} ; NO_3^- ; $K'_{(1)}$) gave (3) a value of $K'_{(1)} = 0.54$.

Knowing the value of $K'_{(1)}$, we can calculate from the preceding system of equations the value of an elution peak for a given nitric acid concentration in the eluant solution.

5.2 Determination of Optimum Elution Rate

A series of tests was made with different elution rates. For all elution rates in which the time of contact of the solution with the resin is greater than the time necessary for establishment of the equilibriums mentioned above, the eluant volumes necessary to desorb the plutonium are identical (region I, Fig. 2).

In this case the elution, and consequently the elution time, is not controlled by the kinetics of the establishment of chemical equilibrium of interest in the elution, but by the elution rate.

APPARATUS FOR Pu TREATMENT

1. Compressed air
2. Entrance for raising air bubbles
3. Resin Loading
4. Resin unloading
5. Heating jacket (H_2O at $60^\circ C$)
6. Resin
7. Filter
8. Needle valve
9. Greiner 2142B flowmeter

- A Solution receiver
 X Two-way valve
 T Three-way valve
 B Exit for low-concentration
 (0.0-2 g Pu/liter) loading
 C Exit for high-concentration
 loading and for elution

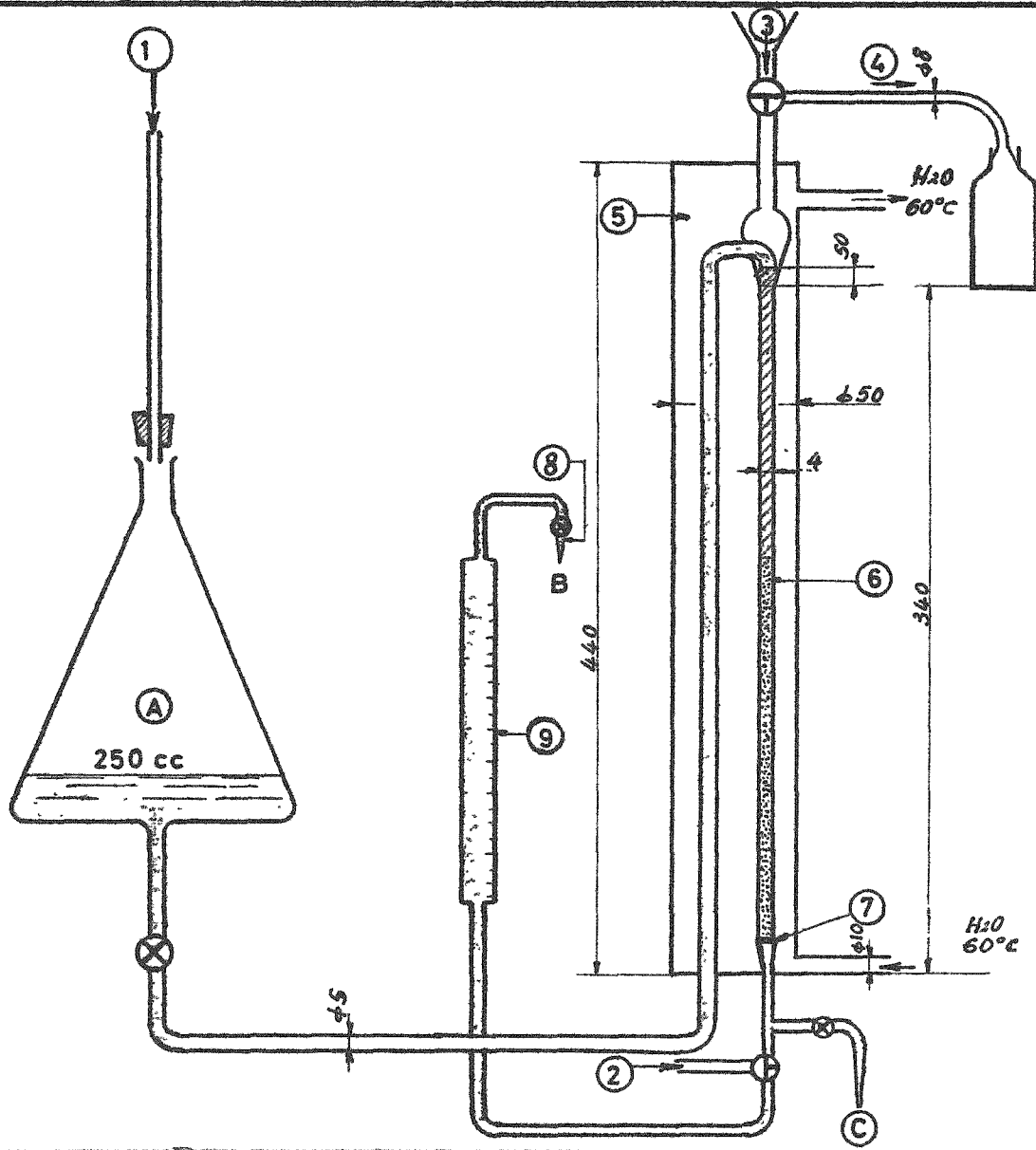
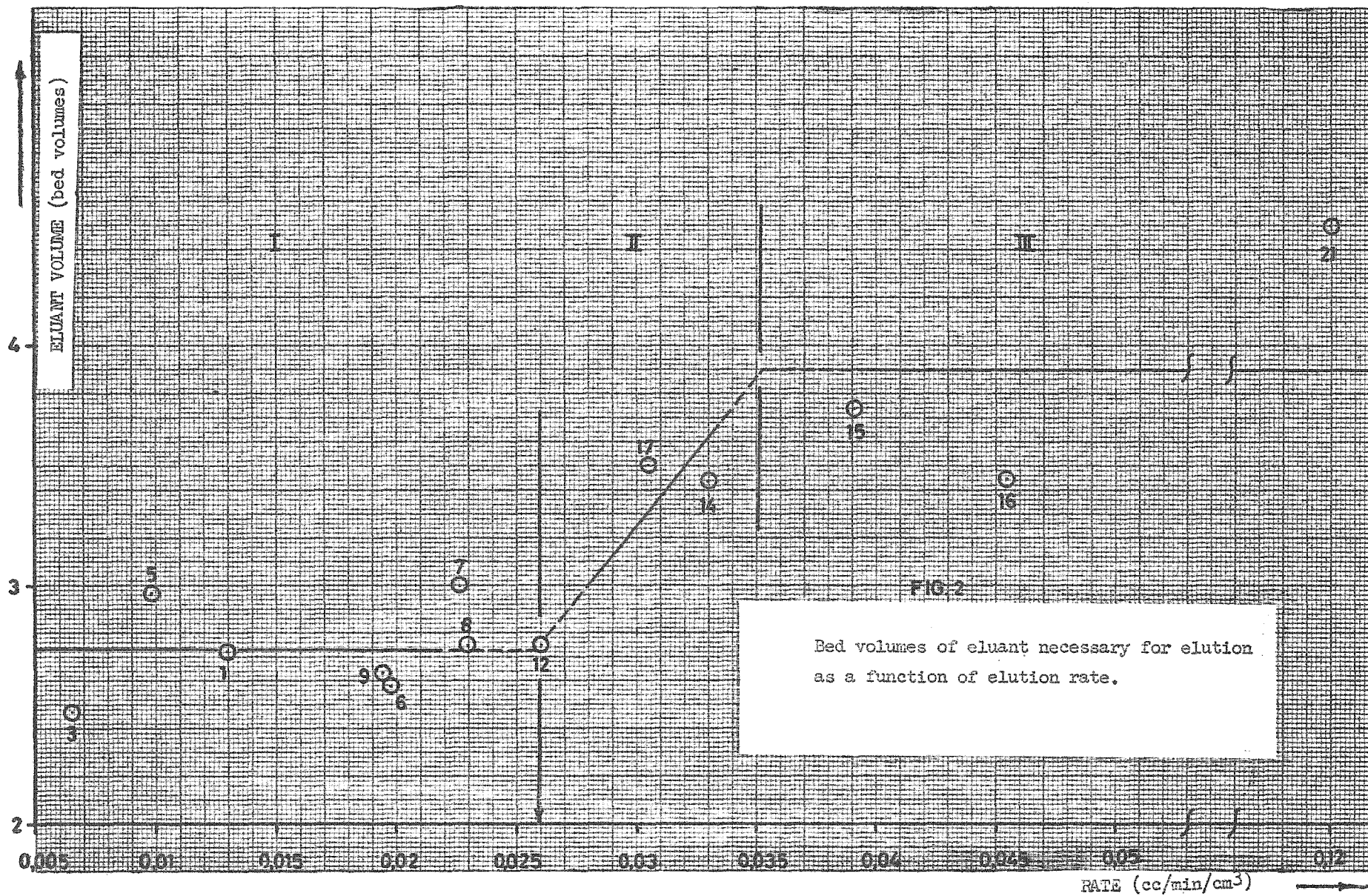
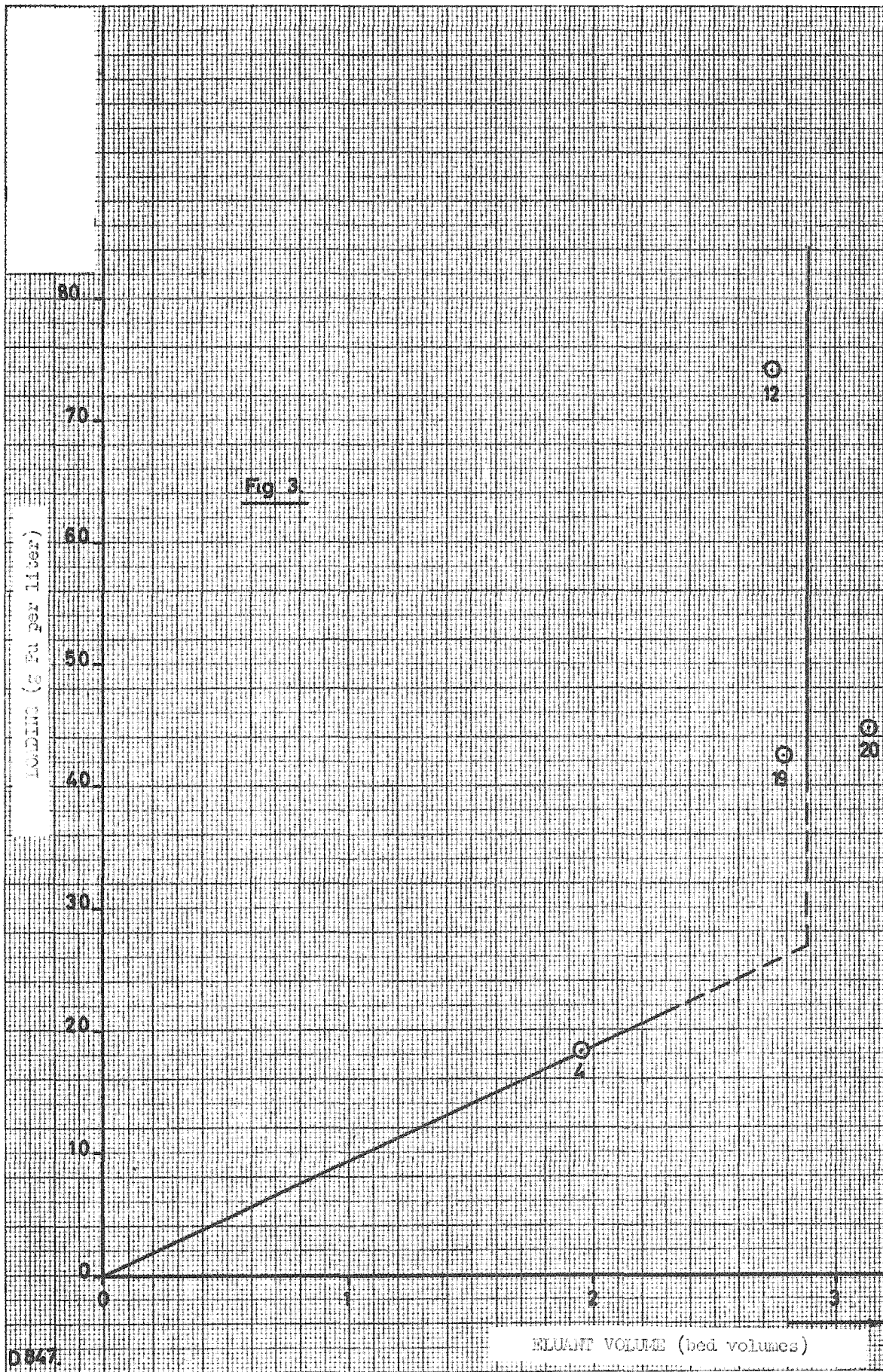


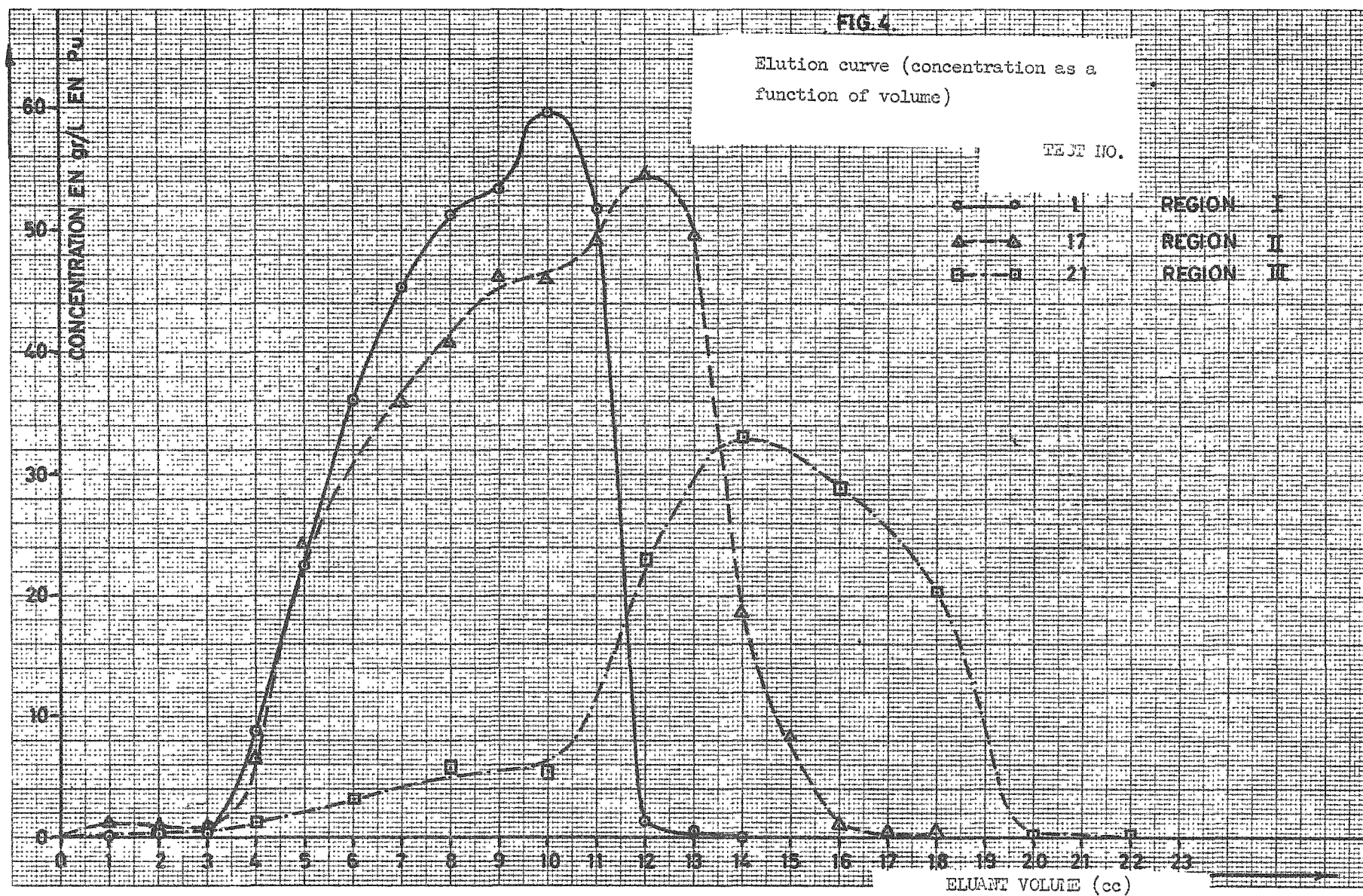
Fig 1





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ELUANT VOLUME (bed volumes)



On the other hand, if the elution rate is such that the time of contact of the solution with the resin is less than the time necessary for establishment of the equilibriums of interest, the elution is controlled by the equilibrium kinetics and not by the elution rate (region II of Fig. 2). In other words, in this region the duration of the elution remains practically constant, while the eluant volumes necessary vary according to the rate.

There is a third region (region III of Fig. 2) in which the elution rate is sufficiently high that it is not possible to connect the concentration to the peak equal to the equilibrium concentration. The more the elution rate is increased, and the lower is the elution peak, the exchange kinetics are higher; the result is that in this region the eluant volume remains practically constant but the elution curve has a lower and more extended peak.

Figure 4 shows a complete elution curve for each rate region considered in Fig. 2.

The optimum elution rate is found at the intersection of regions I and II.

This rate, determined graphically in Fig. 2, is about 0.0260 cc/min/cm³.

5.3 Calculation of Constant K'(1) for Expression 4

As shown in Sect. 5.2, in Region I of Fig. 2 there is a region in which the elution equilibrium is connected; in this way the value of the elution peak corresponds to the equilibrium values of expressions 3-6.

Table 3 gives the values of the peaks measured for the tests made in region I.

Table 3

Test No.	Peak, g Pu/liter	Test No.	Peak, g Pu/liter
1	60	7	54
3	58	8	49
5	49	9	63
6	60	12	55

The average of these values is 56 g/liter. If the value of K'(1) is now calculated by the system of equations 3-6, it is found to be 0.46; Ryan and Wheelwright (Sect. 5.1), under the same conditions of acidity and temperature and with Permutit SK 20-50 mesh, found a value of the elution peak of 59 g/liter, which gives a K'(1) value of 0.54.

Considering the analytical errors in counting Pu ($\pm 5\%$) and of the nitric acid titration ($\pm 2\%$) which affect our tests and not knowing the analytical errors that affect the tests of Ryan and Wheelwright, it may at first glance be concluded that that the K'(1) are practically the same in the two cases.

6. CONCLUSIONS

6.1 Elution Rate

The optimum elution rate is 0.026 cc/min/cm³ of resin.

6.2 Volume of Eluant

The volume of eluant necessary is 2.73 resin bed volumes (Fig. 2).

6.3 Effect of the Plutonium Loading on the Resin

For a concentration of 40 g of plutonium per liter or higher, the resin loading does not have an important effect on the volume of eluant necessary (Fig. 3).

For lower concentrations, the volume of eluant needed decreases (Fig. 3).

7. TESTS STILL TO BE MADE

No tests have been made with very different columns; these tests will be made next. In general, there is an effect of height and of the bed cross section on the volume necessary for the elution (5).

7.1 Effect of Bed Height

From studies made (5) on different kinds of resins and different ions, the general relation between the volume of eluant necessary and the height of the column was demonstrated to be

$$V_1/V_2 = \sqrt{h_2/h_1} \quad (7)$$

where V_1 and V_2 are the volumes of eluant necessary for columns of heights h_1 and h_2 , respectively.

Tests should be made to determine whether a similar relation should be considered in our case. In this way, if the effect of the bed height on the eluant volume is not taken into account, a large error (relation 7) is made in the sense that a larger volume of eluant is used (total required for complete elution).

7.2 Effect of Bed Cross Section

From studies made (5) on other ions it was not possible to determine whether there was an effect of the bed cross section on the eluant volume.

In some cases it appears that there is a relation similar to the above:

$$V_1/V_2 = \sqrt{D_1/D_2} \quad (8)$$

where V_1 and V_2 are the volumes of eluant necessary for columns of diameter D_1 and D_2 , respectively. However, a definitive and general relation could not be set up.

If a column with a larger surface than that in which our tests were made should be designed, then the error is made by default (relation 8).

If relations 7 and 8 are considered, it is seen that a larger column may be constructed, the ratio h/D remaining equal to the ratio in the column we used, without changing the volume of eluant necessary.

II. STUDY OF THE 7.2 M HNO₃ REMAINING IN THE RESIN BED

1. INTRODUCTION

After the plutonium has been eluted from the resin by 0.6 M HNO₃, the bed is restored to a condition for receiving the next load with 7.2 M HNO₃.

Cold preliminary test on restoring the bed treated with 0.6 M HNO₃ with 7.2 M HNO₃, we demonstrated that 5 to 6 bed volumes are necessary to restore the column; these tests were made at a temperature of 25°C.

2. METHOD OF OPERATION

2.1 Preparation of Reagents

The 7.2 M HNO₃ was prepared by diluting Merck p.a.* acid with distilled water; the concentration was determined by titration with NaOH of known strength.

2.2 Apparatus

The operation of restoring the bed being made after the elution, the apparatus used is the same as that used for the elution tests. The characteristics of this apparatus are given in ref. 1 and in Sect. 3.2 of part I of this report.

Figure 1 is a schematic view of the apparatus used.

2.3 Restoration of the Resin

When the elution is finished, the solution for restoring the resin, 7.2 M HNO₃, is placed in receiver A and passage of solution through the resin at a fixed rate is continued.

2.4 Sampling

After passage through the resin, the solution is received in capillary B (Fig. 1).

Samples for analytical determinations (Pu and HNO₃) are taken at regular intervals.

2.5 Analytical Methods

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

3. EXPERIMENTAL RESULTS

3.1 Aims of the Tests

Having determined in the cold the amount of solution needed to restore the resin bed for further sorption (see Sect. 1), the only remaining unknown is the determination of the average concentration of plutonium in the same solution. Several tests were conducted for this purpose. The results are given in Table 1.

*as on original

Table 1

Test No. (1)	Rate, cc/min/cm ³ (2)	Vol of Solution Vol of Bed (3)	Pu Loading on Column before Treatment, mg (4)	Avg Solution Concentration		% Pu Remaining in Apparatus (4) - (6) (7)
				mg/liter (5)	Total mg (6)	
1	0.258	5 + 1	15	67	1.57	90
3	0.258	5 + 1	33	67	1.57	95
5	0.258	5 + 1	32	66	1.5	95
6	0.258	5 + 1	81	63	1.44	98
7	0.258	5 + 1	55	75	1.75	98

Determination of Solution Volume (Column 3)

The volumes reported in this column correspond to 5 bed volumes plus the volume corresponding to the dead volume in the apparatus after the column, which, in our particular case, corresponds to one bed volume.

4. CONCLUSIONS

1. During the restoration of the column, only part of the plutonium that remains in the apparatus (canalizations + resin) is picked up by the restoring medium (5-6%).
2. The plutonium concentration in the solution is about 60-70 mg/liter, which necessitates recycling, eventually, in the nitric acid recovery apparatus.
3. Five bed volumes plus the volume corresponding to the dead volume in the apparatus used (canalizations) are sufficient to restore the resin.

III. CALCULATIONS FOR A PERMUTIT SK 40-70 mesh PLUTONIUM PURIFICATION COLUMN

1. INTRODUCTION

For simplification, loading and wash data already obtained (1,2) are reported here.

2. CALCULATIONS FOR A Pu PURIFICATION CYCLE

2.1 Basis

Loading Solution (1)

Pu concentration	4 g/liter
HNO ₃ concentration	7.2 M
Amount of Pu to be treated per cycle	1000 g
Max tolerable loss	3 mg/liter
Loading rate	0.35 mg/min/cm ³ of resin
Operating temperature	60°C

Wash Solution (2)

HNO ₃ concentration	7.2 M
Volume of wash solution	27 resin bed volumes
Washing rate	0.5 cc/min/cm ³
Operating temperature	60°C

Elution Solution (part I)

HNO ₃ concentration	0.6 M
Elution solution volume	2.73 resin bed volumes
Elution rate	0.026 cc/min/cm ³
Operating temperature	60°C

Resin Regeneration (part II)

HNO ₃ concentration	7.2 M
Solution volume	5 resin bed volumes
Regenerating solution rate	0.258 cc/min/cm ³
Operating temperature	25°C

Note: In the calculations below, no account is taken of the dead volume of the installation; this volume cannot be determined until after the project is final.

2.2 Column Calculations

The dimensions of the column were calculated in ref 2, Sect. 6.2. These are:

Diameter	11.7 cm
Cross section	107.5 cm ²
Height	168 cm
Volume	18 liters

2.3 Calculations for Different Chemical Schemes

2.3.1 Loading

For the detailed calculations see refs 1 and 2.

Rate	1.57 liters/min
Pu rate	58.4 mg/min/cm ²
Duration of operation	160 min
Total solution volume	250 liters
Total Pu treated	1000 g
Pu load on resin	55.5 g/liter
Pu loss from column	1.7 mg/liter or 0.043%

2.3.2 Washing

For calculations detailed see ref 2 .

Rate	9 liters/min
Total solution volume	480 liters
Duration operation	54 min
Pu concentration in wash solution	< 3 mg/liter
Max. Pu loss	0.14%

2.3.3 Elution

Volume of Eluant Necessary. As said in part I of this report, there are no data on the volume of eluant needed for eluting a large-dimensioned column. In the calculation it is assumed that the volumes of eluant needed are the same as those found for the conditions of our tests.

The column volume is 18 liters, and the eluant volume needed is 2.73 resin bed volumes; thus we obtain:

$$\text{Vol} = 2.73 \times 18 = 50 \text{ liters}$$

Eluant flow: The optimum elution rate is $0.026 \text{ cc/min/cm}^3$, which gives

$$D = 0.026 \times 18000 = 470 \text{ cm}^3/\text{min}$$

or 0.470 liters/min

Average concentration in eluate: If an elution efficiency of 90% is assumed and the loss to loading and washing is neglected, an average eluate Pu concentration of 18 g/liter is obtained.

This calculated concentration is lower than that found for a similar column by Ryan and Wheelwright (50 g/liter) (3). This probably is due chiefly to two factors: effect of the column height which was neglected in our calculations although it can have a large effect (formula 7 of part I).

For the chemical scheme proposed by Ryan and Wheelwright (3), during loading the loss from the column is much higher (300-400 mg/liter), which permits obtaining a higher loading on the column (75 g/liter), affecting the final plutonium concentration in the eluate.

In the chemical scheme proposed by Ryan and Wheelwright, the problem of recovering the plutonium from the loading and wash solutions arises. In our chemical scheme, the product has a rather low concentration, but there is no problem of recovering the plutonium from the loading and wash solutions.

Duration of the operation: $50/0.470 = 106 \text{ min.}$

2.3.4 Resin Regeneration

Volume of solution needed: 5 resin bed volumes are needed to ensure resin regeneration; we have thus:

$$18 \times 5 = 90 \text{ liters of regenerating solution}$$

Rate of regeneration: $0.258 \text{ cc/min/cm}^3$ or $0.258 \times 18,000 = 4650 \text{ cc/min}$ or 4.65 liters/min.

Plutonium concentration in the solution: As seen in part II of this report, the plutonium concentration is 60-70 mg/liter.

This may be recovered by sending the solution to the acid recovery system.

Duration of the operation: $90/4.65 = 20$ min.

2.4 General Summary

Conditions	Loading	Wash	Elution	Regeneration
Rate, liters/min	1.57	9	0.470	4.65
Volume per cycle, liters	250	480	50	90
Duration of operation, min	160	54	106	20

Notes

1. Total cycle duration: 340 min or 5 hr 40 min: If the various dead times are considered, this becomes 6-7 hr.

2. Proposed rates: All the rates proposed are maximum; if for the particular circumstances of the plant it is necessary to use lower rates, this does not affect the preceding conclusions. Higher rates are not advisable because they may substantially modify the operating conditions for the resin.

3. Amount of plutonium to be treated per day: The cycle of the chemical scheme proposed lasts 6-7 hr. Taking into account the inevitable dead time in such an installation, two cycles per day may be made, which corresponds to treatment of 2000 g of plutonium.

4. The proposed installation can treat daily any amount of plutonium lower than the assumed (2000 g); the two following conditions should be realized: the plutonium concentration of the loaded solution should be between 0.55 and 5 g/liter (1); the duration of the loading should be such that the amount of plutonium on the resin at the end of the loading is 1000 g.

Example: If the production of plutonium is 100 g/day, the duration of the loading operation would be

$$t = 1000/100 = 10 \text{ days}$$

The duration of the other operations (washing, elution, ...) would be unchanged.

5. Direction of passage of various solutions: The loading and washing should be made in the same direction (2), preferably from top to bottom to prevent displacing resin particles from the bed. The elution and regeneration may be made either way.

In order to rehomogenize the bed after each cycle, it would be preferable to make the elution or the regeneration in the direction opposite to that of loading and washing. The direction to be adopted, in our opinion, are:

Loading: top to bottom
Washing: top to bottom

Elution: bottom to top
Regeneration: top to bottom

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