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THE CHEMISTRY OF
THE ANION EXCHANGE PROCESS FOR THE RECOVERY OF PLUTONIUM

CRDC - 818

by

R.G. HART, J.A. BROTHERS, I.W. ALLAM

Chalk River, Ontario

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ABSTRACT

A semi theoretical study has been made of the chemistry of the Anion Exchange Process for the recovery of plutonium. Pseudo-equilibrium constants have been obtained for the systems plutonium-III - Dowex 50, plutonium-III - Dowex 50-W, and plutonium-IV - Dowex 1, in nitric acid solution. Exchange height equations have been obtained for the plutonium-III - Dowex 50 and the plutonium-IV - Dowex 1 systems. Elution equations are given for the systems plutonium-III - Dowex 50 and plutonium-III - Dowex 50-W. The equilibrium plutonium-IV - Dowex 1 in the presence of uranium is also briefly discussed.

INTRODUCTION

The existence of anionic nitrate complexes of plutonium-IV in concentrated nitrate solutions has led to the development of a separations process involving the absorption of these complexes on strongly basic anion exchange resins (15). The process has been applied to the separation of plutonium from both irradiated uranium and plutonium-aluminum fuels (1). It could also be applied to the separation of plutonium from any species that is cationic or non ionic in strong nitrate solution. Because the process is applicable to a wide variety of separations a study has been made of its chemistry so that operating conditions can be predicted for any given separation.

THE PROCESS FLOW

A simplified flowsheet of the separations process is given in Figure 1. The nitrate solution of plutonium-IV is applied to a strongly basic anion exchange resin which has previously been converted to the nitrate form. The plutonium is absorbed on the resin while the cationic and weakly held anionic impurities pass through the column into the feed effluent. After the column has been washed with strong nitric acid the plutonium is eluted by converting it to cationic plutonium-III with a reducing agent such as hydroxylamine nitrate. Because of the instability of hydroxylamine in strong nitric acid a 1 N nitric acid spacer wash is inserted between the wash and reducing solution. The hydroxylamine can be decomposed by controlled addition to boiling 7.5 M nitric acid giving a plutonium-IV product in nitric acid solution.

If a single pass does not give a product of the desired purity (1) the plutonium-IV product in 7.5 M nitric acid can be applied directly to a second anion column as shown in route 1, Figure 1. Alternatively the plutonium-III may be absorbed from the hydroxylamine solution onto a strongly acidic cation exchange resin as shown in route 2. The hydroxylamine is washed from the column with 0.5 M nitric acid and the plutonium is eluted from the column with 7.5 M nitric acid. The eluate can then be applied directly to a second anion exchange column for further purification. In practice route 2 has proven to be preferable to route 1 because of corrosion product catalysed oxidation of plutonium-IV to plutonium-VI in the hydroxylamine decomposition step (2)(3).

EQUILIBRIUM STUDIES

Anion Exchange Equilibria

In a study of the nitrate complexes of tetravalent plutonium Brothers et al (4) have shown that $R_2Pu(NO_3)_6$* is the species formed when plutonium-IV absorbs on an anion exchange resin. They also show that below 6.5 M nitrate ion plutonium-IV is present almost entirely in the form $Pu(NO_3)_4$ and that above 11 M nitric acid plutonium-IV is present almost entirely in the form $H_2Pu(NO_3)_6$. Thus the reactions of plutonium-IV with an anion exchange resin can most conveniently be expressed as

$$Pu(NO_3)_4 + 2\ RNO_3 \rightleftharpoons R_2Pu(NO_3)_4 \qquad [1]$$

in low acid concentrations, i.e. < 7 M, and

$$H_2Pu(NO_3)_6 + 2\ RNO_3 \rightleftharpoons R_2Pu(NO_3)_6 + 2\ HNO_3 \qquad [2]$$

at high acid concentrations, i.e. >11 M. At intermediate acid concentrations both these reactions and possibly the reaction

$$HPu(NO_3)_5 + 2\ RNO_3 \rightleftharpoons R_2Pu(NO_3)_6 + HNO_3 \qquad [3]$$

could be involved.

If it is assumed that reaction [1] is the predominant reaction at most acid concentrations of interest it is possible to write the following equilibrium expression to represent the system.

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* Here and in what follows R represents the resin matrix.

$$K_1 = \frac{C_{R_2Pu(NO_3)_6}}{C_{Pu(NO_3)_4} \cdot \left(V - C_{R_2Pu(NO_3)_6}\right)^2} \cdot \frac{\gamma_{R_2Pu(NO_3)_6}}{\gamma_{Pu(NO_3)_4} \cdot \gamma^2_{RNO_3}} \qquad [4]$$

In equation [4] C's represent appropriate concentrations in moles/litre for the solution phase and milliequivalents/gram of dry resin in the resin phase, γ's represent the appropriate activity coefficients, V is the saturation capacity of the resin in milliequivalents per gram of dry resin, and K_1 is an equilibrium constant. V can be readily obtained by acid titration of a known weight of exhausted resin and for most anion exchange resins is around 3.0 (5). $\gamma_{Pu(NO_3)_4}$ can be obtained from tributyl phosphate equilibrium data (4) and is plotted as a function of ionic strength in Figure 2 with the value at an ionic strength of 1 arbitrarily set at 1. In nitric acid solutions the ionic strength can be obtained from the dissociation data of Krawetz (6), Figure 3. The activity coefficient ratio $\gamma_{R_2Pu(NO_3)_6}/\gamma^2_{RNO_3}$ has not been determined but should be a function solely of the resin plutonium concentration.

The equilibrium between 50-100 mesh Dowex-1 - 1% cross-linked and various nitric acid solutions of plutonium-IV has been studied by Durham and Mills (7). The low cross-linked, fine mesh resin was used to hasten attainment of equilibrium. Since the titration capacity of 1% cross-linked 50-100 mesh Dowex-1 is very similar to the titration capacity of most other quaternary ammonium polystyrene base resins (5) it is reasonable to assume that the equilibrium data obtained will be applicable to any strongly basic anion exchange resin of this type. The rate of attainment of equilibrium will however vary drastically with particle size and degree of cross linkage. The results of these equilibrium experiments are given in Table I. These data have been substituted in equation [4] using V = 3.5 (8) to give the values of $K_1 \dfrac{\gamma^2_{RNO_3}}{\gamma_{R_2Pu(NO_3)_6}}$, hereafter referred to as $K_1{}^1$, given in Table I and plotted in Figure 4.

TABLE I

PLUTONIUM-IV - DOWEX 1 EQUILIBRIUM DATA

HNO_3 Conc. moles/l.	Ionic Strength	Sol'n.Pu Conc. $C_{Pu(NO_3)_4}$ moles/l.	Resin Pu Conc. $C_{R_2Pu(NO_3)_6}$ meq./gm. dry resin	Resin NO_3 Conc. $V-C_{R_2Pu(NO_3)_6}$ meq./gm. dry resin	Pseudo Equilibrium Constant K_1^1
10	5.05	0.51×10^{-3}	0.321	3.18	0.65
		0.53 "	0.430	3.07	0.91
		0.61 "	0.455	3.04	0.85
		0.74 "	0.597	2.90	1.00
		0.80 "	0.637	2.86	1.02
		0.82 "	0.766	2.73	1.33
		1.31 "	1.76	1.74	4.7
9	5.05	0.29 "	0.472	3.03	1.8
		0.31 "	0.572	2.93	2.3
		0.41 "	0.760	2.74	2.6
		0.44 "	0.766	2.73	2.5
		0.49 "	1.32	2.18	6.0
		0.53 "	1.11	2.39	3.9
		0.58 "	1.46	2.04	6.4
		0.66 "	2.03	1.47	13.8
		2.53 "	3.20	0.30	148.0
8	5.00	0.15 "	0.446	3.05	3.3
		0.17 "	0.680	2.82	5.1
		0.19 "	0.702	2.80	4.9
		0.22 "	0.770	2.73	4.8
		0.24 "	1.18	2.32	9.3
		0.27 "	1.52	1.98	14.6
		0.54 "	2.72	0.78	84.
7	4.85	0.15 "	0.436	3.06	3.8
		0.18 "	0.653	2.85	5.4
		0.21 "	1.03	2.47	9.6
		0.44 "	2.34	1.16	50.
		0.48 "	2.55	0.95	71.
		1.20 "	2.85	0.65	67.
		2.11 "	3.21	0.29	216.
5	3.95	0.317×10^3	0.465	3.04	3.5
		0.364 "	0.731	2.77	6.0
		0.400 "	0.958	2.54	8.6
		0.433 "	1.32	2.18	14.2
		0.895 "	2.46	1.04	56.
		1.08 "	2.55	0.95	61.
		1.14 "	2.65	0.85	77.
3	2.70	0.940 "	0.486	3.01	3.8
		1.06 "	0.540	2.96	4.3
		1.24 "	0.712	2.79	5.5
		1.31 "	0.820	2.68	6.3

An examination of Figure 4 shows that the values of $K_1{}^1$ fall on three parallel lines, one comprising all the values from equilibrations at acid concentrations from 3 to 8 M, one comprising the values for equilibrations at 9 M and one comprising the values at 10 M. The increase in $K_1{}^1$ with resin loading is due to an increase in the value of $\frac{\gamma^2_{RNO_3}}{\gamma_{R_2Pu(NO_3)_6}}$ and this is independent of the aqueous phase as it should be. The fact that $K_1{}^1$ is constant at constant resin loading for all nitric acid concentrations from 3 to 8 M indicates that reaction [1] holds up to 8 M and that equation [4] described the system remarkably well up to this point. The reduction in $K_1{}^1$ above 8 M is due to the increasing contribution of reactions [2] and [3] .

It would seem of value to measure $K_1{}^1$ values for typical salt solutions to check them with the nitric acid values but this has not been done to date.

In the presence of uranium the system is complicated by the absorption of uranium species in the resin phase. Experiments similar to those used in the identification of the plutonium species absorbing (4) have shown that both $R\ UO_2(NO_3)_3$ and $R_2UO_2(NO_3)_4$ are formed when an anion exchange resin is contacted with a nitrate solution of uranium-VI (Figure 5). Consequently the reactions

$$UO_2(NO_3)_2 + RNO_3 \rightleftharpoons R\ UO_2(NO_3)_3 \qquad [5]$$

and $$UO_2(NO_3)_2 + 2\ RNO_3 \rightleftharpoons R_2UO_2(NO_3)_4 \qquad [6]$$

compete with reactions [1], [2], and [3] for resin sites thus greatly reducing the capacity of the resin for plutonium (1). Attempts to resolve this system quantitatively have been unsuccessful.

Cation Exchange Equilibria

In general plutonium-III does not have a strong tendency to form complex ions (9) and, although some evidence does exist for weak complex formation in nitrate solution (9), it has been assumed for the purposes of this study that the absorption of plutonium-III by cation exchange resins can be adequately represented by the reaction

$$Pu^{+3} + 3\ HR \rightleftharpoons Pu\ R_3 + 3\ H^+ \qquad [7]$$

Writing an equilibrium expression for reaction [7] similar to that for reaction [1] gives

$$K_2 = \frac{C_{PuR_3} \cdot C^3_{H^+}}{C_{Pu^{+3}} \cdot \left(V - C_{PuR_3}\right)^3} \qquad \frac{\gamma_{PuR_3}}{\gamma_{Pu^{+3}}\ \gamma^3_{HR}} \qquad [8]$$

where C's again represent concentrations in moles/litre for the solution phase and milliequivalents/gram dry resin for the resin phase, γ's again are activity coefficients, V is the saturation capacity of the resin in milliequivalents/gram of dry resin and K_2 is an equilibrium constant. V can be readily determined by caustic titration of a known weight of exhausted resin and for most strongly acid cation exchangers is around 5.0 (5). C_{H^+} can be obtained from the dissociation data of Krawetz (6) Figure 3. The activity coefficient ratio

$\frac{\gamma_{PuR_3}}{\gamma_{Pu^{+3}}\ \gamma^3_{HR}}$ has not been determined.

Durham and Aikin (10) have studied the equilibrium between 8% cross-linked, 50-100 mesh Dowex 50 and plutonium-III - 0.1 M hydroxylamine nitrate solutions at various concentrations of nitric acid. Their data have been substituted into equation [8] using their value of V = 5.5, to obtain the values of $K_2\ \frac{\gamma_{Pu^{+3}}\ \gamma^3_{HR}}{\gamma_{PuR_3}}$, hereafter referred to as $K_2{}^1$, given in Table II. A similar set of data is given for 8% cross-linked, 50-100 mesh Dowex 50-W, using V = 5.15, in Table III.

Fortuitously the ratio $\frac{\gamma_{Pu^{+3}}\ \gamma^3_{HR}}{\gamma_{PuR}}$ appears to be reasonably constant over the range of concentrations studied in the Dowex 50 equilibrations giving an average value of $K_2{}^1 = 24$. The ratio $\frac{\gamma_{Pu^{+3}}\ \gamma^3_{HR}}{\gamma_{PuR}}$ is not constant for the Dowex 50-W equilibrations giving values of $K_2{}^1$ varying from 21 at 0.48 M HNO_3 to 7 at 2.39 M HNO_3.

TABLE II

PLUTONIUM-III - DOWEX 50 EQUILIBRIUM DATA

HNO_3 Conc. moles/l.	Sol'n. H^+ Conc. C_{H^+} moles/l.	Soln. Pu Conc. $C_{Pu^{+3}}$ moles/l.	Resin Pu Conc. C_{PuR_3} meq./gm. dry resin	Resin H^+ Conc. $V-C_{PuR_3}$ meq./gm. dry resin	Pseudo Equilibrium Constant K_2^1
0.54	0.54	0.658×10^{-3}	2.70	2.80	29
0.54	0.54	0.221 "	1.88	3.62	28
0.54	0.54	0.269 "	2.00	3.50	28
1.31	1.24	0.614 "	0.765	4.73	23
1.32	1.24	0.193 "	0.310	5.19	23
1.30	1.24	0.229 "	0.350	5.15	21
2.81	2.60	0.439 "	0.081	5.42	20
2.78	2.60	0.469 "	0.092	5.41	22
5.89	4.30	0.485 "	0.023	5.48	23
5.89	4.30	0.469 "	0.022	5.48	23

TABLE III

PLUTONIUM-III - DOWEX 50-W EQUILIBRIUM DATA

HNO_3 Conc. moles/l.	Soln. H^+ Conc. C_{H^+} moles/l.	Soln. Pu Conc. $C_{Pu^{+3}}$ moles/l.	Resin Pu Conc. C_{PuR_3} meq./gm. dry resin	Resin H^+ Conc. $V-C_{PuR_3}$ meq./dry resin	Pseudo Equilibrium Constant K_2^1
0.48	0.48	1.49×10^{-3}	2.96	2.54	21
0.48	0.48	1.28 "	2.88	2.62	21
1.17	1.14	1.58 "	0.89	4.61	11
1.18	1.14	1.90 "	0.99	4.51	11
2.39	2.20	0.29 "	0.024	5.48	6.6

Kinetic Studies

The equilibrium studies, which provide a method of calculating the equilibrium resin plutonium concentration, must be supplemented by kinetic studies, which provide a method of applying the equilibrium data to column operation. Several methods of treating the dynamic ion exchange system have been described in the literature (11) (12) but most involve, for univalent-polyvalent exchange, making a linear approximation for the equilibrium expression. Treatment of a system in this manner is desirable, if possible, since the numbers obtained are applicable to columns of any practical size, operating at any practical flowrate and loaded with resin of any practical particle diameter. In the plutonium systems considered no satisfactory approximation for the equilibrium expression could be found. Consequently, the data were treated by the exchange zone method of A.S. Michaels (13). Michaels relates the column capacity to any given breakthrough to the equilibrium column capacity by the relation

$$C_E = C_T \left\{ 1 - \frac{(1-F)h_z}{h_t} \right\} \qquad [9]$$

where C_E is the column capacity to any given breakthrough*

C_T is the equilibrium column capacity*

h_t is the total column height in cms.

F is the fractional residual capacity of the exchange zone

h_z is the height of the exchange zone in cms.

$$F = \frac{\int_{V_E}^{V_T} (X_O - X)\, dV}{X_O \ V_Z} \qquad [10]$$

where X_O is the influent metallic ion concentration meq./cc.

X is the effluent metallic ion concentration meq./cc.

V_E is the volume of effluent collected to breakthrough

V_T is the volume of effluent collected to exhaustion

V_Z is $V_T - V_E$.

Michaels has shown that, for a symmetrical breakthrough curve, a reasonable assumption for most systems, $F = 0.5$ and

$$h_z = h_t \left(\frac{V_Z}{V_T - 0.5\ V_Z} \right) \qquad [11]$$

This method eliminates the necessity of making a linear approximation for the equilibrium expression but requires either graphical or empirical correlation of exchange zone height with flowrate and particle size. Consequently, exchange heights have been determined experimentally and have been plotted against flow rate and particle size. Figure 6 shows the exchange heights for

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* C_E and C_T can be expressed in any consistent units.

plutonium-III on Dowex 50. The points for 60 mesh Dowex 50 can be closely approximated by the equation $h_z = 70\ d\sqrt{v}$ where v is the flow rate in mls./min./cm.^{2} and d is the diameter of the resin particle in cm. The $\sqrt{v}$ relationship has also been found by Michaels (13) and Marr and David (12). The two points obtained on 20-24 mesh Dowex 50 give reasonable agreement with the above equation but this should be further checked on a pilot plant scale. Preliminary results on the absorption of plutonium-IV on Dowex 1-8X and Dowex 1-1X give exchange heights represented by the empirical equations $h_z = 300\ d\sqrt{v}$ and $h_z = 170\ d\sqrt{v}$ respectively. It should be emphasized that these values are based on a minimum number of experimental results and should only be used as a guide to future experimental work.

ELUTION CONSIDERATIONS

The reaction governing the elution of plutonium from Dowex 1 can be expressed as

$$R_2Pu(NO_3)_6 \rightleftharpoons Pu(NO_3)_4 + 2\ RNO_3 \qquad [12]$$

consequently the rate of elution is going to be governed at least to some extent by the concentration of $Pu(NO_3)_4$ in the aqueous phase. Brothers et al (4) have shown that even at 1 M HNO_3 the predominant plutonium-IV species is $Pu(NO_3)_4$ thus it is not surprising that plutonium is eluted slowly with dilute nitric acid (1) (14). In the presence of excess reducing agent the concentration of $Pu(NO_3)_4$ in the aqueous phase is reduced virtually to zero by reduction to plutonium-III and the rate of the elution reaction is greatly increased (15). In the anion exchange pilot plant at Chalk River two column volumes of 0.15 M hydroxylamine nitrate at a flow rate of 0.375 ml./min./cm.^{2} give complete elution (16).

The reactions governing the elution of plutonium-III from Dowex 50 or Dowex 50-W with strong nitric acid are complex and probably involve the absorption and elution of some plutonium-IV species as well as the elution and oxidation of plutonium-III. The results of a series of elution experiments are given in Figure 7. In these experiments 50-60 mesh Dowex 50 and Dowex 50-W resin columns were loaded to 1% breakthrough with plutonium-III from a 0.05 N hydroxylamine - 0.95 N nitric acid solution. The columns were washed with 1.0 N nitric acid to displace the hydroxylamine and were then eluted with 8 N nitric acid at flow rates varying from 0.5 to 4.0 ml./min./cm.^{2}. A consideration of Figure 7 shows that the rate of elution appears to be dependent only on the time of contact at least over the flow rate range studied. A plot on semi logarithmic paper of the experimental points using the square root of the time of contact as abscissa, Figure 8, gives a single straight line for Dowex 50 and a second straight line for Dowex 50-W. These lines can be represented by the equations $C/C_o = e^{-0.082\sqrt{t}}$ for 50-60 mesh Dowex 50 and $C/C_o = e^{-0.112\sqrt{t}}$ for Dowex 50-W, where C/C_o is the fraction of plutonium left on the resin after any given time of contact, t, and t is expressed in minutes. The solid lines in Figure 7 are plots of these functions and show the fit of the elution equations to the experimental points. It should be possible to obtain an equation of similar form for 20-50 mesh Dowex 50 but this has not as yet been done. It will be noted that no mention of column size was made in the above treatment. The volumes of eluant involved in these elutions are very large in comparison to the volumes of the columns consequently the movement of the exchange band through the column is probably a relatively minor factor. Thus the equations should be virtually independent of column dimensions.

CONCLUSIONS

1) The equilibrium between plutonium-III and Dowex 50 can be described adequately by the equation

$$\frac{C_{PuR_3}}{(V - C_{PuR_3})^3} = K_2{}^1 \frac{C_{Pu^{+3}}}{C^3_{H^+}} \qquad [13]$$

where $K_2{}^1 \approx 24$. This equation can be solved as a cubic equation or by trial and error to give a value of the plutonium concentration on the resin in equilibrium with any practical nitric acid solution. This gives, for any column, a value of C_T, the saturation capacity. The capacity to breakthrough, C_E, can be determined from the equation

$$C_E = C_T \left(\frac{1 - 35\sqrt{v}}{h_t}\right) \qquad [14]$$

$K_2{}^1$ for Dowex 50-W varies from ~21 at 0.5 M nitric acid to ~6.6 at 2.4 M nitric acid. Exchange heights for Dowex 50-W have not as yet been obtained. Dowex 50-W is much more readily eluted than is Dowex 50 and consequently may find considerable usage.

2) The equilibrium between plutonium-IV and 8% and 1% cross-linked Dowex 1 can be represented by the equation

$$\frac{C_{R_2Pu(NO_3)_6}}{\left(V - C_{R_2Pu(NO_3)_6}\right)^2} = K_1{}^1 \; C_{Pu(NO_3)_4} \; \gamma_{Pu(NO_3)_4} \qquad [15]$$

where $K_1{}^1$ can be obtained from Figure 4 and $\gamma_{Pu(NO_3)_4}$ can be obtained from Figure 2. This equation can be solved as a simple quadratic or by trial and error to give a value of the plutonium concentration on the resin in equilibrium with any given nitric acid solution. As above this gives, for any column, a value of C_T, and C_E can be calculated from the equations

$$C_E = C_T \left(\frac{1 - 150\, d\sqrt{v}}{h_t}\right) \qquad [16]$$

for 8% cross-linked resin and

$$C_E = C_T \frac{(1 - 85\, d\sqrt{v})}{h_t} \qquad [17]$$

for 1% cross-linked resin. It should be emphasized that equations [16] and [17] are based on a minimum number of results and should be further confirmed before too much faith is put in them.

3) The equilibrium between plutonium-IV and Dowex 1 in the presence of uranium is complicated by the formation of the species $R\, UO_2(NO_3)_3$ and $R_2\, UO_2(NO_3)_4$ and it has not been possible to resolve this system quantitatively.

4) The elution of plutonium from 8% cross-linked 50-100 mesh Dowex 50 and Dowex 50-W can be represented respectively by the equations

$$C/C_o = e^{-0.082\sqrt{t}} \qquad [18]$$

and $$C/C_o = e^{-0.112\sqrt{t}} \qquad [19]$$

Elution from 20-50 mesh resins could probably be expressed by equations of the same form but experiments to evaluate the constants have not been completed.

5) The elution of plutonium from anion exchange resins is rapid with a reducing agent but slow without one. A fairly complete study of elution with dilute acid is given by Ryan and Wheelwright (14).

SUGGESTIONS FOR FUTURE WORK

1. The K_2^1 values, quoted for use in equation [15] , have been obtained on 50-100 mesh resins. The validity of these values should be checked with resin of another mesh size, preferably 20-50 mesh.

2. The equation for the exchange height in the plutonium-III - Dowex 50 system, $h_z = 70\, d\sqrt{v}$, should be further checked on 20-50 mesh resin.

3. K_1^1 values for use in equation [15] should be obtained for nitrate salt solutions and all values should be checked on 20-50 mesh 8% cross-linked resins.

The values quoted have been obtained on 50-100 mesh Dowex 1, 1% cross-linked.

4. Equations [16] and [17], representing the exchange heights in the plutonium-IV - Dowex 1 system, are based on a minimum number of experimental results and should be checked on both 50-100 and 20-50 mesh resins.

5. Equations should be obtained to represent the elution of plutonium-III from 20-50 mesh Dowex 50 and Dowex 50-W.

ACKNOWLEDGMENT

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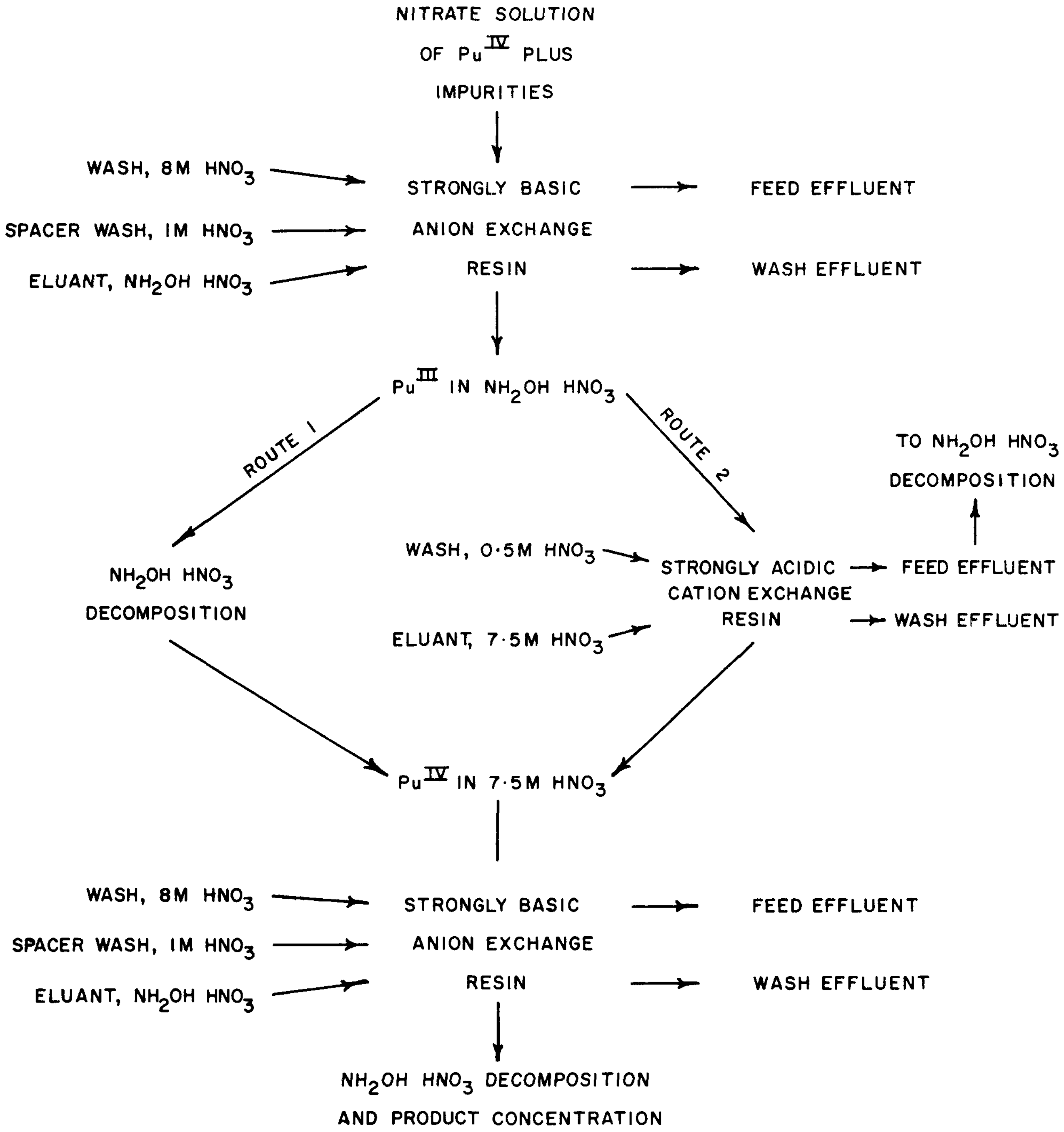


FIGURE I

THE PROCESS FLOW

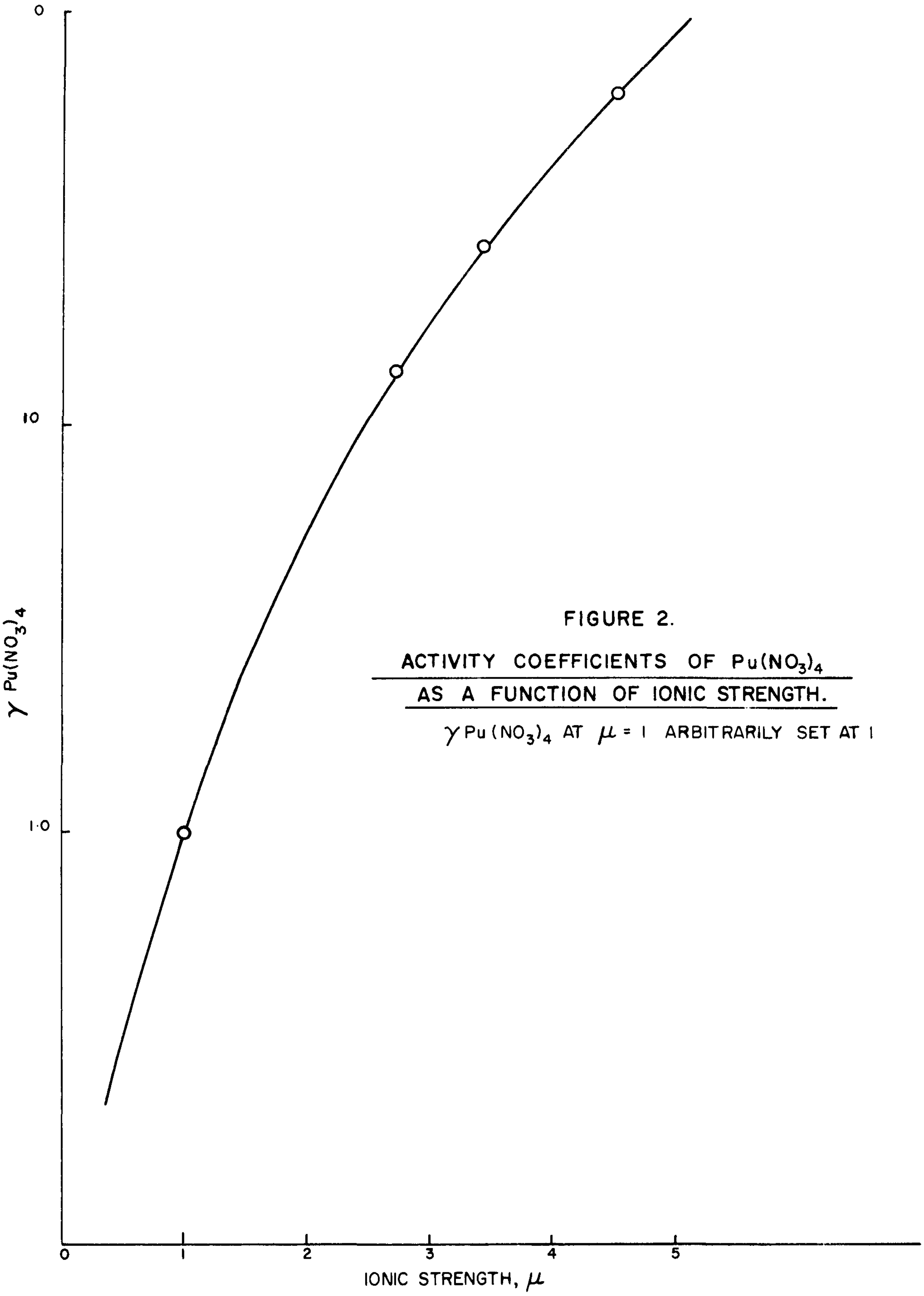


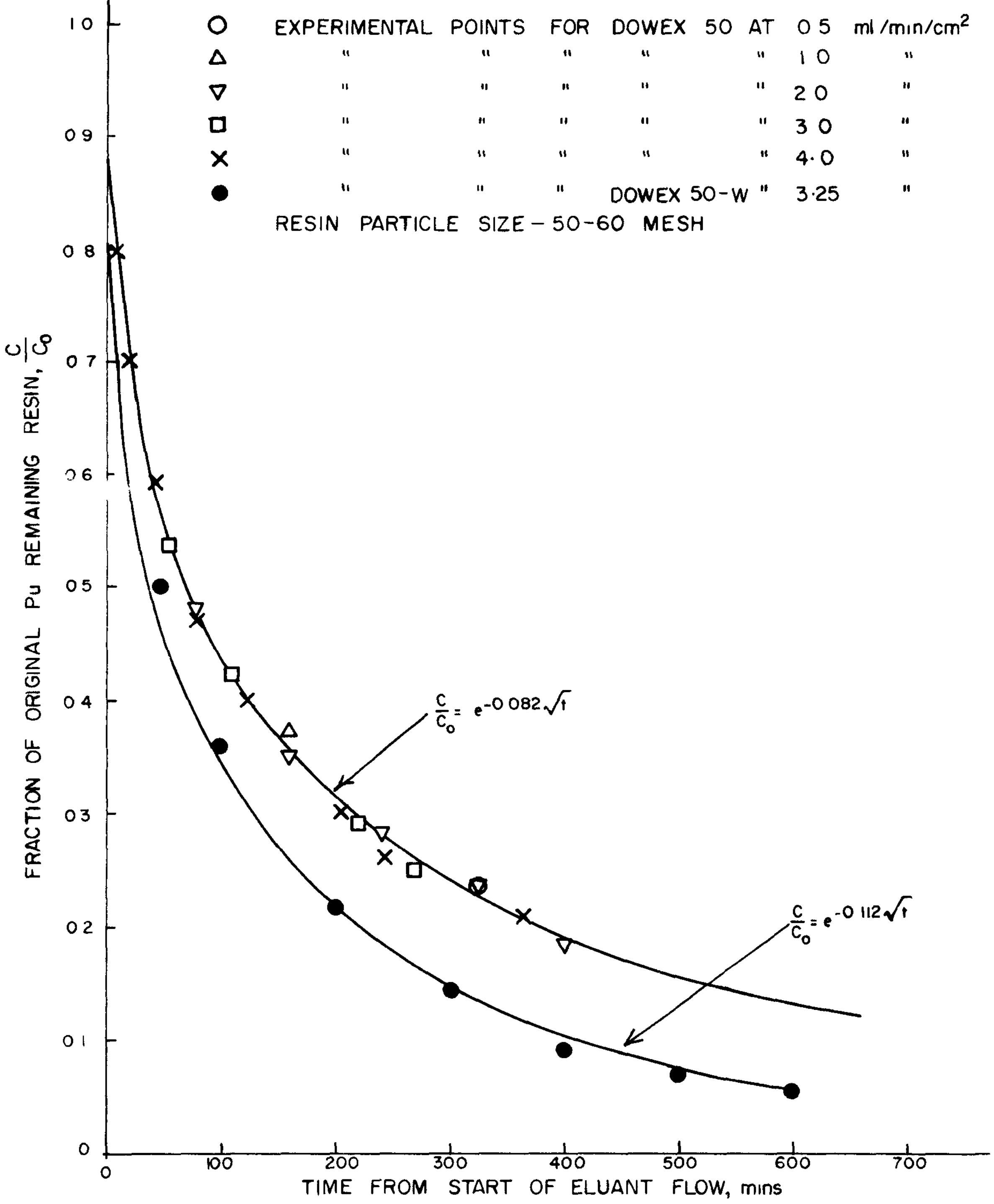
FIGURE 2.

ACTIVITY COEFFICIENTS OF $Pu(NO_3)_4$ AS A FUNCTION OF IONIC STRENGTH.

γ $Pu(NO_3)_4$ AT $\mu = 1$ ARBITRARILY SET AT 1

FIGURE 7.

ELUTION OF PLUTONIUM III FROM DOWEX 50 AND DOWEX 50 - W. WITH 8 M HNO_3



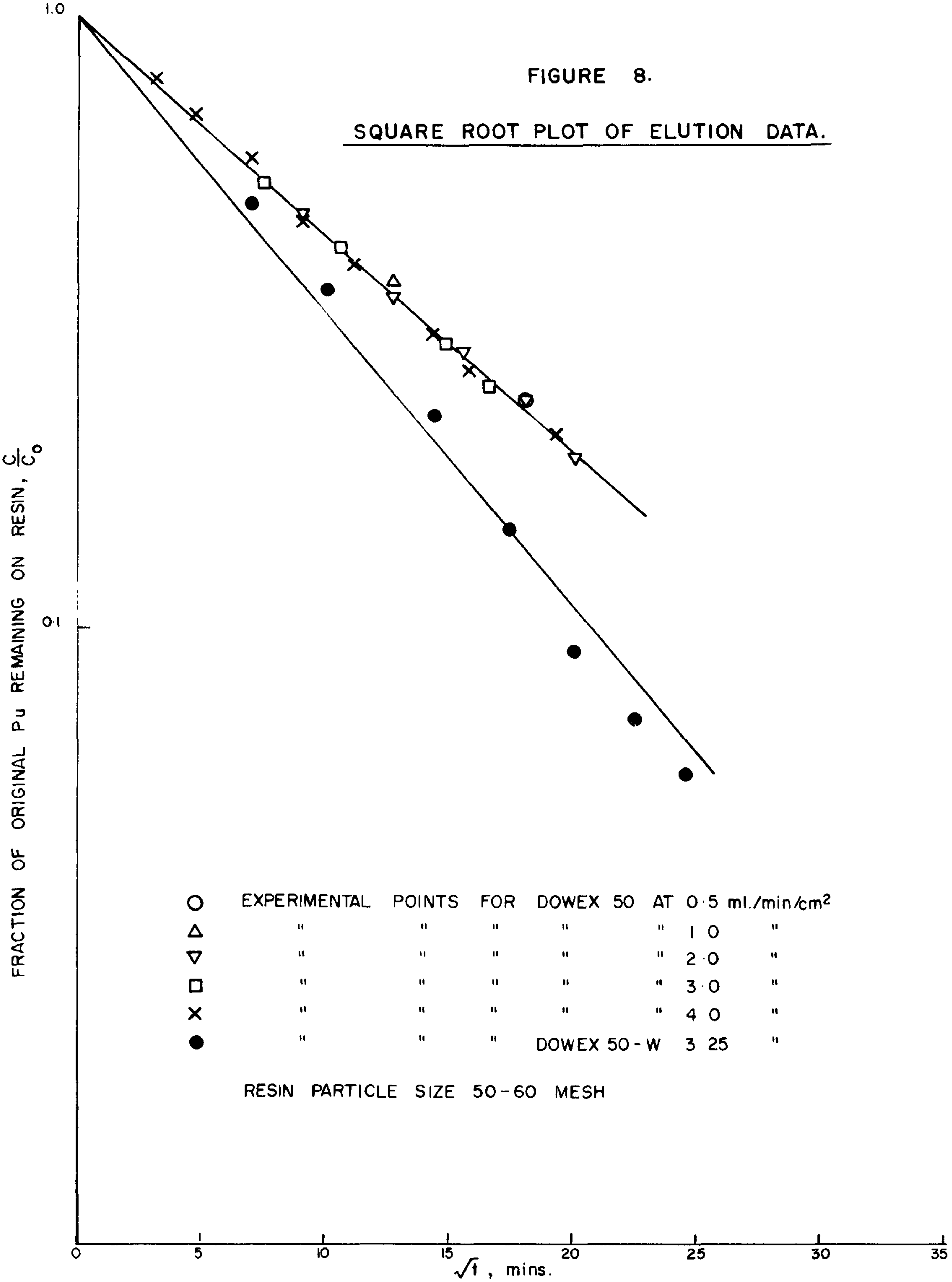


FIGURE 8.

SQUARE ROOT PLOT OF ELUTION DATA.