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THE 1A HYBRID FLOWSHEET

by

B. V. Coplan
J. K. Davidson
W. O. Hass
E. L. Zebroski

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I. INTRODUCTION

A "hybrid" 1A flowsheet in which the extraction section operates substantially acid while the scrub section is acid deficient has been proposed at Argonne and KAPL.

It is believed that in this way it may be possible to obtain the low plutonium losses possible with the acid flowsheet (ANL) as well as the high decontamination factors demonstrated for the acid-deficient flowsheet (ORNL). A flowsheet incorporating 0.2M acid in the 1AX stream has been tentatively adopted by the Hanford group for the Hanford Redox Plant.

This report is concerned with the effect of added nitric acid on the performance of the acid deficient 1A flowsheet. A preliminary Hybrid 1A Flowsheet is presented. The need for close specification of the acidities of the entering streams in such a flowsheet is evaluated. Finally, preliminary batch-countercurrent decontamination and plutonium recovery data are included.

II. DISCUSSION

The development of the acid deficient ORNL flowsheet was prompted by the relatively poor decontamination obtainable with the acid flowsheet. The hybrid flowsheet has been proposed in an attempt to retain the excellent decontamination obtainable with the acid deficient ORNL flowsheet while avoiding the erratic and occasionally relatively high plutonium losses which have been encountered. (cf. ORNL-289).

For those fission products whose behavior is reversible the decontamination in the hybrid flowsheet should be closely comparable to the strictly acid deficient system. For ruthenium, whose behavior is not reversible, there is reason to believe that decontamination may deteriorate if any part of the column is acid, but with an acid deficient scrub, decontamination should be significantly higher than for the acid flowsheet.

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The increased recovery of plutonium in the hybrid flowsheet results from the three to fourfold increase in extraction factor of Pu (VI) in going from $-0.2M$ to $+0.2M$ acid in the aqueous phase. In the ORNL flowsheet acid is scrubbed out of the aqueous phase increasing the acid-deficiency of the IAW to more than 0.3. The low extraction factor prevailing at these acidities makes from 14 to 16 extraction stages necessary for Pu (VI) recovery under (ORNL) flowsheet conditions. Plutonium losses due to reduction in the column or adsorption on suspended matter may also be largely nullified due to the acid gradient in the hybrid column.

The hybrid system permits the use of an acid IAF stream which is desired. This affords increased convenience in feed preparation and may eliminate a possible source of plutonium loss resulting from addition of caustic. The effect of acid versus acid deficient IAF on decontamination is as yet undetermined.

The boundary conditions adopted for this study of the hybrid flowsheet were:

- (1) At least three acid extraction stages
- (2) Acid deficient scrub stages
- (3) Acid IAF Stream

To satisfy the boundary conditions it is necessary to control the acid gradient throughout the system. A clear picture of the nitric acid equilibrium behavior is therefore desirable.

III. THEORETICAL

A. 1A NITRIC ACID SYSTEM

The 1A nitric acid system will be discussed on the basis of the schematic diagrams, Figures 1 and 2. These diagrams were drawn using a nitric acid equilibrium line of approximately the true shape, and a system of concentration and flow units in which the operating lines are straight.

Several facts about this type of diagram are important:

1. It can be shown that for any countercurrent extraction with constant flow

parameters, the two operating lines intersect at the central feed concentration. (See Appendix #I).

2. In the acid-deficient portion of the diagram the slope of the equilibrium line is very low and there is a wide angle between the equilibrium line and the scrub operating line. The significance of this wide angle is seen when stages are stepped off on the diagram. It becomes apparent that only one stage will be acid-deficient unless the IAP acid concentration is essentially on the equilibrium curve, permitting a number of stages to be stepped off in the corner of the diagram where the operating and equilibrium lines approach. The origin of the scrub section operating line is the IAS-IAP point. The slope of the scrub operating line is fixed by the relative flow rates and thus this line can be drawn from its origin with the correct slope completing the scrub section part of the McCabe-Thiele type diagram. The IAF concentration is known and can be drawn as a vertical line on the diagram. Its intersection with the scrub operating line is a point on the extraction section operating line, as shown in Appendix I. The slope of the extraction operating line is also fixed by the relative flow rates and therefore this line can be drawn on the diagram. The ordinate of its upper end is the value for the acid concentration of the IAX stream which can be chosen, permitting the completion of the entire acid diagram. The three possible choices are shown in Figure 2.

(a) The IAX acidity is higher than the ordinate of the intersection of the extraction operating line and the equilibrium curve (I). It is no longer possible for the IAP to have an acid concentration substantially in equilibrium with the IAS, and the operating line will be displaced upward as shown. The entire extraction section will be acid. In addition, all except perhaps one or two of the scrub section stages will be acid since their concentrations will fall in the pinch at the intersection of the scrub section operating line and the equilibrium curve.

(b) The IAX acidity coincides with the ordinate of the intersection (II). As

before, all of the extraction stages are acid. The concentration of the organic phase leaving the stage above the feed stage must fall at or below the intersection of the scrub section operating line and equilibrium curve. Since an infinite number of stages would be required to reduce the acid concentration to this point, the operating line must rise infinitesimally so that only the available number of stages are required. As before, most of the scrub section stages will be acid.

(c) The LAX acidity is lower than the ordinate of the intersection (III). The number of acid stages can now be found by stepping them off on the diagram. In general, there will be only a few acid stages, the greater portion of the stages being in the acid-deficient pinch region.

The method of plotting the LA nitric-acid diagram is illustrated in Appendix #II.

B. STABILITY OF THE HYBRID-TYPE FLOWSHEET

The stability of the hybrid-type flowsheet is dependent upon the nitric acid system. Cases I and II above are examples of unstable operation since in each case the scrub section was not acid deficient. Case III represents stable operation on the mixed-type flowsheet. It can be seen that before any such flowsheet can receive serious consideration, the limits within which stable operation is assured must be defined. The problem of stability thus centers on the relative positions of the LAX-LAW operating point and the lean end equilibrium line. The position of the LAX-LAW point depends upon:

- (1) The LAX, LAS, and LAF feed concentrations
- (2) The feed flowrates

If realistic estimates of the error in each of the above quantities are available, the stability of any mixed flowsheet can be checked or a stable flowsheet designed.

The feasibility of a mixed-type flowsheet depends therefore on the accuracy of the control laboratory analyses and upon the reliability of the feed stream flowrate control.

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C. HANFORD NO. 2 FLOWSHEET

The acid diagram for the 1A Bank on the Hanford No. 2 flowsheet is shown in Figure 3. The diagram is drawn on the non-UNT basis from the published nominal flowsheet feed concentrations.

Since no analytical accuracies were specified for the Hanford flowsheet, present KAPL Analytical Section values for the limiting errors in each feed stream acid analysis were used. These accuracies are based on the best standard method available, and represent more than ordinary analytical care.

The nominal feed stream acid concentrations and their limiting absolute errors are:

	<u>N HNO₃</u>
1AF	-.20 ± .02
1AS	-.20 ± .02
1AX	+.20 ± .005

The diagram is drawn using the nominal flowrate and does not consider variations from the nominal.

The shaded area in the upper right hand side of the diagram represents the possible positions of the 1AX-1AW point. The shaded area falls partially beyond the equilibrium line and represents a definite probability for entirely acid operation. Since flowrate variations have been neglected, this probability is conservative.

It would seem on the basis of the acid diagram that the nominal Hanford No. 2 flowsheet is not entirely stable. Consideration of the acid diagram shows however that a slight reduction in the 1AX acid molarity together with control of feed molarities within the limits stated above would made the flowsheet stable.

D. EXPERIMENTAL CRITERIA FOR STABILITY CONTROL

A method of analysis involving mixing of portions of the three feed streams was suggested to Dr. Rider of the Analytical Chemistry Section. Two tests have been

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developed by Dr. Rider which show promise of reducing the analytical limits used in the foregoing discussion, and thus reducing the shaded areas in the acid diagram.

TEST I. A standard test solution is prepared for each feed. The standard is adjusted to an HNO_3 or NaOH molarity exactly equal to the flowsheet molarity but of an acidity or basicity that will neutralize the feed.

A portion of the feed is mixed with an equal volume of the test solution. Thus if the feed is on flowsheet, the resulting solution will have zero free acid. The pH of this solution is measured and must be within specified, empirically determined limits. Comparison of the measured pH with a calibration curve enables the error from the flowsheet value to be determined.

TEST II. All three of the feeds found acceptable from Test I are mixed in proportions based on flowsheet concentrations that will give a neutral solution. The pH of the aqueous phase is measured and must be within specified, empirically determined limits.

Incorporation of these tests will reduce the limiting absolute errors to:

IAF	$\pm .01 \text{ M}$
IAS	$\pm .01 \text{ M}$
IAX	$\pm .005 \text{ M}$

Details of the two test methods together with experimental data are given in the KAPL monthly report for March 1950.

E. KAPL NO. 1 FLOWSHEET

The acid diagram for the IA Bank on the KAPL No. 1 flowsheet is shown in Figure 4. The diagram is drawn on the non-UNT basis, and the calculation of the concentrations and slopes involved is shown in Appendix #II.

The nominal feed stream molarities are shown below. The limiting analytical errors obtainable with the new analytical methods described in the preceding section

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are listed. These lower errors as well as a flowrate variation of 1% were used in plotting Figure 4.

	$\text{Na}_2\text{Cr}_2\text{O}_7$, m/l	U, m/l	Al, m/l	HNO_3 , m/l	Relative Flowrate
IAF	0.1	2.00	----	$0.11 \pm .01$	$1 \pm .01$
IAS	0.01	----	$1.65 \pm .01$	minus $0.50 \pm .01$	$1 \pm .01$
IAX	----	----	----	$0.190 \pm .005$	$4 \pm .04$

The shaded area representing the possible points of intersection of the two operating lines and the area representing the possible location of the IAX-LAW point are still quite large, and make it possible to define the number of acid stages only within relatively wide limits. The diagram is, however, stable with no probability for entirely acid operation if the accuracies of the feed analysis and feed rates are maintained.

The range of IAS aluminum molarity in hybrid flowsheets has been investigated from 2.0 M down to 1.3 M. (Runs G1-10, 12, 16, 17, 20. KAPL Monthly Reports October 1949 - March 1950). Low aluminum molarity is desirable for several reasons other than the reduction in salt consumption. Lowered salt strength decreased the distribution coefficient for nitric acid in the lean end of the extraction section and thereby makes the angle between the operating line and equilibrium line more acute. The same number of acid stages may now be obtained with a IAX-LAW point further from the intersection point of the equilibrium and operating lines and the acid diagram is therefore made more stable. There is of course a minimum salt strength that will still allow acceptable uranium recovery. Runs in the pump-mix mixer settler have defined this minimum molarity for the KAPL No. 1 flowsheet to be 1.5-1.6 molar in the IAS. It is on this basis that 1.65 M Al is chosen for the IAS stream. This represents a 17.5% reduction in the aluminum consumption of the ORNL acid deficient flowsheet. (2M IAS).

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The KAPL No. 1 flowsheet makes use of an acid feed which should be obtainable from dissolver solution by an acid-butt, avoiding the need for running the dissolver to acid deficiency or addition of caustic to the acid dissolver solution.

IV. EXPERIMENTAL

A. COLD PUMP-MIX MIXER-SETTLER RUNS

Two cold experimental mixer-settler runs have been made with conditions closely approximating the KAPL No. 1 flowsheet. Runs (G1-12, G1-20).

The data for these runs are presented in Tables 1 and 2 in Appendix #III. The uranium and nitric acid data for Run G1-12 are plotted on the non-UNT basis in Figures 5 and 6 respectively. Figures 7 and 8 show the same data for Run G1-20. In each run the break between acid and acid deficient stages occurred at the feed stage. The uranium loss from eleven operating extraction stages in G1-12 was 0.0005% while from Run G1-20 the loss was 0.002%. Operability in both runs was high, no emulsion being present in any of the stages.

B. BATCH COUNTERCURRENT RUNS (HOT)

Three batch-countercurrent runs have been made using the KAPL No. 1 flowsheet. The first run was made only to test plutonium recovery.

The next two runs were made using irradiated Clinton metal spiked with dissolver solution from an ORNL run.

Due to the age of the material the zirconium content was relatively low.

After three throughputs across the scrub section the LAP gross beta-gamma analysis became essentially constant. All stages were sampled after four throughputs. Stage analyses for uranium, nitric acid, and plutonium are presented in Figures 9, 10, and 11 in Appendix #IV, and in Tables 5, 6, and 7.

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1. Dissolving Procedure

Clinton metal from which the jacket had been previously removed was dissolved in 60% nitric acid. An efficient reflux condenser was mounted on the dissolver; air sweeping was not used. A final density of 1.89 was reached after a total time of five hours and a final temperature of 100°C. The solution was then about 0.23 acid deficient.

2. Oxidation Procedure

The metal solution was spiked with about 5% by volume of dissolver solution from ORNL Run #52R and adjusted to 0.14 M nitric acid and 0.09 M sodium dichromate in 1.94 M UNH. This solution was heated at 85°C for six hours. A holding-oxidant- LaF_3 analysis at this point showed 0.3% of the initial alpha activity was carried. Later analysis established that the Americium-Curium content of the solution amounted to about 0.2% of the alpha activity.

3. Decontamination Results

The decontamination factors obtained to date, both overall and individual, are only moderately good. (Table 3). Comparable data from acid deficient runs at Argonne (ANL-4292, P. 11) in which the hexone phase contained 0.1 and 0.2 nitric acid showed excellent decontamination, the individual values being indistinguishable from purely acid-deficient runs. The use of acid LAX during the latter half of Run 52R at Oak Ridge, on the other hand, gave rise to serious and progressive deterioration in the decontamination factor, particularly for ruthenium. (ORNL-463, P. 48).

The relatively low decontamination obtained in the batch countercurrent runs to date is probably due to the limited number of acid deficient stages which were employed. This in turn was due to the difficulty in getting sufficiently reliable acid analysis (prior to the introduction of the tests using the pH criterion), and in the case of Run #9, an error in setting of flowrates.

It is possible that the poor result obtained in the ORNL run was due to

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operating with an acid balance in the column in which only one or two stages in the scrub section were acid deficient.

An alternative possibility which must be considered is that the reflux of ruthenium in the region of change from acid to acid deficient conditions is sufficiently great that the ruthenium concentration in the column will continue to increase indefinitely. This could be the case if the distribution coefficient of some ruthenium species was reversibly dependent on acidity so that its exit from the ends of the column occurred more slowly than the rate at which it was fed into the column. This alternative cannot be evaluated until sufficiently long runs are made under controlled conditions.

It is necessary to determine whether it is sufficient to provide a minimum number of acid deficient scrub stages, or whether it is also essential to maintain the feed plate itself acid-deficient. Batch extraction followed by acid deficient batch scrubs with fresh scrub solutions show that if the initial extraction is from an acid deficient solution, the limiting decontamination is about three fold higher than if the initial extraction is from an acid medium. The limiting decontamination in the case of acid extraction was found to be about dF 3.7, obtained after four to five scrub stages. (The limiting decontamination is defined as the value at which the extraction factor for the fission products remaining in the hexone is greater than unity.) This is an indication that considerably more than five acid deficient countercurrent scrub stages are necessary to obtain adequate decontamination and that it is probably desirable to maintain the feed plate acid deficient.

It is evident that if the potential advantages of the hybrid flowsheet are to be realized very good control of acidities and flowrates of the feed streams must be achieved and routinely maintained.

4. Plutonium and Uranium Recovery

No difficulty has been experienced in obtaining plutonium and uranium recovery equal to or well in excess of specifications on all runs. (Tables 5, 6, and 7.)

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APPENDIX I - INTERSECTION OF OPERATING LINES

For a countercurrent extraction with constant flow parameters, the two operating lines intersect at the central feed concentration. This can be shown as follows:

A = flowrate of aqueous phase. Constant at any cross section in section M.

$A+A'$ = flowrate of aqueous phase. Constant at any cross section in section N.

Expressed in same units as A .

H = flowrate of organic phase. Constant throughout the bank. Expressed in same units as A .

X = concentration of solute in aqueous phase. Expressed in units consistent with flow-rate units above.

Y = concentration of solute in organic phase expressed in same units as X .

Material balance around left end of the bank and stage m :

$$Hy_{m+1} + Ax_o = Hy_p + Ax_m$$

$$y_{m+1} = \left(\frac{A}{H}\right)x_m + \left(y_p - \frac{A}{H}x_o\right)$$

Material balance about left end of the bank and stage n :

$$Hy_{n+1} + A'x_f + Ax_o = Hy_p + (A+A')x_n$$

$$y_{n+1} = \left(\frac{A+A'}{H}\right)x_n + \left(y_p - \frac{A'}{H}x_f - \frac{A}{H}x_o\right)$$

at the intersection, x_1, y_1 :

$$y_1 = \left(\frac{A}{H}\right)x_1 + \left(y_p - \frac{A}{H}x_o\right) \quad (1)$$

$$y_1 = \left(\frac{A+A'}{H}\right)x_1 + \left(y_p - \frac{A'}{H}x_f - \frac{A}{H}x_o\right) \quad (2)$$

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Subtract equation 2 from 1:

$$0 = -\frac{A'_1 x_1}{H} + \frac{A'_1 x_f}{H}$$

$$\frac{A'_1 x_1}{H} = \frac{A'_1 x_f}{H}$$

$$\text{Therefore: } x_1 = x_f$$

APPENDIX II - CALCULATIONS FOR THE 1A NITRIC ACID DIAGRAM (KAPL NO. 1 FLOWSHEET)

The calculations for the 1A nitric acid diagram for the KAPL No. 1 Flowsheet are shown below. Dichromate is not included in this calculation or in Figure 4.

Feed Acid Molarities On The Non-UNT Basis

IAS

1.65 M Al
minus .5 M HNO₃
.3 M NaNO₃

$$P_{25} = 1.0012 + .155(1.65) - .032 (.5) + .032 (.3)*$$
$$= 1.256$$

$$X = \frac{-(.5 - .01)63}{1256} = -.0251 \pm .0005 \quad \frac{\text{g.HNO}_3}{\text{g.Non-UNT}}$$

IAF

2.00 M U
choose 0.11 M HNO₃

$$P_{25} = 1.0012 + 2(.318) + .11 (.032)$$
$$= 1.640$$

$$X = \frac{(0.11 - .01)63}{1640 - 2(448.1)} = .0093 \pm .0008 \quad \frac{\text{g.HNO}_3}{\text{g.Non-UNT}}$$

Operating Line Slopes On Non-UNT Basis

Flow Ratios: IAS:IAF:IAW = 1:1:4

Assume:

$$\frac{\text{Volume IAS}}{\text{Volume IAW}} = .555 \quad (\text{determined from 1A experimental runs})$$

*density equation, HW-12045

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Scrub Section

$$\frac{A}{H} = \frac{(1.00 \pm .01) 1.256}{(4.00 \pm .04) .808^{**}} = .389 \pm .008$$

Extraction Section

$$\begin{array}{lll} \text{IAW aluminum molarity} & = & .555(1.65) = .916 \\ \text{NaNO}_3 & " & = .555(.3) = .17 \\ \text{HNO}_3 & " & .3 \text{ (initial assumption)} \end{array}$$

$$\begin{aligned} P_{\text{IAW}} &= .9987 + .916(.156) + .17(.052) + .3(.032) \\ &= .9987 + .143 + .009 + .0096 = 1.160 \end{aligned}$$

$$\frac{A}{H} = \left(\frac{1}{.555} \right) \frac{(1.00 \pm .01) (1.160)}{(4.00 \pm .04) .808} = .645 \pm .013$$

Lean End Equilibrium Line

$$\begin{aligned} \mu &= \frac{1}{2} \sum c_i z_i^2 = \frac{1}{2} [(Al^{+++})^2 + (Na^+)^2 + (H^+)^2 + (NO_3^-)^2] \\ &= \frac{1}{2} (.92(9) + .17 + .3 + 3.23) = \frac{1}{2} (11.98) \\ &= 5.99 \end{aligned}$$

$$\frac{E_H}{A} \text{ HNO}_3 = .77 \text{ (from plot of distribution coefficient vs. } \mu, \text{ Page 27, KAPL-285)}$$

Calculation of a Non-UNT point on the lean end of the equilibrium line:

$$\begin{aligned} \text{When } x &= .3 \text{ M} \\ y &= .231 \text{ M} \end{aligned}$$

Converting to non-UNT basis:

$$X = \frac{.3(63)}{1160} = .0163 \qquad Y = \frac{.231(63)}{808} = .018$$

The nitric acid diagram may now be drawn. It remains for the IAX acid value to be chosen to complete the diagram. This is done by choosing a maximum value from the diagram, and subtracting the analytical error.

**assumed density based on approximate concentration

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From the diagram:

$$Y_{max.} = .0156$$

$$Y_{max.} = \frac{.0156(808)}{63} = 0.20 \text{ M}$$

$$Y_{IAX} = 0.195 \pm .005$$

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APPENDIX III - EXPERIMENTAL DATA

TABLE I

Run No. G1-12

Operating Conditions:

Two banks - 10 stages per bank

Stages in operation: 6 in scrub section (#1 and #5 off)

11 in extraction section (#20 off)

IAF introduced at stage #9, IAS at stage #1, and IAX at stage #20.

Average feed rates (cc/min): IAS 19.5, IAX 79.2, IAF 19.5.

Room temperature: 71°F

Stirrer rpm 700

Analysis of Feeds:

IAS - 1.66 M Al(NO₃)₃, minus 0.472 N HNO₃, 1.263 Sp.Gr. at 25°C

IAX - 0.213 N HNO₃, 0.810 Sp.Gr. at 25°C

IAF - 2.02 M UNT, 0.106 N HNO₃, 1.648 Sp.Gr. at 25°C

<u>Stage No.</u>	<u>Phase</u>	<u>UNT</u> <u>M/L</u>	<u>HNO₃</u> <u>M/L</u>	<u>Sp. Gr.</u> <u>at 25°C</u>	<u>g UNT</u> <u>g Non-UNT</u>	<u>g HNO₃</u> <u>g Non-UNT</u>
1	Stirrer off					
2	Hex	0.46	0.00	0.963	0.272	0.000
	Aq	.12	- .44	1.294	.0434	-0.022
3	Hex	Not sampled				
	Aq					
4	Hex	.50	.00	.976	.298	0.000
	Aq	.14	- .41	1.300	.0507	-0.021
5	Stirrer off					
6	Hex	.51	.03	.980	.304	+0.0025
	Aq	.11	- .22	1.290	.0397	-0.0111
7	Hex	Not sampled				
	Aq					
8	Hex	.48	+ .07	.974	.283	+0.0058
	Aq	.083	- .04	1.279	.0300	-0.0020
9	Hex	.48	+ .10	.973	.284	+0.0083
	Aq	.45	+ .11	1.293	.1848	+0.0064
10	Hex	.22	+ .13	.884	.1255	+0.0104
	Aq	.21	+ .15	1.222	.0834	+0.0076
11	Hex	.10	+ .16	.841	.0563	+0.0127
	Aq	.091	+ .19	1.189	.0355	+0.0104
12	Hex	.045	+ .185	.826	.0250	+0.0145
	Aq	.039	+ .22	1.173	.01513	+0.0120
13	Hex	.018	+ .195	.816	.0100	+0.0152
	Aq	.014	+ .23	1.166	.0054	+0.0125
14	Hex	.0066	.20	.813	.00365	+0.0156
	Aq	.0044	.24	1.161	.0017	+0.0130
15	Hex	.0018	.21	.811	.000996	+0.0163
	Aq	.0011	.25	1.160	.00042	+0.0136

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TABLE I - Continued

Stage No.	Phase	UNT M/L	HNO ₃ M/L	Sp. Gr. at 25°C	g UNT g Non-UNT	g HNO ₃ g Non-UNT
16	Hex	.00046	.22	.811	.00025	+0.0171
	Aq	.00030	.26	1.160	.00011	+0.0141
17	Hex	.000130	.22	.811	.000719	+0.0171
	Aq	.000068	.26	1.160	.000026	+0.0141
18	Hex	.000030	.22	.810	.0000165	+0.0171
	Aq	.000020	.26	1.160	.0000077	+0.0141
19	Hex	.000008	.22	.810	.00000444	+0.0171
	Aq	.000005	.26	1.164	.0000017	+0.0141
20	Stirrer off					

TABLE II

Run No. G1-20

Operating Conditions:

Two banks - 10 stages per bank

Stages in operation: 7 in scrub section (#1 off)

11 in extraction section (#20 off)

IAF introduced at stage #9, IAS at stage #1, and IAX at stage #20.

Average feed rates (cc/min): IAS 20.3, IAX 79.5, IAF 19.6.

Stirrer rpm 700

Analysis of Feeds:

IAS - 1.66 M Al(NO₃)₃, minus 0.51 M HNO₃, ~ 0.335 M NaNO₃, Sp.Gr. 1.261 at 25°C

IAX - 0.20 M HNO₃, Sp.Gr. 0.808 at 25°C

IAF - 1.99 M UNH, +0.119 M HNO₃, Sp.Gr. 1.636 at 25°C

Stage No.	Phase	UNT M/L	HNO ₃ M/L	Sp. Gr. at 25°C	g UNT g Non-UNT	g HNO ₃ g Non-UNT
IAS	Aq	-----	-0.51	1.261	-----	-0.0255
2	Hex	0.463	-0.001	.965	0.2739	-0.000083
	Aq	0.130	-0.46	1.275	0.0478	-0.0238
3	Hex	0.491	0.00	.977	0.2907	0.00
	Aq	0.155	-0.48	1.303	0.0563	-0.0245
4	Hex	0.498	0.00	.978	0.2956	0.00
	Aq	0.155	-0.51	1.304	0.0563	-0.0260
5	Hex	0.496	-0.001	.977	0.2945	-0.000083
	Aq	0.147	-0.49	1.305	0.0532	-0.0249
6	Hex	0.499	-0.0005	.978	0.2964	-0.000042
	Aq	0.172	-0.51	1.305	0.0628	-0.0262
7	Hex	0.498	-0.002	.979	0.2952	-0.000167
	Aq	0.160	-0.47	1.296	0.0586	-0.0242
8	Hex	0.488	.009	.978	0.2880	+0.000748
	Aq	0.138	-0.19	1.291	0.0503	-0.00974

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TABLE II - Continued

<u>Stage No.</u>	<u>Phase</u>	<u>UNT</u> <u>M/L</u>	<u>HNO₃</u> <u>M/L</u>	<u>Sp. Gr.</u> <u>at 25°C</u>	<u>g UNT</u> <u>g Non-UNT</u>	<u>g HNO₃</u> <u>g Non-UNT</u>
9	Hex	0.483	.08	.978	0.2842	+0.00621
	Aq	1.99	0.119	1.636	1.195	+0.01
1AF	Aq	0.465	0.01	1.288	0.193	+0.00063
10	Hex	0.214	0.12	.877	0.1228	0.00968
	Aq	0.192	0.12	1.215	0.0762	0.00670
11	Hex	0.093	0.16	.839	0.0523	0.0126
	Aq	0.086	0.15	1.192	0.0334	0.00819
12	Hex	0.406	0.17	.820	0.0227	0.0134
	Aq	0.0348	0.18	1.171	0.0135	0.00981
13	Hex	0.0145	0.18	.810	0.0081	0.0141
	Aq	0.0119	0.19	1.168	0.00459	0.0103
14	Hex	0.0504	0.180	.809	0.0028	0.0141
	Aq	0.00342	0.21	1.161	0.00132	0.0114
15	Hex	0.00142	0.182	.807	0.00079	0.0143
	Aq	0.00057	0.22	1.162	0.000220	0.0119
16	Hex	0.00041	0.187	.807	0.00022	0.0146
	Aq	0.00014	0.23	1.165	0.000054	0.0124
17	Hex	----	----	----	----	----
	Aq	0.00007	0.23	1.164	0.000027	0.0124
18	Hex	0.00003	0.186	.809	0.00001	0.0145
	Aq	----	----	----	----	----
19	Hex	0.00003	0.191	.809	0.00001	0.0149
	Aq	0.00002	0.24	1.164	0.000008	0.0130
1AX	Hex	----	0.20	.808	----	0.0156

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TABLE III

Decontamination in Batch Countercurrent Runs

<u>Run No.</u>	BCE-9	BCE-10
<u>Log dF-final</u>		
gross β, γ *	2.8	3.0
Ru	1.3	1.9
Zr	2.8	3.7
Ce	4.8	

*Measured through 5.00 mg/cm² Al absorber

TABLE IV

Plutonium and Uranium Losses in Batch Countercurrent Runs

<u>Run No.</u>	BCE-8	BCE-9	BCE-10
<u>Loss in LAW</u>			
Plutonium	0.17%	0.04%	0.002%
Uranium	0.03%	0.03%	0.002%

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TABLE V

Run No. BCE-8

Operating Conditions:

Batch simulated countercurrent

Stages: 3 in scrub section

9 in extraction section

IAP introduced at stage 4, IAS at stage 1, and IAX at stage 12.

Feed ratios: IAX:IAS:IAP::4:1:1.

Stirring: 2 minutes at each contacting

Analysis of Feeds:

IAS - 1.63 m/l $\text{Al}(\text{NO}_3)_3$, 0.41 m/l NaNO_3 , minus 0.52 m/l HNO_3

Sp. Gr. 1.259 at 25°C., 0.0095 m/l $\text{Cr}_2\text{O}_7^{=}$

IAP - 2.00 m/l UNH, 0.13 m/l HNO_3 , 0.098 M $\text{Cr}_2\text{O}_7^{=}$

Sp. Gr. 1.664 at 25°C., 6×10^6 c/m/ml Pu, no fission products

IAX - 0.202 m/l HNO_3 , Sp. Gr. 0.803 at 25°C.

<u>Stage No.</u>	<u>Phase</u>	<u>UNT</u> <u>M/L</u>	<u>Pu</u> <u>c/m/ml</u>	<u>HNO_3</u> <u>M/L</u>	<u>Sp. Gr.</u> <u>at 25°C</u>
1	Hex	0.535	1.45×10^6	0.005	0.985
	Aq	.188	6.20×10^5	-0.445	1.296
2	Hex	.556	1.47×10^6	0.013	.994
	Aq	.172	7.10×10^5	-0.033	1.308
3	Hex	.556	1.40×10^6	0.110	.999
	Aq	.127	5.10×10^5	0.020	1.294
4	Hex	.556	1.35×10^6	.113	1.001
	Aq	.640	2.23×10^6	.117	1.382
5	Hex	.263	1.04×10^6	.130	.902
	Aq	.310	1.02×10^6	.143	1.269
6	Hex	.148	6.32×10^5	.141	.863
	Aq	.167	5.13×10^5	.186	1.226
7	Hex	.071	3.12×10^5	.207	.835
	Aq	.093	2.38×10^5	.237	1.203
8	Hex	.036	1.30×10^5	.183	.823
	Aq	.038	7.70×10^4	.268	1.186
9	Hex	.016	5.00×10^4	.188	.816
	Aq	.0136	2.30×10^4	.270	1.186
10	Hex	.0058	1.55×10^4	.188	.812
	Aq	.0046	8.20×10^3	.270	1.186
11	Hex	.0023	4.70×10^3	.190	.811
	Aq	.0024	2.7×10^3	.327	1.186
12	Hex	.00064	1.80×10^3	.194	.810
	Aq	.00042	5.49×10^3	.321	1.184

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TABLE VI

Run No. BCE-9

Operating Conditions:

Batch simulated countercurrent

Stages: 5 in scrub section

9 in extraction section

IAF introduced at stage 6, IAS at stage 1, and IAX at stage 14.

Feed ratios: IAX:IAS:IAF::4:1.06:1.06

Stirring: 2 minutes at each contacting

Analysis of Feeds:

IAS - 1.62 m/l $Al(NO_3)_3$, 0.41 m/l $NaNO_3$, minus 0.495 m/l HNO_3

Sp. Gr. 1.256 at 25°C., 0.0095 m/l $Cr_2O_7^{2-}$

IAF - 1.94 m/l UNH, 0.136 m/l HNO_3 , 0.09 m/l $Cr_2O_7^{2-}$

Sp. Gr. 1.640 at 25°C., 3.53×10^6 c/m/ml Pu

2.1×10^7 c/m/ml Ru, 1.45×10^8 c/m/ml Ce, 1.50×10^6 c/m/ml Zr

IAX - 0.21 m/l HNO_3 , Sp. Gr. 0.808 at 25°C.

<u>Stage No.</u>	<u>Phase</u>	<u>UNT</u> <u>M/L</u>	<u>Pu</u> <u>c/m/ml</u>	<u>HNO₃</u> <u>M/L</u>	<u>Ru</u> <u>c/m/ml</u>
1	Hex	0.436	9.76×10^5	0.02	2.53×10^5
	Aq	.173	3.4×10^5	-0.445	1.16×10^6
2	Hex	.440	1.69×10^6	0.01	1.55×10^6
	Aq	.185	4.5×10^5	-0.432	8.23×10^6
3	Hex	.432	1.27×10^6	0.088	3.05×10^6
	Aq	.162	3.6×10^5	-0.294	5.64×10^6
4	Hex	.432	1.25×10^6	0.099	2.28×10^6
	Aq	.097	2.31×10^5	-0.010	6.46×10^6
5	Hex	.440	1.28×10^6	0.112	2.27×10^6
	Aq	.121	2.63×10^5	0.021	7.17×10^6
6	Hex	.440	1.21×10^6	0.156	1.74×10^6
	Aq	.513	1.18×10^6	0.071	1.44×10^7
7	Hex	.203	5.68×10^5	0.140	1.59×10^6
	Aq	.330	7.69×10^5	0.189	
8	Hex	.148	2.77×10^5	0.154	1.25×10^6
	Aq	.150	5.38×10^5	0.156	
9	Hex	.072	1.42×10^5	0.189	1.3×10^6
	Aq	.065	9.16×10^4	0.222	
10	Hex	.0325	4.85×10^4	0.188	1.2×10^6
	Aq	.0275	2.58×10^4	0.254	
11	Hex	.011	1.25×10^4	0.190	1.07×10^6
	Aq	.0055	1.75×10^3	0.280	
12	Hex	.0019	2.5×10^3	0.189	8.83×10^5
	Aq	.0010	767	0.273	
13	Hex	.00033	445	0.197	6.32×10^5
	Aq	.00020	633	0.268	
14	Hex	.00008	149	0.191	4.16×10^5
	Aq	.00005	807	0.242	

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TABLE VII

Run No. BCE-10

Operating Conditions:

Batch simulated countercurrent

Stages: 5 in scrub section

11 in extraction section

IAF introduced at stage 6, IAS at stage 1, and IAX at stage 14.

Feed ratios: IAX:IAS:IAF::4:1:1

Stirring: 2 minutes at each contacting

Analysis of Feeds:

IAS - 1.63 m/l $\text{Al}(\text{NO}_3)_3$, 0.41 m/l NaNO_3 , minus 0.47 m/l HNO_3

Sp. Gr. 1.259 at 25°C., 0.01 m/l $\text{Cr}_2\text{O}_7^{=}$

IAF - 1.97 m/l UNT, 0.116 m/l HNO_3 , 0.09 m/l $\text{Cr}_2\text{O}_7^{=}$

Sp. Gr. 1.653 at 25°C., 3.53×10^6 c/m/ml Pu

2.1×10^7 c/m/ml Ru, 1.45×10^8 c/m/ml Ce, 1.50×10^6 c/m/ml Zr

IAX - 0.196 m/l HNO_3 , Sp. Gr. 0.808 at 25°C.

Stage No.	Phase	UNT M/L	Pu c/m/ml	HNO_3 M/L	Ru c/m/ml
1	Hex	0.400	8.72×10^5	0.002	5.55×10^4
	Aq	0.172	3.58×10^5	-0.448	
2	Hex	0.600	1.03×10^6	0.007	1.17×10^5
	Aq	0.210	4.81×10^5	-0.454	
3	Hex	0.556	1.00×10^6	-0.002	2.16×10^5
	Aq	0.182	4.80×10^5	-0.421	
4	Hex	0.582	1.05×10^6	0.000	7.10×10^5
	Aq	0.213	5.49×10^5	-0.316	
5	Hex	0.540	1.04×10^6	0.101	2.92×10^6
	Aq	0.109	3.21×10^5	-0.008	
6	Hex	0.524	9.70×10^5	0.101	2.25×10^6
	Aq	0.584	1.29×10^6	0.118	
7	Hex	0.224	5.28×10^5	0.119	2.00×10^6
	Aq	0.268	7.28×10^5	0.198	
8	Hex	0.125	3.17×10^5	0.139	1.95×10^6
	Aq	0.174	4.75×10^5	0.130	
9	Hex	0.079	1.92×10^5	0.148	
	Aq	0.093	3.04×10^5	0.180	
10	Hex	0.0360	8.76×10^4	0.183	
	Aq	0.048	8.06×10^4	0.237	
11	Hex	0.0123	2.85×10^4	0.180	
	Aq	0.0145	1.71×10^4	0.221	
12	Hex	0.0044	7,430	0.191	
	Aq	0.0060	6,360	0.213	
13	Hex	0.0015	1,700	0.189	
	Aq	0.0021	813	0.259	
14	Hex	0.00093	385	0.190	
	Aq	0.0014	727	0.242	
15	Hex	0.00053	80	0.190	
	Aq	0.0011	640	0.230	
16	Hex		65	0.191	
	Aq	0.00004	607	0.263	

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APPENDIX IV - FIGURES

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FIGURE-1

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FIGURE 2

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WATER-ILU' EQUILIBRIUM AND OPERATING LINE DATA
R.DOX BANK 1A

RUN NO. G1-12

DATE 11/49

PILOT BASIS: A, H, = Flowrates, grams non-UNT/min.
Y, Y, = Concentrations, grams UNT/gram non-UNT
A, X, refer to Aqueous. H, Y, refer to Organic.

PROCES. STRAITS:

	U, m/l	HNO ₃ , m/l	Al, m/l	Cr ₂ O ₇ , m/l	Avg. Flow, cc/min.
LAF	2.02	0.106	----	----	19.5
LAS	----	-0.17	1.66	----	10.5
LAX	----	0.213	----	----	79.2
LA7	5x10 ⁻⁶	0.26	0.94	----	35.4
LAP	0.46	0.90	----	----	24.4

COMMENTS:

Eleven extraction stages

FIGURE 5

1076957

HNO₃ EQUILIBRIUM AND OPERATING LINE DATA
REDOX BANK 1A

RUN NO. 01-12

DATE 11/49

PLOT BASIS: A, H, = Flowrates, grams non-UNIT/min.
X, Y, = Concentrations, grams HNO₃/gram non-UNIT
A, X, refer to Aqueous, H, Y, refer to Organic.

PROCESS STREAMS:

	U, m/l	HNO ₃ , m/l	Al, m/l	Cr ₂ O ₇ , m/l	Avg. Flow, cc/min.
1AF	2.02	0.106	----	----	19.5
1AS	----	-0.47	1.66	----	19.5
1AX	----	0.213	----	----	79.2
1AW	5x10 ⁻⁶	0.26	0.94	----	35.4
1AP	0.46	0.00	----	----	84.4

COMMENTS:

Eleven extraction stages
Stages 2 thru 8 acid-deficient. Stages 9 thru 19 acid.

1076958

FIGURE 7

EXTRACTOR EQUILIBRIUM AND OPERATING LINE DATA
RUDY BANK 1A

RUN NO. G1-20

DATE 2/28/50

PLOT BASIS: u, H , = Flowrates, grams non-UNT/min.
 X, Y , = Concentrations, grams UNT/gram non-UNT
 A, Y , refer to Aqueous, H, Y , refer to Organic.

PROCESS STREAMS:

	U, m/l	HNO ₃ , m/l	Al, m/l	Cr ₂ O ₇ , m/l	Avg. Flow, cc/min.
L.F	1.99	0.12	----	----	19.6
L.S	----	-0.51	1.66	----	20.3
L.X	----	0.20	----	----	79.5
L.V	2×10^{-5}	0.24	0.94	----	35.1
L.F	0.46	0.00	----	----	83.5

COMMENTS:
Eleven extraction stages

1076955

FIGURE 8

HNO₃ EQUILIBRIUM AND OPERATING LINE DATA
REDUX BANK 1A

RUN NO. 01-20

DATE 2/28/50

PLOT BASIS: A, M, = Flowrates, gram non-UNT/min.
X, Y, = Concentrations, gram HNO₃/gram non-UNT
A, Y, refer to Aqueous. H, Y, refer to Organic.

PROCESS STREAMS:

	U, m/l	HNO ₃ , m/l	Al, m/l	Cr ₂ O ₇ , m/l	Avg. Flow, cc/min.
IAP	1.09	0.12	----	----	19.6
IAS	----	-0.51	1.66	----	20.3
IAX	----	0.20	----	----	79.5
IAW	7x10 ⁻⁵	0.24	0.94	----	35.1
IAP	0.46	0.00	----	----	81.5

COMMENTS:

Eleven extraction stages
Stages 1 thru 9 acid-deficient. Stages 10 thru 19 acid.

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1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119	120	121	122	123	124	125	126	127	128	129	130	131	132	133	134	135	136	137	138	139	140	141	142	143	144	145	146	147	148	149	150	151	152	153	154	155	156	157	158	159	160	161	162	163	164	165	166	167	168	169	170	171	172	173	174	175	176	177	178	179	180	181	182	183	184	185	186	187	188	189	190	191	192	193	194	195	196	197	198	199	200	201	202	203	204	205	206	207	208	209	210	211	212	213	214	215	216	217	218	219	220	221	222	223	224	225	226	227	228	229	230	231	232	233	234	235	236	237	238	239	240	241	242	243	244	245	246	247	248	249	250	251	252	253	254	255	256	257	258	259	260	261	262	263	264	265	266	267	268	269	270	271	272	273	274	275	276	277	278	279	280	281	282	283	284	285	286	287	288	289	290	291	292	293	294	295	296	297	298	299	300	301	302	303	304	305	306	307	308	309	310	311	312	313	314	315	316	317	318	319	320	321	322	323	324	325	326	327	328	329	330	331	332	333	334	335	336	337	338	339	340	341	342	343	344	345	346	347	348	349	350	351	352	353	354	355	356	357	358	359	360	361	362	363	364	365	366	367	368	369	370	371	372	373	374	375	376	377	378	379	380	381	382	383	384	385	386	387	388	389	390	391	392	393	394	395	396	397	398	399	400	401	402	403	404	405	406	407	408	409	410	411	412	413	414	415	416	417	418	419	420	421	422	423	424	425	426	427	428	429	430	431	432	433	434	435	436	437	438	439	440	441	442	443	444	445	446	447	448	449	450	451	452	453	454	455	456	457	458	459	460	461	462	463	464	465	466	467	468	469	470	471	472	473	474	475	476	477	478	479	480	481	482	483	484	485	486	487	488	489	490	491	492	493	494	495	496	497	498	499	500	501	502	503	504	505	506	507	508	509	510	511	512	513	514	515	516	517	518	519	520	521	522	523	524	5
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REDOX BANK 1A

NON-UNT BASIS

DATE 1-19-50

U, m/l HNO₃, m/l Al, m/l Cr₂O₇, m/l Rel.Flow

IAF	2.00	0.13	----	0.098	1
IAS	----	-0.52	1.63	0.01	1
IAX	----	0.202	----	----	4

IAF - 6×10^6 c/m, ml. Pu
IAW - Plutonium loss-0.15%. Uranium loss-0.04%.
Nine extraction stages. Three scrub stages.
Stages 1 thru 2 acid deficient. Stages 3 thru 12 acid.

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FIGURE 96

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URANIUM EQUILIBRIUM AND OPERATING LINE DATA

REDOX BANK 1A

RUN BCE-8

DATE 1-19-50

NON-UNT BASIS

PROCESS STREAMS:

	U, m/l	HNO ₃ , m/l	Al, m/l	Cr ₂ O ₇ , m/l	Rel. Flow
IAF	2.00	0.13	----	0.098	1
IAS	----	-0.52	1.63	0.01	1
IAX	----	0.202	----	----	4

COMMENTS:

IAF - 6×10^6 c/m, ml. Pu

IAW - Plutonium loss-0.15%. Uranium loss-0.04%.

Nine extraction stages. Three scrub stages.

Stages 1 thru 2 acid deficient. Stages 3 thru 12 acid.

EQUILIBRIUM LINE

EXTRACTION OPERATING LINE

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FIGURE 90

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PLUTONIUM EQUILIBRIUM AND OPERATING LINE DATA

REDOX BANK 1ARUN BCE-8DATE 1-19-50

NON-UNT BASIS

PROCESS STREAMS:

	U, m/l	HNO ₃ , m/l	Al, m/l	Cr ₂ O ₇ , m/l	Rel. Flow
1AF	2.00	0.13	----	0.098	1
1AS	----	-0.52	1.63	0.01	1
1AX	----	0.202	----	----	4

COMMENTS:

1AF - 6×10^6 c/m, ml. Pu
 1AW - Plutonium loss-0.15%. Uranium loss-0.04%.
 Nine extraction stages. Three scrub stages.
 Stages 1 thru 2 acid deficient. Stages 3 thru 12 acid.

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FIGURE 10B

Y GM UNT
GM NON-UNT

4 5 3/2

EQUILIBRIUM LINE

EXTRACTION OPERATING
LINE

X GM UNT
GM NON-UNT

URANIUM EQUILIBRIUM AND OPERATING LINE DATA

REDOX BANK 1A

NON-UNT BASIS

DATE 2-3-50

RUN BCE-9

PROCESS STREAMS:

	U, m/l	HNO ₃ , m/l	Al, m/l	Cr ₂ O ₇ , m/l	Rel. Flow
1AF	1.94	0.136	----	0.09	1.06
1AS	----	-0.47	1.62	0.01	1.06
1AX	----	0.21	----	----	4.00

COMMENTS: 1AF -

1AW - Plutonium loss-.04%. Uranium loss-.004%.

Gross Δ YdF - 1000. Nine extraction stages.

Stages 1 thru 4 acid deficient. Stages 5 thru 14 acid.

ENLARGED LEAN END

1076947

FIGURE 106

Y C/M Pu
GM NON-UNT

PLUTONIUM EQUILIBRIUM AND OPERATING LINE DATA

REDOX BANK 1A

RUN BCE-9

NON-UNT BASIS

DATE 2-3-50

PROCESS STREAMS:

	U, m/l	HNO ₃ , m/l	Al, m/l	Cr ₂ O ₇ , m/l	Rel. Flow
IAF	1.94	0.136	----	0.09	1.06
IAS	----	-0.47	1.62	0.01	1.06
IAX	----	0.21	----	----	4.00

COMMENTS:

IAF -

IAW - Plutonium loss-.04%. Uranium loss-.004%.

Gross ϕ, γ df - 1000. Nine extraction stages.

Stages 1 thru 4 acid deficient. Stages 5 thru 14 acid.

2000

ENLARGED
LEAN END

2000

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FIGURE 11A

HNO₃ EQUILIBRIUM AND OPERATING LINE DATA
REDOX BANK 1A

RUN BCE-10

DATE 2-8-50

NON-UNT BASIS

PROCESS STREAMS:

	U, m/l	HNO ₃ , m/l	Al, m/l	Cr ₂ O ₇ , m/l	Rel. Flow
IAF	1.97	0.116	----	0.09	1
IAS	----	-0.47	1.63	0.01	1
IAX	----	0.196	----	----	4

COMMENTS:

IAF -

IAW - Plutonium loss-.005%. Uranium loss-.006%

Gross β, γ dF - 1000. Eleven extraction stages.

Stages 1 thru 5 acid deficient

Stages 6 thru 16 acid

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FIGURE 11B

URANIUM EQUILIBRIUM AND OPERATING LINE DATA

REDOX BANK 1A

RUN BCE-10

DATE 2-8-50

NON-UNT BASIS

PROCESS STREAMS:

	U, m/l	HNO ₃ , m/l	Al, m/l	Cr ₂ O ₇ , m/l	Rel. Flow
1AF	1.97	0.116	----	0.09	1
1AS	----	-0.47	1.63	0.01	1
1AX	----	0.196	----	----	4

COMMENTS:

1AF -
 1AW - Plutonium loss-.005%. Uranium loss-.006%
 Gross δ df - 1000. Eleven extraction stages.
 Stages 1 thru 5 acid deficient
 Stages 6 thru 16 acid

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FIGURE 11C

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PLUTONIUM EQUILIBRIUM AND OPERATING LINE DATA
REDOX BANK 1A

RUN BCE-10

DATE 2-8-54

NON-UNT BASIS

PROCESS STREAMS:

U, m/l HNO_3 , m/l Al, m/l Cr_2O_7 , m/l Rel. Flow

1AF	1.97	0.116	----	0.09	1
1AS	----	-0.47	1.63	0.01	1
1AX	----	0.196	----	----	4

COMMENTS:

1AF -

1AW - Plutonium loss-.005%. Uranium loss-.006%

Gross β , δ dF - 1000. Eleven extraction stages.

Stages 1 thru 5 acid deficient

Stages 6 thru 16 acid

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