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AEC RESEARCH AND DEVELOPMENT REPORT

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PROGRESS REPORT

CHEMICAL DEVELOPMENT

SEPARATIONS TECHNOLOGY

DECEMBER 31, 1952.

To: File

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PROGRESS REPORT - CHEMICAL DEVELOPMENT
DECEMBER, 1952

REDOX PLANT ASSISTANCE

Plant Performance

The Redox Plant operated at a 68.8 per cent time efficiency (IAF basis) and averaged 4.26 tons of uranium per operating day during the month. The following is an over-all summary of plant production performance:

	<u>Approximate</u>
Tons uranium shipped	91.4
Plutonium processed (batch equivalents)	129.6
Per cent uranium to waste	1.2
Per cent plutonium to waste	1.2

Operating Performance

Redox operations continued at a 4.25-ton/day rate (IA Column operating at a 3.0-ton/day rate in parallel with IS Column at a 1.25-ton/day rate) until December 2. At this time a complete shutdown was effected so that the following major items were accomplished, the first three of these being aimed toward increasing the over all capacity of the plant:

- (1) The IB Column (packed with 1/2-inch Raschig rings) was replaced with the spare column (packed with 3/4-inch Raschig rings and without a mid-column feed cone).
- (2) The 2D Column (packed with 3/4-inch Raschig rings) was replaced with a spare column (packed with 1-inch Raschig rings).
- (3) The G-2 Organic Still Condenser, D-11 Waste Concentrator Condenser, and D-3 Condensate Evaporator Condenser were replaced with spare condensers with revised inlets which have reduced the pressure drop at the inlets by approximately 8, 8, and 18 inches of H₂O, respectively.
- (4) The H-4 (Oxidizer) pot was replaced (due to a coil leak) with the spare pot. The old pot was placed on the G-5 (Centrifuge Feed Tank) pad for potential later use.
- (5) IA, IS, 3D, 2A, and 3A Columns were given a hot 60% HNO₃ soak for 24 hours with additional water flushing.
- (6) Plutonium concentrators E-16 and E-17 were cleaned by HNO₃ boil-up.
- (7) H-1, F-7, and F-8 (IAF Tanks) were given 5% HNO₃ and water flushes.

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On December 10, production was reestablished with the IA Column operating at a 3.0-ton/day rate. The IA Column rate was increased to 4.0 tons/day on December 12 and continued at this level until the IS Column was started in parallel with the IA Column on December 13. The combined IA and IS Column production rate was 4.75 tons/day from December 14 to 16, during which time the Second Uranium Cycle was by-passed on a test basis. At the completion of the test, the production rate was reduced to 4.25 tons/day and maintained at this level until December 24 when the plant was shut down for the Christmas holiday.

Activity released to the atmosphere through the ventilation stack averaged less than 1 curie of ^{131}I and 0.01 curie of Pu per day. Fission-product activity in the swamp area has not increased since the replacement of the H-4 (Oxidizer) pot.

Process Performance

The following tables summarize decontamination and waste losses by solvent extraction cycle for representative periods in December:

12/14/52 through 12/17/52; nominal production rate of 4.25 tons/day (IA and IS Columns in parallel with 1.8 M ANN in IAS), processing 95-day "cooled" metal:

<u>Cycle</u>	<u>Gamma dF's</u>		<u>% to Waste</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
1st	4.0	3.7	1.1	1.2
2nd U	1.8	0.0	0.0	0.2
3rd U	0.7	0.0	0.0	0.0
2nd Pu	0.0	2.3	0.0	0.01
3rd Pu	0.0	0.9	0.0	0.01
Total	6.5	6.9	1.5	1.4

12/19/52 through 12/24/52; nominal production rate of 4.25 tons/day (IA and IS Columns in parallel with 2.0 M ANN in IAS), processing 102-day "cooled" metal:

<u>Cycle</u>	<u>Gamma dF's</u>		<u>% to Waste</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
1st	3.6	3.5	0.2	0.5
2nd U	2.0	0.0	0.0	0.1
3rd U	0.8	0.0	0.0	0.0
2nd Pu	0.0	2.1	0.0	0.01
3rd Pu	0.0	0.9	0.0	0.01
Total	6.4	6.5	0.8	0.6

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Feed Preparation

The dissolvers were charged during the month with seventeen 4.95-ton charges of uranium, one 3.85-ton charge, one 4.4-ton charge, and one 6.05-ton charge, having an average pile exposure of 597 MWD/T. The three off-standard size charges were required for special dissolving tests which proved the feasibility of taking only two instead of three "cuts" per 4.95-ton charge. Details of the tests are being reported separately (in Document HW-26674).

The average age of twenty-five 3.5-ton IAF batches prepared was 97 days. The first ten of these batches were oxidized with a 0.15 M $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$. The feed preparation procedure revisions described last month are still being followed.

Uranium Extraction and Decontamination

In general, nominal conditions of the ORNL June, 1949 (acid-deficient) Flow-sheet (Document HW-22834) were employed for the First Extraction Cycle and the Second Uranium Cycle. These conditions as currently employed are summarized below:

IAF:IAS:IAX:IBX:IBS:ICX volume flow ratios = 100:110:420:40:200:180

- (1) with IAS composition of 2.0 M ANN, 0.2 M HNO_3 -deficient, Cr^{+6} ,
- (2) 100% flowsheet IBX and IBS ratios used for new IB Column until reduced flow ratios can be reestablished and proven, and
- (3) 0.075 M HNO_3 in IBS.

2DF:2DS:2DX:2EX volume flow ratios = 100:100:400:140

with 2DS composition of 2.0 M ANN, 0.2 M HNO_3 -deficient, no Fe^{+2} .

The 3D Column has been operated as a dual-scrub column with the 3DS introduced at the 3DF inlet tee and 3DA introduced at the top of the column. The Third Uranium Cycle flowsheet is summarized below:

3DF:3DS:3DA:3DX:3EX = 100:120:50:500:140

- (1) with 3DS composition of 2.2 M ANN, neutral, no Fe^{+2} , and
- (2) 3DA of 0.04 M HNO_3 .

During the period from December 10 to December 17, the First Extraction Cycle flowsheet was modified to the following conditions on a test basis:

IAF:IAS:IAX:IBX:IBS:ICX = 100:124:450:40:200:200

with IAS composition of 1.8 M ANN, 0.2 M HNO_3 -deficient, Cr^{+6} .

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Performance with this modified flowsheet appears to have improved the First Cycle U dF by a factor of almost 2 and the overall Pu dF by a similar factor. However, the test has not yet been repeated, and therefore, no conclusions have been made.

The Second Uranium Cycle was effectively by-passed from December 14 to December 16 by pumping through the 2D-2E Columns from the 2DF to the 3DF Tank. Thus, the 1A Column flowsheet, using 1.8 M ANN in IAS, was used in series with the 3D Dual-Scrub Column flowsheet, using neutral 3DS. Operation prior to and following the test was at a 4.25 ton/day rate, and during the test the rate was 4.75 tons/day. Based on the analytical data obtained during the test, it was concluded that the decontamination factor across the Dual-Scrub 3D Column is slightly higher than that across the 2D Column. The following uranium product batches were out of specifications and required special handling:

<u>Batch Nos.</u>	<u>Gamma ratio*</u>	<u>Disposition</u>
11 and 13	10.6 and 8.0, respectively	Being stored in Tanks SU-151 and 152 (with November Batches 9 and 10) for additional "cooling".
12	8.6	Being reworked because the plutonium content was 2.6 times the permissible maximum.

*) Ratio of gamma radioactivity to that of natural uranium.

Performance of the spare 1B Column (packed with 3/4-inch Raschig rings) is currently being evaluated. During operation at a 4.75-ton/day rate, the plutonium content of 1BU (with flowsheet 1BX) was 0.3% of Pu in IAF, and at a 4.25 ton/day rate, the loss was approximately 0.1%. Performance at the end of the month shows a trend toward improvement.

Plutonium Extraction and Decontamination

Nominal conditions (except flow ratios) of the HW#4 Flowsheet were maintained for the Plutonium Cycle operation until December 18. At this time the flowsheet was changed so that a new HNO₃ specification (less than 3.0 lb. HNO₃/gallon) in Redox PR solutions could be met. The flowsheet currently employed is described below:

Solution Specifications:

2BX and 3BX - Changed from 0.1 M HNO₃ to 0.04 M HNO₃.

2AF, 2AS, 2AX, 3AF, 3AS, 3AX - Same as HW#4 Flowsheet.

2A and 3A Column Flow Rates:

<u>Feed Rate, Gal./Min.</u>	<u>Scrub Rate, Gal./Min.</u>	<u>Extractant Rate</u>
less than 0.42	twice feed rate	(feed + scrub rates)/1.5
greater than 0.42	0.84	(feed + scrub rates)/1.5

The 2AS and 3AS rates are still maintained at the "double-scrub" ratios in order to minimize emulsification.

2B and 3B Column Flow Rates:

$$\text{Aqueous flow} = (\text{Organic flow})/5$$

However, the minimum aqueous rate is set by critical mass control limitations which determine the maximum plutonium concentrations in the 2BP and 3BP streams. 2BP Butt rate = 1.5 (2BX flow rate)

The uranium content of the plutonium product has averaged approximately 0.001 units U per unit Pu. No plutonium rework has been required through Redox or 224-T Plant (except 231 Building recycle). The PR Batch shipments from Redox to 231 Bldg. were increased from 375 to 400 units (maximum) on December 10.

Solvent Treatment

The plant solvent recovery system, IO Column and Organic Still (G-3), operated satisfactorily with water as a scrub solution in both units. Caustic washing of three consecutive batches on a bi-weekly basis has continued, and the hexone quality has been maintained at a high level. Solvent losses for the period have averaged approximately 1.3 per cent of throughput.

Rotating Equipment

Two agitators, A-D-8 in the Waste Neutralizer and AH-1 in the IAF Make-Up Tank, failed when the shafts sheared off at the coupling between the stub shaft and the agitator shaft. Both of these shafts failed at the relief groove cut into the shaft at the shoulder which seats on the coupling. These agitators, as well as A-D-9 which failed in the same manner, are being repaired. The new shafts will not have a relief groove at the shoulder.

A Peerless 8 inch LA deepwell turbine pump with a 14 ft. 6 in. drive shaft has been installed in D-8, the Waste Neutralizer, to replace the D-8 to 241 transfer jet. Use of this pump will eliminate an estimated 12 per cent dilution which results from operation of the transfer jet.

Instrumentation

Rotameters and parts for Phase II of the plant capacity increase project have been shipped by the manufacturer. "Cold" service Hammel-Dahl parts needed for Phase II operation have been received. Shipment of the "hot" service valves has been delayed indefinitely because the vendor does not have an approved design for the auxiliary stem seal.

Process Chemistry

Head-end treatment. "Cold" runs in the Moxie (miniature oxidizer) were continued during the month. Little (less than 1%) decomposition of KMnO_4 occurred at or below 30°C ., in either H_2O or UNH solutions, but as the temperature was

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increased to above 90°C., decomposition was noticeable at once. The rate of decomposition was greater in the UNH solutions and also when the oxidizer pot contained MnO₂ from the previous run, but in any case was stopped rapidly and almost completely when the temperature was reduced to 30°C. The observed rates of KMnO₄ reduction at 92°C., were approximately 10% (of the initial concentration) per hour when the pot contained MnO₂. This work is continuing and will be extended to include study of the effect of radiation.

The slug rinsing experiments (following Al jacket removal) are nearing completion. The amount of residual solids was approximately the same (0.15 vol.%) in each of the three rinse solutions, H₂O, 5% aqueous NaOH, and 5% aqueous HNO₃, but were brown and granular in the case of the first two, and white and gelatinous in the third. A similar white, gelatinous solid was found in the first "cut" when each of the slugs was dissolved. The IAF solutions produced from these slugs are being evaluated by means of the vibrational disengaging time test, with the lowest times so far resulting from the use of HNO₃-washed slugs. The significance of these disengaging time data with respect to column operation however, is not yet known.

Studies of nitric acid dissolving of slug jackets have been started. The use of Hg as a catalyst and 45% HNO₃ for the initial reaction will permit jacket removal in 6 hours or less, producing at the same time a granular form of residual solids (rather than the gelatinous) which should be more easily removed in the centrifuging step. The optimum conditions, viz., concentration of HNO₃, Hg, etc., have yet to be determined, as well as the path, the effect, and the chemical state of the Hg throughout the system.

Solvent Extraction. The effects of age and temperature on fission-product distribution between phases are being determined by contacting IAW samples (held at 65°C. and 100°C., respectively) with aliquots of new hexone for extended lengths of time and periodically determining the beta content of the hexone. The IAW contained 7.4×10^6 beta counts/(min.)/(ml.) initially. For the sample held at 65°C., the hexone-phase beta count has risen from an initial value of 6.6×10^2 c/m/ml. to 1.6×10^3 c/m/ml. after 37 days, whereas in the same period, the 100°C. sample has shown an increase in beta distribution into the organic phase from 6.6×10^2 c/m/ml. to 2.3×10^4 c/m/ml.

In continuance of the search for causes of "emulsions" in the Redox columns, the vibrational disengaging and dispersion time technique was applied to the following systems:

2DS vs. 2DX
2DW vs. 2DX
2DFS vs. 2DX
2DFS vs. 2DU
2DS vs. 2DU

in which the 2DX was (a) hexone from Tank O-2, (b) hexone from Tank O-2 plus 0.5 M HNO₃, and (c) hexone from Tank O-2 plus 1 g./l. non-volatile oily residue (from evaporation of hexone). The following conclusions can be drawn:

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1. In the 2DS-2DX system, the disengaging time increases by a factor of approximately 8 as the 2DS acidity is decreased from + 0.1 M HNO_3 to - 1.0 M HNO_3 .
2. The presence of 1 g./l. non-volatile residue approximately doubles the disengaging time.
3. Where the aqueous acidity is positive or at least 0.5 M deficient, the addition of acid to the 2DX increases the disengaging time. At intermediate levels of acid-deficiency in the aqueous, the reverse appears to be true.
4. Colloidal silicic acid increases the disengaging time. However, increasing the acidity (in the range from 0.5 M acid-deficient to 0.1 M HNO_3) minimizes or eliminates this effect of silicic acid.
5. The addition of acid to the 2DX increases the 2DW-2DX dispersion time by factors of from 5 to 10. This could conceivably lower the extraction efficiency at the bottom of the column.
6. In general, both the disengaging and the dispersion times for the systems 2DFS vs. 2DX or 2DFS vs. 2DU are lower than in the systems containing no uranium, since the uranium has a counter-effect to the agents which increase the disengaging time.

The data indicate that pronounced emulsion tendency may be produced in the lower extraction section of the 2D column as a result of the presence of colloidal silicic acid in the aqueous influent either alone or in combination with acid-deficiency in the aqueous phase and contamination of the hexone with non-volatile residue. These factors would not cause emulsion tendency to the same extent, however, at or near the feed plate where the uranium concentration is high. The energy required to produce a given dispersion is also at a minimum at and immediately below the feed plate, rising abruptly when the uranium has transferred to the organic phase.

URANIUM RECOVERY DEVELOPMENT

Process Chemistry

Solvent Extraction. An extensive series of batch tests (still continuing) was carried out in an attempt to improve decontamination of the RCU from what was reported to be primarily Ce^{144} . These tests are recorded in Tables I and II. During this period, however, it was discovered that the principal fission product appearing in the RCU was not Ce^{144} but Y^{90} , 60-hour half-life daughter of 25-year Sr^{90} . Four Plant RAF samples and one laboratory-prepared RCU sample were submitted for specific F.P. analysis. The results are presented in Table III. Conclusions which may be drawn from the results of these tests are:

1. The inadequate decontamination obtained was primarily the result of insufficiently effective scrubbing. The data imply that the plant is obtaining considerably less than the one theoretical scrub stage which is required. (Cf. tests 10 and 12, Table II).
2. Scrub performance improves with an increasing degree of agitation.
3. Decontamination is highly dependent upon the per cent uranium saturation of the solvent.
4. Fluoride ion and $C_2O_4^{2-}$ have little or no effect on decontamination performance.
5. The addition of strong oxidizing agents to the RAF decreases the D.F. obtainable. This may be due to complex ion formation in the case of $Cr_2O_7^{2-}$ or an oxidation mechanism.
6. The addition of strong and moderate reducing agents to the RAF has little or no effect on decontamination.
7. The addition of inert $Ce(III)$ to the RAX, RAF, or RAS increases the D.F. by a factor of about 2.
8. Increasing the RAS flow ratio increases the D.F. obtainable.
9. Greater D.F.'s are obtained with 103-U slurry than with 101-C slurry.
10. The decay curves and analyses tentatively show that the beta activity from fission products in the Plant RCU is 75 to 90% due to Y^{90} (yttrium).

Several Mini (miniature mixer-settler) runs were made using 101-U, 103-U, and 101-C slurry feeds with synthetic and plant RAX containing 12.5% and 30% TEP. Cerium dilution and $(NH_4)_2SiF_6$ addition were both tried. All results, however, were essentially nullified as far as beta decontamination was concerned due to the delay of from 2 hours to 15 days in sample analyses, with attendant yttrium decay. The beta contamination in the RCU ranged from 1 to 400% of natural U, with a close correlation between beta per cent and time of analysis. The Mini work is continuing in an attempt to obtain reliable operation and reproducible results which will duplicate plant performance, for use as a reference standard.

UO₂ Reactivity. Reactivity runs on plant lots yielded the following results based on the previously used Harshaw control as a standard:

<u>Lot</u>	<u>Per Cent UF₆</u>	<u>Conversion Ratio</u>
204	88.07 (79.71)	1.08 (0.901)
206	90.96, 91.49	1.10, 1.11
209	90.46, 92.38	1.11, 1.04
214	90.51, 88.15	1.10, 1.07
244	95.35, 94.26	1.16, 1.14
Carload 012	82.32	1.01
Carload 016	91.60	1.13

Fair checks were obtained on reruns except for Lot 204.

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Reactivity runs were made on a new reactivity standard (Mallinckrodt T-268) against the Harshaw control which has been in use. Conversion ratios obtained for several runs were 1.12, 1.06, 1.13, 1.14, and 1.05. On this basis the above-listed plant lots are about equivalent in reactivity to the new Mallinckrodt standard. The ratio of 1.13 is believed to be the best result, which gives a reactivity ratio of 0.88 for the Harshaw control sample formerly used relative to the new Mallinckrodt standard.

Three UO_3 samples (of Harshaw, Mallinckrodt, and Purex origins) were prepared at Oak Ridge and shipped to K.A.P.L. and Hanford for comparison of analytical results obtained at the three sites. The results, including reactivity, obtained at Hanford are compared in Table IV, with the results of Oak Ridge analyses. It is to be noted from this table that the Mallinckrodt UO_3 is relatively pure, with the possible exception of calcium, and has approximately the composition of the monohydrate, yet its reactivity is very poor (approximately 0.71 relative to the new Mallinckrodt reactivity standard, T-268). The Purex UO_3 , which is appreciably more impure, shows a reactivity about the same as the Mallinckrodt standard. The Mallinckrodt test sample had a very low surface area particularly for hydrated material. These results are additional evidence that many little-understood factors are involved in UO_3 reactivity.

SEPARATIONS PROCESS DEVELOPMENT

Process Studies

O.R.N.L. Purex Investigations. In October 1952 the decision was made to make five more "hot" Purex runs in the O.R.N.L. pilot plant, primarily to be of assistance in the design of the Hanford Works Purex plant. Four of these runs are now complete, and the fifth is scheduled to be made in early January. Two trips were made to Oak Ridge to plan the runs and to review the data obtained. These trips are reported in HW-26379 and HW-26650.

While the data from these first four runs have not yet been completely analyzed, the following tentative conclusions have been drawn. Decreasing the pulse amplitude in the IA Column from 0.94 to 0.50 inch resulted in only a small loss in decontamination efficiency (approximately a factor of 2 or less). Substituting ferrous ammonium sulfate plus sulfamic acid for ferrous sulfamate as the reductant in the IB Column caused no mal-operation of that column, but the 2AW losses were ten times normal. The IC Column was successfully run with the organic phase continuous, with little difference in process performance being noted. Reducing the height of the ID scrub section from 13 ft. to 9 ft. reduced the decontamination factor for Zr and Nb by a factor of about two. The acid recovery system has been in poor condition and is too "hot" to repair without extensive decontamination. Consequently, the data obtained have been of questionable value. Centrifugation has been demonstrated to remove the solids and entrained water from regenerated solvent so that separation in after-treatment storage no longer occurs. The niobium activity of the regenerated solvent is low, corroborating the previous indications that this activity is carried by the solids.

The following informal report, dealing with the O.R.N.L. Purex work, was issued during the month:

HW-26650 Report of Discussions at O.R.N.L.
by A. M. Platt and D. P. Granquist,
dated December 24, 1952.

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Purex Plant Design Liaison.

Written comments were issued on all Purex Engineering Flow Diagrams and Instrument Engineering Flow Diagrams. Approximately 90% of the Engineering Flow Diagrams were given Project Committee approval.

Specifications or recommendations transmitted to the Design Section included:

1. Revised specifications for the three basic Purex extraction columns (A-type, C-type, and 2A).
2. Specifications for nuclear safety considerations on 2A, 2B, and IB Scrub Columns.
3. Specifications for the IO, 2D, IT, and 2T Columns.
4. Purex design scope revision requests for (a) an improved method of 2A Feed control and (b) an alternate routing for ICU to provide stripping in the 2EU concentrator.
5. Recommendations for the deletion of the Purex sand filter from the design scope and installation of a glass-wool filter on the condenser and vessel vent systems.

Chemical Engineering Development

Uranium Recovery Solvent-Extraction Studies. During the month ninety uranium recovery process solvent-extraction pulse column studies with "cold" (unirradiated) uranium were carried out in the 321 Building pilot plant. Included in these studies were twenty-seven dual-purpose RA Column runs in a 3-in.-diameter glass pulse column with a 4-in.-diameter scrub section and four RA Column scrub section and fifty-nine RC Column runs in 3-in.-diameter glass pulse columns. The general objective of these studies was to investigate the performance characteristics of the RA and RC Columns with 30% TBP as solvent with a variety of feed compositions and flowsheet modifications of plant interest. The RC Column results will also have bearing on Purex-process HC, IC, and 2E Column development. The experimental conditions and results of each of these runs are presented in Tables V to VIII. The highlights of the new findings are as follows:

1. The RA Column operated satisfactorily with the solvent flow rate decreased by 30%, to give approximately 75% (rather than 50%) saturation of the TBP with uranium at the feed point. Capacity was unimpaired, with losses at 800 gal./hr. (sq.ft.), sum of both phases (corresponding to 5 tons U/day in a 20-in.-diameter U.R. Plant Column) as low as 0.03% (with a 12-ft.-high extraction section, 1.25-in. pulse amplitude, 50 cyc./min.)

2. There was no emulsion-induced instability in the RA Column with 30% TBP even when the RAS (scrub) HNO_3 concentration was increased to 5 M. However, the local flooding threshold and optimum frequencies were about 10 cyc./min. lower with 5 M HNO_3 in the RAS than with 2 M. The cyclic local flooding frequency with 5 M HNO_3 in the RAS at 900 gal./ (hr.)(sq.ft.), sum of flows (corresponding to 5 tons U/day in a 20-in.-diam. column) was approximately 43 cyc./min. (at 1.25-in. amplitude), compared with approximately 53 cyc./min. for 2 M HNO_3 in the RAS.
3. Operation of the 30% TBP RA Column for 24 hours with simulated supernate and sludge feeds indicated no signs of emulsion-induced instability. Losses as low as 0.01% from a 12-ft. extraction section were obtained with both types of feed. With sludge-derived feeds the RA Column operated successfully at volume velocities in the neighborhood of 1200 gal./ (hr.)(sq.ft.), sum of both phases (equivalent to 7 to 8 tons U/day in a 20-in. column). With supernate-derived feed, with 18 g. U/l. of RAF, the good-performance capacity of the RA Column exceeds 900 gal./ (hr.)(sq.ft.), sum of both phases (corresponding to 2.5 tons U/day in a 20-in.-diameter column). The exact capacity remains to be established.
4. RA Column operation with simulated supernate-derived feed indicated that 50% saturation of the TBP with uranium at the feed point resulted in too close an approach to an operating-diagram "pinch" at the concentrated end to provide assurance against long-sustained high waste losses due to slight variations in feed composition and in the feed/extractant flow ratio. Preliminary indications are that this difficulty may be overcome by increasing the solvent flow by as little as 15%, to give a 42% feed-point saturation. This is being investigated further.
5. The RA Column operated stably for over 36 hours with a split scrub stream, with 2 M HNO_3 as the terminal scrub stream. (RAIS entering half-way up the 6-ft.-high scrub section, containing 2M HNO_3 and twice flowsheet ferrous ammonium sulfate and sulfamic acid concentrations. Volume flow ratios, RAF:RAS:RAIS:RAX = 100:25:25:105.) There was less buildup of "shad-roe" emulsion at the (iron-free) interface than had been observed under similar conditions with 12.5% (rather than 30%) TBP as the extractant. It was further observed that buildup of "shad-roe" emulsion is aided by the agitation caused by excessive air flow through the interface-control dip tubes.
6. RA Column scrub section flooding determinations indicated that an RA Column with a 20-in.-diameter scrub section would not be able to process successfully more than about 3 tons U/day even with 30% TBP as the extractant.
7. There was no evidence of emulsion-induced instability in a 30% TBP RC Column.

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8. RC Column capacity and losses were relatively insensitive to variations in RCX (aqueous extractant) HNO_3 concentrations between 0 and 0.1M. Tentatively, 0.01M is thought to be the most desirable RCX HNO_3 concentration, since, on the one hand, it may result in slightly lower losses than higher RCX acidities, and, on the other hand, it is believed to render the column less susceptible to emulsification than pure water as the RCX.
9. Increase in the RA feed-point saturation of the TBP with uranium from 50% to 75% exerted no significant effect on the capacity of the RC Column, but resulted in sharply increased RC Column losses. Uranium losses with 75% saturation at optimum conditions were about 0.4%, as compared with less than 0.01% obtained with 50% RA feed-point saturation (with 12-ft.-high plate section).
10. RC Column capacity was not significantly affected by increasing RA Column scrub acidity from 2 M to 5 M. Optimum RC uranium losses increased slightly - from about 0.002% with 2 M RAS HNO_3 to about 0.01% with 5 M RAS HNO_3 (with a 12-ft.-high extraction section).
11. Operation of an RC Column with a "standard cartridge" (i.e. stainless steel plates, with 0.125-in. holes, 23% free area, 2-in. plate spacing) with the organic phase - rather than the aqueous phase - continuous resulted in an approximately 3-fold reduction in capacity.
12. With 75% saturation, an RC Column packed with 1-in. stainless-steel Raschig rings and pulsed gave losses at least as low and a capacity at least as high as an RC pulse column with a "standard" perforated-plate cartridge. A 0.3% uranium loss was obtained from a 12-ft.-high packed section at 800 gal./(hr.)(sq.ft.) sum of both phases, with a 0.6-in. pulse amplitude and a frequency of 70 cyc./min. However, unlike the perforated plates, the Raschig rings gave no significant improvement in losses upon decreasing the feed uranium concentration from 75% to 50% RA feed-point saturation.

Purex IT Column. The complete flooding capacity of the Purex IT Column (unpulsed) packed with 1-in. stainless-steel Raschig rings was determined at 1500 $\pm$ 100 gal./(hr.)(sq.ft.), sum of both phases. The equipment used was a 3-in.-diameter glass column with a 12-ft. packed height. The feed (ITF) was 30 vol. % TBP in Shell Deodorized Spray Base, batch washed with 5% Na_2CO_3 (2:1 organic:aqueous vol. ratio) after an RA-RC pulse column pass. Distilled water was used as the scrub (ITS). An aqueous:organic volume flow ratio of 1:5 was employed. The aqueous phase was continuous.

Continuous U_3 Conversion Development

Design of the pilot-plant-size model fluidized-bed and screw calciners has been completed and fabrication has been initiated.

A laboratory model turbine dust separator has been built.

Mechanical Development

Pump Development. The 5 HP G.E. & C.L. submerged motor pump has operated on life test as an in-line pump for 1560 hours. The unit is pumping simulated, non-radioactive Purex 2EU solution at 18 gal./min. at a pressure of 11 lb./sq.in. Total operation logged for this pump, both submerged and in-line, is 3138 hours.

P-1 pump, a submerged regenerative turbine pump, with a 2-ft. drive shaft has operated for 94 hours pumping water at a rate of 2.25 gal./min. against an 8 ft. discharge head. The shaft of this pump is guided by one Crown No. 2 borosilicate glass bearing lubricated with the pumped solution. After 94 hours of operation, inspection and measurements revealed only insignificant wear of the glass bearing and the journal.

A Johnston 7BS deepwell turbine pump with a 17 ft. 5 in. drive shaft has been equipped with eleven No. 7740 Pyrex glass bearings. This pump has operated smoothly for 80 hours pumping water at a rate of 150 gal./min. against a 75-ft. discharge head.

Bearing Development. A bearing machined from Corning #7280 glass was run 140 hours using 50% NaOH as a lubricant. The load was 70 lb./sq.in. for the first 40 hours and 150 lb./sq.in. for the last 100 hours. The radial clearance between the journal and the bushing increased from 0.001 in. to 0.0012 in. and the final weight of the bearing was 0.06% less than the initial weight. There were no signs of wear on the journal, but the bearing was slightly scuffed under the point of maximum load. The final surface finish under the point of load was 24 micro-inches. root-mean-square, as compared to 6 micro-inches r.m.s. initially.

Pulse Generator Development. Five Teflon bellows are operating on life test. None of these bellows has shown any indication of failure. Operating data for the bellows are tabulated below:

<u>Nominal Diameter</u>	<u>Length, Face to Face</u>	<u>Stroke Length</u>	<u>Total Cycles</u>
3 in.	4 in.	1 1/2 in.	22,916,962
3 in.	4 in.	1 1/2 in.	16,593,540
10 in.	5 in.	1 1/2 to 1 1/2 in.	2,686,380
10 in.	5 in.	1 1/2 to 1 1/2 in.	2,686,380
6 in.	4 in.	0.7 to 0.9 in.	3,575,820

A new pulse generator which replaces the stainless-steel bellows pulse generator in the Demonstration Unit has been installed. This pulse generator has a mechanical drive which operates one 3-in. diameter Teflon bellows with a 4-in. face-to-face dimension. This unit has operated a total of 348,000 cycles.

Centrifugation of Purex 100. Preliminary tests of the 12-inch Bird centrifuge modified for three-phase separation have been completed. This machine was shipped December 2 to Oak Ridge National Laboratory for "hot" tests. Results of the "cold" tests are tabulated below.

<u>Centrifugal Force at Organic Overflow, G</u>	<u>Organic Residence Time, Min.</u>	<u>Appearance of Organic Effluent</u>
600	2.25	Very cloudy
	2.75	Slightly cloudy - cleared rapidly
	3.25	Sparkling clear
1200	1.75	Very cloudy
	2.0	Slightly cloudy - 0.1 vol.% aqueous settled in 36 hours (at rest)
	2.25	Slightly cloudy - cleared rapidly
	2.75	Sparkling clear

The organic-aqueous emulsion fed to the 12-inch centrifuge was prepared from Purex ICW obtained from the Demonstration Unit. The ICW was given a 1/10 volume water wash, agitating for 10 minutes and settling over night. Then 1/20 volume of aqueous 3.5% Na_2CO_3 was added to the solvent, and the two phases were agitated a minimum of ten minutes before feeding the emulsion to the centrifuge with the agitator continuing to run in the centrifuge feed tank. A sample of the centrifuge feed still contained approximately 1.2 vol. % aqueous, as a semi-permanent emulsion, after being allowed to settle for 15 minutes.

Concentrator for the Purex Prototype Testing Facility. The concentrator for the Purex prototype column testing facility will be of the vertical-tube type. The firm design incorporates a bundle of 640 tubes, 1.0-in. O.D. x #12 BWG wall, 10 ft. long. The tube bundle is surmounted by a stripping section, 6 ft. in diameter by 18 ft. high, packed with 2 in. stoneware Raschig rings, with top entry of the concentrator feed. Shop drawings for the concentrator are being made.

Materials Testing

Protective Coatings. Andrew Brown I-204 white vinyl paint, applied to mild steel coupons, resisted 10% HNO_3 and 50% NaOH for 16 days, the duration of the test. This paint failed within 6 hours in hexone, CCl_4 , plus 30% TBP, and debase plus 12.5% TBP. Although the manufacturer claimed that this paint would resist HNO_3 in concentrations up to 80%, the sample immersed in 60% HNO_3 blistered badly after 6 hours.

Two samples of Amercoat 1574 applied to mild steel rods by the Amercoat Co. were immersed in 20% and 40% HNO_3 at room temperature. The sample immersed in 20% HNO_3 showed no change after 18 days, but the sample exposed to 40% HNO_3 turned brown after one day and was badly blistered after 3 days.

Dual-Face Plates for Pulse Columns. Stainless-steel plates, coated on one side with Kel-F NW-25, have been under test in pulsing, simulated, plutonium-and-fission-product-free Purex 2A Column solutions for 74 days. Weight loss of the coating on these plates ranges from 1.61% to 9.02%.

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Corrosion Studies. A series of corrosion tests were made on coupons cut from a pulse generator piston-ring expander spring. The corrosion tests were made to determine the effect of fabricating methods on the corrosion resistance of the Type 304 stainless-steel springs. Stressed and relaxed specimens were exposed to 60% nitric acid at 70°F. and 220°F. and RCU solution at 70°F. Corrosion rates were found to be approximately 0.0006 inches per month in nitric acid at 220°F. and were negligible in nitric acid and RCU solution at 70°F.

A series of corrosion tests to determine the extent of pit corrosion of SAE-1010 mild steel when exposed to the vapor phase of boiling Purex neutralized waste is two-thirds complete. After two months of exposure the maximum pit depth on the polished specimens was 2.1 mils, a 10% increase over pit depths observed after one month exposure. Pit depths of 1.8 mils were observed on sand-blasted specimens after two month exposure.

A corrosion study of stainless steel in the Purex No. 1 and No. 2 acid concentrator bottoms has been initiated. Duplicate specimens of 309 SCb, 304L, 316L, and Carpenter 20 are being exposed to boiling liquid and vapor for five 48-hour periods.

Process Chemistry

Purex Waste Concentration Equilibrium Data. The procurement of vapor-liquid equilibrium data for the conditions of the Purex waste concentrators has been essentially completed with the determination of six additional equilibrium points by means of an Othmer still. The equilibrium nitric acid concentrations, determined with initial concentrations of from 174 to 584 g. HNO_3 /l., ranged from 205 to 640 g./l. and from 6.8 to 301 g./l. in the liquid and in the condensed vapor, respectively. The results are presented in more detail in Table IX.

Contact Angles on Kel-F NW-25. Contact angle measurements were made on a Kel-F - NW-25 coated 3-inch stainless steel pulse column plate received from Chemical Proof Construction Co., via D. E. Pederson on 11-24-52. The method used was as outlined in the memo: "Purex Pulse Column Plate Contact Angle Measurements", M. K. Harmon to G. Sege, 9-3-52. The following results were obtained:

Water in air (5 random positions)

(1) 82°, (2) 84°, (3) 84°, (4) 84°, (5) 90°

Water in 30% TBP - hydrocarbon

180° (no wetting)

30% TBP - hydrocarbon in air

0° (complete wetting)

30% TBP - hydrocarbon in water

0° (complete wetting)

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321 Building Operation

Three shift per day, 5 day per week (Monday through Friday) operation of "cold" semiworks experimental solvent-extraction columns was carried on during the month. The results of these studies are presented elsewhere in this report.

Excavation for the new tank farm pit for the Purex prototype equipment was completed and the concrete pipe trench was poured. Specifications for the concrete floor of the pit and the structural steel portions of the tank farm were completed. Specifications for the water fog system and demineralized water equipment were transmitted to the Project Section.

Hot Semiworks

Operation of the Hot Semiworks equipment was confined to processing of un-irradiated uranium. Initial "cold" runs were completed on all equipment pieces. Feed for the second "cold" run was prepared according to Run Plan HE-2. Operation has been intermittent to allow necessary alterations and maintenance work to be performed. Alterations to the sampling equipment were completed except for additional inert-gas headers in the sample galleries. Design of a down-draft heat exchanger tower for the dissolver was completed.

Uranium feed from head-end run HE-1 was processed through the first decontamination cycle at varying IAF flow rates to determine the operating range of the equipment. Similarly varying 2DF and 2AF flow rates were employed in the second uranium and second plutonium decontamination cycles. Uranium waste losses obtained on the first and second cycles are summarized below. The wide range resulted from variations in flow rates and from mechanical shakedown difficulties

Run No.	Stream	Per Cent U Loss	
		Lowest	Highest
HE-1-1A-1	IAW	<0.01	3.6
	IBP	<0.01	0.04
HE-1-2D-1	2DW	<0.01	0.02

The lowest waste losses were obtained with a 1A Column volume velocity of 505 gal./ (hr.) (sq.ft.), sum of IAX and IAW flow rates, and a 2D Column volume velocity of 490 gal./ (hr.) (sq.ft.), sum of 2DX and 2DW flow rates.

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TABLE I

DECONTAMINATION OF 101-C SLURRY AS A FUNCTION OF
SCRUB COMPOSITION AND INERT Ce DILUTION

Laboratory batch extractions.

RAX - Plant sample of 30% TBP in hydrocarbon

RAF - 101-C Slurry

RAS - C.P.

One extraction (RAFS + RAX) and one strip stage (no scrub)

S/F/X/Strip = 1/2/2/4

<u>No.</u>	<u>Description</u>	<u>B in RCU, % of Natural U</u>
1	Flowsheet RAS ^(a)	7112
1a	RAS - 2 M HNO ₃	7150
2	RAS - Flowsheet • 0.05 M C ₂ O ₄ ⁼	6512
2a	RAS - 2 M HNO ₃ • 0.05 M C ₂ O ₄ ⁼	6984
3	RAS - Flowsheet • 0.01 M F ⁻	9224
3a	RAS - 2 M HNO ₃ • 0.01 M F ⁻	6983
4	RAX Saturated with Ce(NO ₃) ₃ , 0.007 M Flowsheet RAS	2900
4a	RAX Saturated with Ce(NO ₃) ₃ , 0.007 M RAS - 2 M HNO ₃	3088
5	Flowsheet RAS - C.P. RAX	5770

(a) Flowsheet RAS contained 0.05 M ferrous ammonium sulfate and 0.1 M sulfamic acid in addition to 2 M HNO₃.

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TABLE II

DECONTAMINATION OF 101-C SLURRY WITH
ONE SCRUB STAGE

Laboratory batch extractions conducted by shaking the phases vigorously for one minute in separatory funnels.

RAX - Plant sample of 30% TBP in hydrocarbon.

RAF - 101-C Slurry.

RAS - C.P. chemicals: 2 M HNO_3 , 0.05 M ferrous ammonium sulfate, 0.1 M sulfamic acid. One extraction (RAF + RAX), one scrub, and two strip stages.

S/F/X/Strip = 1/2/2/4.

All data were obtained under the above conditions unless otherwise noted. Samples were counted 2 to 3 hr. after extraction.

<u>No.</u>	<u>Description</u>	<u>B in RCU, % of Natural U</u>
1	Control (conditions as above)	340
2	RAS - 2 M HNO_3 , 0.05 M $\text{C}_2\text{O}_4^{2-}$	294
3	RAS - 2 M HNO_3 , 0.01 M F^-	311
4	Two-minute scrub with flowsheet scrub solution	214
5	Scrubbed in vibrational D.T. tester at 42 cycles/min. for 1 minute	478
6	Ditto, at 86 cycles/min. for 1 minute	130
7	150% of RAS flowsheet flow ratio	53*
8	200% of RAS flowsheet flow ratio	76
9	RAS - 2 M HNO_3 , 0.01 M $\text{Cr}_2\text{O}_7^{2-}$	981
10	Two extraction contacts to increase % U saturation	19
11	Ditto	27
12	75% of RAX flow ratio	55
13	85% of RAX flow ratio	109
14	70% of RAX flow ratio	216
15	RAS - 2 M HNO_3 , 0.01 M MnO_4^- 70% of RAX flow ratio	50
16	RAS - 2 M HNO_3 , 0.01 M Cr(III) RAF made 0.05 M $\text{C}_2\text{O}_4^{2-}$ held at simmer for 1 hr.	232
17	RAF made 0.05 M NO_2^- , held at simmer for 1 hr.	213
18	Flowsheet RAS + 0.01 M SnCl_2	363
19	RAX equilibrated with 2 M NaNO_3 - 0.01 M $\text{Ce(NO}_3)_3$	186
20	RAX saturated with $\text{Ce(NO}_3)_3$, 0.007 M	172
21	RAF containing 0.01 M $\text{Ce(NO}_3)_3$ flowsheet scrub	191
22	RAS - 2 M HNO_3 , 0.01 M $\text{Ce(NO}_3)_3$	210
23	75% of RAF flow ratio	339
24	RAF - 103-U Slurry	111
25	UO_3 formed by calcination of product from Run 24	112

*Counted 12 hours after extraction.

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TABLE III

SPECIFIC FISSION PRODUCT ANALYSES

Sample	Gross		Ce		Ru	Sr	Cs		Y	Trivalent Rare Earths		Material Balance, %	
	B	G	B	G	B	B	B	G	B	B	G	B	G
Synthetic HCU(a) (from 101-U RAP)	1560 (100%) (12-16)	333	61(3.9)	173(51.9)	57(3.7)	8(0.5)	22(1.4)	148(44.4)	1160(74.4) (12-15-52)	278(17.8)	136(40.8)	101.7	137.1
	1230 (12-17)								668 (12-18-52) 124 (12-24-52)				
RAF-5(b) (101-U, Nov. 2)	1.40 ⁷	7.94 ⁶	6.8 ⁵ (4.86%)	1.35 ⁵ (1.7)	3.8 ⁵ (2.7)	2.65 ⁶ (18.9)	5.74 ⁶ (41.0)	8.09 ⁶ (101.8)	3.11 ⁶ (22.2)	1.04 ⁶ (7.4)	6.9 ⁴ (0.9)	97.1	104.4
RAF-15(b) (101-C, Dec. 11)	2.36 ⁷	9.55 ⁶	1.12 ⁶ (4.75)	1.7 ⁵ (1.8)	2.78 ⁵ (1.2)	6.57 ⁶ (27.8)	6.82 ⁶ (28.9)	8.26 ⁶ (86.5)	6.87 ⁶ (29.1)	2.53 ⁶ (10.7)	1.46 ⁵ (1.5)	102.5	89.8
RAF-9(b) (101-C, Nov. 27)	1.79 ⁷	9.49 ⁶	7.38 ⁵ (4.12)	1.06 ⁵ (1.1)	3.04 ⁵ (1.7)	4.0 ⁶ (22.3)	7.63 ⁶ (42.6)	8.58 ⁶ (90.4)	4.50 ⁶ (25.1)	1.63 ⁶ (9.1)	9.5 ⁴ (1.0)	104.9	92.5
RAF-11(b)	2.6 ⁷	9.04 ⁶	1.77 ⁶ (6.8)	2.12 ⁵ (2.3)	3.28 ⁵ (1.26)	6.3 ⁶ (24.2)	6.13 ⁶ (23.6)	7.86 ⁶ (86.9)	7.28 ⁶ (28.0)	2.56 ⁶ (9.8)	1.44 ⁵ (1.6)	93.7 99.98	90.8 102.9
													Average

Notes:

- Prepared and submitted for analysis on 12-16-52; gross B determined on 12-17-52; Y^{90} B determined on 12-18-52. Material balance calculations made on basis of extrapolating both determinations to zero time after extraction. Numbers in parentheses are per cent of initial gross activity contributed by the individual fission product in question.
- TRF Plant solutions.
- B - 2nd. shelf BGO (Beta-Gamma-Offner).
G - Gamma scintillation counter employing 1.7 g./sq.cm. Pb absorber.
All values counts/(min.)(xl.).
Exponential expressions, e.g. 2.6⁷, = 2.6×10^7 .

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TABLE IV

COMPARATIVE ANALYSES OF UO_3 SAMPLES

	Harshaw UO_3 No. SA-2		Mallinckrodt UO_3 No. U-2823		Purex UO_3 No. 2736	
	Hanford	Oak Ridge	Hanford	Oak Ridge	Hanford	Oak Ridge
UO_3	98.8%	99.17%	93.8%	93.83	97.5	97.91
U_3O_8	0.20	0.34	< .01	0.22	0.73	0.65
HNO_3	.30	0.29	.33	0.39	.58	.25
H_2O	.35	0.38	5.51	5.60	.90	1.00
Pu	<1 ppb	<2 ppb	<1 ppb	<2 ppb	.4 ppb	<2 ppb
Bulk Dens.	2.84	3.1	2.54	2.9	2.30	2.5
Part. Size	96%	96.8%	81	69.8	93.5	95.6
Surf. Area	1.9	1.17	0.15	0.18	1.6	1.71
Tot. Beta	100%	76%	100%	98%	100%	101%
Tot. Gamma	100	84	103	94	131	114
Conversion ratio	0.921		0.808		1.10	
(Harshaw control)	0.921		0.801		1.11	

	3706			234-5			3706			234-5			3706			234-5	
Al	5	<1	2	20	5	<1	20	20	5				20	20	5		
B		<1			<1									<1			
Ca	<50	50		<50	100		<50	100					<50	100			
Cr	<5	5	<5	<5	2	2	<5	2	2				5	20	7		
Cu	10	<20	2	10	20	2	10	20	2				50	20	8		
Fe	40	20	3	25	<10	12	25	<10	12				<10	100	80		
K		1			<1									2			
Mg	2	5	0.5	1	2	5	1	2	5				5	5	7		
Mo	<5	<20	<3	<5	<20	<3	<5	<20	<3				<5	<20	<3		
Na	50	<10	12	20	<5	4	20	<5	4				200	<50	30		
Ni	10	<5	<1	<5	<5	2	<5	<5	2				20	50	15		
P	<20	<200	<10	<20	<200	<10	<20	<200	<10				50	<200	<10		
S			6			6			6						16		
Si	11	5	5	35	20	3	35	20	3				23	50	25		

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TABLE 1
GRABBLE 450/251 3-IN. RA PULSE COLUMN, Volume Velocity, 100 U/Day

Flowsheet: TSP-RA #4 Flowsheet, modified as noted; P.P.'s omitted; RAI = 31; volume L/P; interface at 100.
Equipment: Dual-purpose dual-diameter glass pulse column.^(a)

Run No.	Volume Velocity, Gal./(Hr.)(24 Ft.), Sum of both Phases	Amplitude, In.	Frequency, Cyc./Min.	% U Loss	H.T., Ft. (c)	Stability (c)	Total hours of Operation	Remarks
Flowsheet modifications: Volume flow ratios RAF:RAS:RAI = 100:50:75. Volume velocity for 5 Tons U/Day in a 20-in. column = 910.								
3"-95-RA	800	1.25	40	0.25	1.9	Stable	12	
3"-96-RA	810	1.25	50	0.33	1.4	Stable	2.6	
3"-100-RA	800	1.25	55 ± 5	—	—	Moderate instability	—	Cyclic local flooding threshold.
3"-100-RA	800	1.25	65 ± 5	—	—	Pronounced instability	—	Complete flooding threshold.
3"-101-RA	1120	1.25	40	4.6	3.7	Stable	1.7	
3"-102-RA	> 2400	1.25	40	—	—	Stable	—	Complete flooding threshold was not reached.
3"-103-RA	420	1.25	40	0.68	2.3	Stable	5	
Flowsheet modifications: RAS = 5 M HNO ₃ ; volume flow ratios RAF:RAS:RAI = 100:50:105. Volume velocity for 5 Tons U/Day in 20-in. column = 910.								
3"-104-RA	880	1.25	45	0.01	1.3	Slight instability	2.7	
3"-106-RA	860	1.25	40	0.01	1.3	Stable	17	
Flowsheet modifications: Simulated "supernate" feed; RAF = 0.080 M U, 1.6 M H ⁺ , 3.1 M NO ₃ ⁻ , 0.18 M SO ₄ ²⁻ , 0.12 M PO ₄ ³⁻ , 2.4 M Na ⁺ . Volume flow ratios RAF:RAS:RAI = 355:50:105. Volume velocity for 5 Tons U/Day in a 20-in. column = 1730.								
3"-108-RA	1690	1.25	45 ± 5	—	—	Pronounced instability	—	Complete flooding threshold.
3"-110-RA	1800	1.25	40	11	4.3	Stable	4	
3"-111-RA	910	1.25	50	0.08	1.3	Stable	1.1	
3"-112-RA	2050 ± 250	1.25	40	—	—	Pronounced instability	—	Complete flooding threshold.
Flowsheet modifications: Simulated "lean sludge" feed; RAF = 0.41 M U, 4.5 M H ⁺ , 6.1 M NO ₃ ⁻ , 0.06 M SO ₄ ²⁻ , 0.40 M PO ₄ ³⁻ , 2.4 M Na ⁺ . Volume flow ratios RAF:RAS:RAI = 60:50:105; volume velocity for 5 Tons U/Day in a 20-in. column = 840.								
3"-113-RA	1250 ± 200	1.25	40	—	—	Moderate instability	—	Cyclic local flooding threshold.
3"-113-RA	2000 ± 200	1.25	40	—	—	Pronounced instability	—	Complete flooding threshold.

3-116-RA	1030	1.25	40	0.009	1.2	Stable	3.8
3-117-RA	510	1.25	50	0.009	1.2	Stable	4.5
3-118-RA	1200	1.25	38	0.04	1.4	Stable	2.7

Flowsheet modifications: Simulated "soft sludge" feed; RAS = 0.28 M U, 4.9 M H⁺, 6.1 M NO₃⁻, 0.27 M SO₄²⁻, 0.54 M PO₄³⁻, 2.3 M Na⁺. Volume flow ratios RAS:RAS:RAI = 77:50:10. Volume velocity for 5 tons U/day in a 20-in. column = 760.

3-119-RA	1600 ± 200	1.3	40	—	—	Moderate instability	—	Cyclic local flooding threshold.
3-119-RA	1800 ± 200	1.3	40	—	—	Pronounced instability	—	Complete flooding threshold.
3-120-RA	1190	1.3	50 ± 10	—	—	Moderate instability	—	Cyclic local flooding threshold.
3-120-RA	1190	1.3	60 ± 10	—	—	Pronounced instability	—	Complete flooding threshold.
3-121-RA	1190	1.3	50	0.04	1.2	Pronounced instability	1.7	
3-121-RA	1190	1.3	40	0.55	2.1	Stable to slight inst.	1.5	
3-122-RA	1190	1.3	50	0.31	1.2	Stable	3.1	
3-123-RA	660	1.3	50	0.31	1.2	Stable	3.1	
3-124-RA	1400	1.3	40	0.35	2.3	Stable	1.2	

Flowsheet modifications: RAS split into two streams: the RAS (2 M H₂SO₄) entering at the top of the scrub section, and the RAS (2 M H₂SO₄, 2.10 M Fe(NH₄)₂(SO₄)₂, 0.20 M H₂SO₄) entering at the midpoint of the scrub section. Volume flow ratios RAS:RAS:RAI = 100:25:25:155. Volume velocity for 5 tons U/day in a 20-in. column = 910.

3-125a-RA	890	1.3	40	0.02	1.3	Stable	24	(d)
3-125b-RA	900	1.3	45	0.04	1.1	Stable	4.2	
3-125c-RA	900	1.3	50	0.02	1.1	Stable	9.1	
3-125d-RA	900	1.3	60 ± 5	—	—	Moderate instability	—	Cyclic local flooding threshold.
3-125e-RA	900	1.3	70 ± 5	—	—	Pronounced instability	—	Complete flooding threshold.

Flowsheet modifications: Simulated "supernate" feed; RAS = 0.077 M U, 1.8 M H⁺, 3.4 M NO₃⁻, 0.14 M SO₄²⁻, 0.14 M PO₄³⁻, 2.5 M Na⁺. Volume flow ratios RAS:RAS:RAI = 355:50:1.5. Volume velocity for 5 tons U/day in a 20-in. column = 1790.

3-126a-RA	890	1.3	50	2.8	2.8	Stable	11
3-126b-RA	910	1.3	55	2.8	2.8	Stable	2.5

Flowsheet modifications: Simulated "supernate" feed; RAS = 0.081 M U, 1.8 M H⁺, 3.4 M NO₃⁻, 0.15 M SO₄²⁻, 0.14 M PO₄³⁻, 2.3 M Na⁺. Volume flow ratios RAS:RAS:RAI = 355:50:1.21. Volume velocity for 5 tons U/day in a 20-in. column = 1760.

3-126c-RA	900	1.25	55	0.08	1.1	Stable	8.0
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NOTES:

(a) Equipment: Dual-purpose dual-diameter column; extraction sections 3.00 & 0.01-in. i.d. precision bore glass column; scrub sections 4-in. regular bore glass column; plate section heights: scrub = 6.2 ft., extraction = 12.3 ft.; perforated plates: stainless steel, 0.125-in. diameter holes, 23% free area, 2-in. spacing. Top disengagement section consisted of a 2.5-ft. vertical "neck" at the top of which was a 5 ft. horizontal "sidewall" each 4 in. in diameter. The interface was normally maintained in the sidewall.

(b) Height of a transfer unit, over-all aqueous-film basis. Equilibrium data obtained by extrapolating data from BS-17747.

(c) The operation was considered stable when no substantial periodic changes occurred in the observed phase dispersion behavior.

Slight instability was characterized by cyclic tightening of the emulsion. I.e., periodically the dispersed-phase droplets became gradually finer and more closely packed, the tight emulsion then breaking by coalescence of the dispersed-phase droplets into globs (typically 1/4 in. to 2 in. in diameter).

Moderate instability involved cyclic local flooding, with periodic phase inversion over not more than about 2 ft. of the plate section.

Pronounced instability was characterized by phase inversion over more than 2 ft. of the plate section. Typically, pronounced instability started as a cyclic local flood which, when operation was sufficiently prolonged, eventually became a complete flood.

In the cases tabulated, instability invariably originated 3 to 9 ft. from the bottom of the extraction section.

(d) The interface during the first 20 hours of the run was maintained in the "neck" of the horizontal "sidewall", developing during this time about 1/4 to 1/2 in. of closely packed "sidewall" emulsion below the interface. The interface was then lowered into the vertical "neck" of the disengaging section and the sidewall grew to a stable depth of 12 to 14 in. It was noted that the air to the lower interface control dip tube, at a rate of about 0.5 cu.ft./hr., was causing considerable agitation of the sidewall. The air was reduced to about 0.15 cu.ft./hr., whereupon the depth of sidewall decreased to a maximum of about 1 in.

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TABLE VI

URANIUM RECOVERY 3-IN. RA SCRUB PULSE COLUMN
COMPLETE FLOODING STUDIES USING 30% TBP

Flowsheet: TBP-HW#4 with the following modifications: RASF = scrubbed RAU (30 vol.% TBP)^(c) RAS:RAS volume flow ratio = 50:105, F.P.'s omitted. Interface at top.

Equipment: 3-in. glass pulse column^(a).

Run No.	Amplitude, In.	Frequency Cyc./Min.	Volume Velocity, ^(b) Gal./Hr.)(Sq.Ft.), Sum of Both Phases
3"-18-RAS	1.25	50	400 ± 100
3"-19-RAS	1.25	40	600 ± 100
3"-20-RAS	1.25	47.5 ± 2.5	400
3"-21-RAS	1.0	40	600 ± 100

NOTES:

- (a) Equipment: Simple 3.00 ± 0.01-in. i.d. precision bore glass column; plate section height = 12.3 ft.; perforated plates: stainless steel, 0.125-in. diameter plates, 23% free area; 2-in. spacing. Interface maintained in the 4-in. vertical section about 1 foot above the RAS feed point.
- (b) Five tons U/day capacity in a uni-diameter 20-in. column is about 550 gal./hr.)(sq.ft.), sum of both phases, in the scrub section.
- (c) Composition of RAU = 54 g.U/l., 13 g.HNO₃/l. Sp.Gr. = 0.868.

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TABLE VII

RELATIVE RECOVERY 1-10, NO PULSE COLUMN STUDIES LISTED FOR THE

Flow sheets: The H₂ #4, modified as noted; HCF was HAV from KA runs listed on preceding table (30 vol. % THF).
Volume flow ratio basis: HAF (THF H₂ #4 flow sheet composition) = 100.
Equipment: 1-in. glass pulse column. (a)

H.T.U. STUDIES

Run No.	Interface	Ampl., Frequency, Cyc./Min.	Pulse Ampl. x Freq., In./Min.	Sum of both phases, Cyc./Min.	Volume Velocity, Vol./Hr. (59.1% THF)	Vol. Loss, L/V, H.T.U.	Composition HCF H ₂ O	Nominal Volume Flow Ratio HCF/H ₂ O	Volume Velocity for 5 Tons U/Day Production in a 30-in. Column	Remarks
3-43-HC	Top	0.6	80	48	455	1.8	0.03	200:110	490	Operated 12 hours.
3-43-HC	Top	0.6	63	36	750	1.3	0.03	150:110	410	Equilibrated and operating lines crossed. (a)
3-43-HC	Top	0.6	80	48	385	1.3	0.03	150:110	410	
3-43-HC	Top	0.6	70	42	320	1.3	0.04	150:80	360	
3-43-HC	Top	0.6	60	36	410	1.8	0.07	150:80	360	
3-43-HC	Top	0.6	75	45	400	1.8	0.07	150:80	360	The HAT flow rate in the runs was adjusted to

3"-52-RC	Top	0.6	60	36	960	2.5	0.48	2.0	72	0.03	200:180	440
3"-53-RC	Top	0.6	75	45	930	2.4	0.56	2.0	72	0.03	200:180	440
3"-54-RC	Top	0.6	60	36	710	1.7	1.5	1.8	72	0.04	120:80	320
3"-55-RC	Top	0.6	75	45	380	1.6	0.58	1.5	70	0.03	120:80	320
3"-56-RC	Top	0.6	70	42	470	1.9	0.18	1.5	57	0.03	200:110	490
3"-57-RC	Top	0.6	60	36	470	1.9	0.01	1.2	57	0.03	200:110	490
3"-60-RC	Top	0.6	70	42	470	1.9	0.08	1.5	57	0.01	200:110	490
3"-61-RC	Top	0.6	80	48	460	1.8	0.01	1.2	57	0.01	200:110	490
3"-64-RC	Top	0.6	70	42	470	1.9	0.08	1.5	59	0	200:110	490
3"-65-RC	Top	0.6	80	48	450	1.9	0.093	1.1	59	0.006	200:110	490
3"-68-RC	Top	0.6	70	42	450	1.9	0.26	1.2	59	0.1	200:110	490
3"-69-RC	Top	0.6	80	48	460	1.8	0.004	0.82	59	0.1	200:110	490
3"-63-RC	Bottom	0.5	40	20	325	1.9	0.24	1.8	54	0.03	200:110	490

at the RAP feed point that was 75% saturated with respect to uranium.

The RAS HNO₃ concentration was 5% in the RA runs preceding these runs. HNO₃ in the RCF was about 0.54%.

COMPLETE FLOODING STUDIES

3"-47-RC	Top	0.6	115 ± 5	69 ± 3	400	1.7	—	—	73	0.03	150:80	360
3"-48a-RC	Top	0.6	75	45	1100 ± 100	1.9	—	—	73	0.03	150:80	360
3"-48b-RC	Top	0.6	60	36	1100 ± 100	1.9	—	—	73	0.03	150:80	360
3"-58-RC	Top	0.6	110 ± 10	66 ± 6	470	1.9	—	—	57	0.03	200:110	490
3"-59a-RC	Top	0.6	60	36	800 ± 100	1.9	—	—	57	0.03	200:110	490
3"-59b-RC	Top	0.6	50	30	1300 ± 200	1.9	—	—	57	0.03	200:110	490
3"-62-RC	Top	0.6	107 ± 7	64 ± 4	460	1.8	—	—	57	0.01	200:110	490
3"-63-RC	Top	0.6	60	36	1100 ± 200	1.8	—	—	57	0.01	200:110	490
3"-66-RC	Top	0.6	105 ± 5	63 ± 3	470	1.8	—	—	59	0	200:110	490
3"-67a-RC	Top	0.6	60	36	1100 ± 100	1.9	—	—	59	0	200:110	490
3"-67b-RC	Top	0.6	50	30	1200 ± 100	1.8	—	—	59	0	200:110	490
3"-70-RC	Top	0.6	104 ± 4	62.5 ± 2.5	460	1.8	—	—	59	0.1	200:110	490
3"-71-RC	Top	0.6	60	36	1000 ± 100	1.8	—	—	59	0.1	200:110	490
3"-81-RC	Bottom	0.6	40	24	870 ± 50	1.9	—	—	54	0.03	200:110	490

See Runs 3"-49 through 55-RC, above.

See Runs 3"-56 through 69-RC, above.

CYCLIC LOCAL FLOODING STUDIES

3"-44a-RC	Top	0.6	70	42	670 ± 100	1.3	—	—	54	0.03	150:110	410
3"-44b-RC	Top	0.6	65 ± 5	39 ± 3	770	1.3	—	—	54	0.03	150:110	410
3"-44c-RC	Top	0.6	40	24	1080	1.3	—	—	54	0.03	150:110	410
3"-47-RC	Top	0.6	85 ± 5	51 ± 3	400	1.7	—	—	73	0.03	150:80	360
3"-48a-RC	Top	0.6	75	42	900 ± 100	1.9	—	—	73	0.03	150:80	360
3"-58-RC	Top	0.6	90 ± 10	54 ± 6	470	1.9	—	—	57	0.03	200:110	490
3"-62-RC	Top	0.6	90 ± 10	54 ± 6	460	1.8	—	—	57	0.01	200:110	490
3"-66-RC	Top	0.6	90 ± 10	54 ± 6	470	1.8	—	—	59	0	200:110	490
3"-71-RC	Top	0.6	90 ± 10	54 ± 6	460	1.8	—	—	59	0.1	200:110	490
3"-81-RC	Bottom	0.6	40	24	870 ± 50	1.9	—	—	54	0.03	200:110	490
3"-82-RC	Bottom	0.6	40	24	870 ± 50	1.9	—	—	54	0.03	200:110	490

See Runs 3"-49 through 55-RC, above.

See Runs 3"-56 through 69-RC, above.

NOTES

- Equipment: (a) 1-in. i.d. precision bore glass column; plate section height: 12.3 ft.; perforated plate: stainless steel, 0.18-in. diameter holes, 33 free area; 2-in. plate spacing.
- Design of a transfer unit, over-all organic-phase basis.
- The crossing of the operating and equilibrium lines is thought to be due to the continuing transfer of nitric acid after the uranium transfer had reached equilibrium. The H₂O₂ was estimated by assuming one nitric acid stage at the bottom of the column before uranium transfer began, thus bringing the nitric acid equilibrium parameter below the operating line and enabling calculation of the uranium transfer units in the usual way. (Equal D₂O's were assumed for U and H₂O₂).

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URANIUM RECOVERY

Flowsheet: TBP-HW #4, modified as no
Volume flow ratio basis: RAF (TBP-HW)
Equipment: 3" packed glass pulse col

Run No.	Interface Position	Pulse			Volume Velocity, Gal./((Hr.)(Sq.Ft.)) Sum of Both Phases
		Ampl., In.	Freq., Cyc./Min.	Ampl. x Freq., In./Min.	
3"-73-RC	Top	0.6	70	42	800
3"-74-RC	Top	0.6	60	36	800
3"-76-RC	Top	0.6	70	42	920
3"-77-RC	Top	0.6	60	36	920
3"-79-RC	Bottom	0.6	60	36	940
3"-80-RC	Bottom	0.6	50	30	940
3"-84-RC	Top	ca. 0.5	80	ca. 40	930
3"-85a-RC	Top	ca. 0.5	70	ca. 35	920
3"-85b-RC	Top	0.65	70	45	920
3"-86a-RC	Top	ca. 0.5	80	ca. 40	920
3"-86b-RC	Top	0.65	80	52	920
3"-88-RC	Top	ca. 0.5	70	ca. 35	775
3"-90a-RC	Top	ca. 0.5	70	ca. 35	1065
3"-90b-RC	Top	0.65	70	45	1080
3"-72-RC	Top	0.6	85 ± 5	51 ± 3	800
3"-75-RC	Top	0.6	90 ± 10	54 ± 6	930
3"-78-RC	Bottom	0.6	75 ± 5	45 ± 3	940
3"-87-RC	Top	ca. 0.5	> 120	> 60	930
3"-89-RC	Top	ca. 0.5	> 120	> 60	775
3"-91-RC	Top	ca. 0.5	115 ± 5	ca. 58 ± 3	1065
3"-92-RC	Top	0.65	70	45	1500 ± 100(d)

NOTES:

- (a) Equipment: 3-in. packed glass rings.
- (b) Height of a transfer unit, over
- (c) The RAX flow rate for the RA ru
RAF feed point that was 75% sat
- (d) Cyclic local flooding occurred

TABLE VIII

Y 3-IN. RC PULSED PACKED COLUMN USING 30% TBP

ed; RCF was RAU from RA runs listed on preceding table (30 vol.% TBP).
#4 flowsheet composition) = 100.
mn.(a)

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H.T.U. STUDIES

L/V, Aq./Org. Volume Flow Ratio	% U Loss	H.T.U., Ft.(b)	Stream Composition		Nominal Volume Flow Ratio RCX:RCF	Volume Velocity for 5 Tons U/Day Production in a 30-In. Column	Remarks
			RCF G.U/L	RCX M HNO ₃			
1.8	0.34	1.5	70	0.03	150:80	360	} RCF from RA "75% saturation" runs.(c)
1.8	4.2	2.7	70	0.03	150:80	360	
1.8	0.23	1.7	49	0.03	200:110	490	Slight instability during r
1.8	1.5	2.2	49	0.03	200:110	490	
1.9	2.2	2.6	48	0.03	200:110	490	
1.8	5.1	3.2	48	0.03	200:110	490	
1.8	1.3	2.1	55	0.03	200:110	490	
1.8	2.2	2.6	55	0.01	200:110	490	
1.8	1.3	2.2	51	0.01	200:110	490	
1.9	1.4	2.3	55	0.01	200:110	490	
1.8	0.46	1.9	51	0.01	200:110	490	
1.45	11	3.9	55	0.01	150:110	410	
2.3	1.4	2.5	55	0.01	250:110	570	
2.3	0.53	2.1	50	0.01	250:110	570	

COMPLETE FLOODING STUDIES

1.8	—	—	70	0.03	150:80	360	(c)
1.8	—	—	49	0.03	200:110	490	
1.8	—	—	49	0.03	200:110	490	
1.9	—	—	55	0.01	200:110	490	
1.45	—	—	55	0.01	150:110	410	
2.2	—	—	55	0.01	250:110	570	
1.8	—	—	50	0.01	200:110	490	

column; packed height = 12 feet; packings: 1-in. stainless steel Raschig

all organic-film basis.

preceding those runs was adjusted to produce an RAU solution at the
rated with respect to uranium.

1300 ± 100 gal./hr.)(sq.ft.), sum of both phases.

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TABLE IX

PUREX WASTE CONCENTRATION

Vapor-Liquid Equilibrium Data

Apparatus: 1-liter Othmer equilibrium still.

Run No.	Initial Composition					Equilibrium Properties				Simulated Plant Stream
	HNO ₃ g./l.	H ₂ SO ₄ g./l.	Fe ₂ (SO ₄) ₃ g./l.	NaNO ₃ g./l.	Na ₂ SO ₄ g./l.	Liquid HNO ₃ g./l.	Density, g./ml.	Condensed Vapor HNO ₃ g./l.	Density, g./ml.	
1	174	10	4.0	3.4	1.4	204.9	1.104	6.83	0.998	INF
2	248	3.5	9.0	11.9	3.2	290.0	1.160	20.1	1.005	INF Conc.
3	323	5.9	14.0	20.4	5.0	376.3	1.213	49.1	1.019	"
4	398	8.4	19.0	28.9	6.7	464.9	1.263	92.1	1.045	"
5	473	10.8	24.0	37.4	8.5	479.3	1.257	110.6	1.056	INF
6	584	13.1	31.0	50.0	10.5	639.9	1.353	300.8	1.147	INF Conc.

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