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PROGRESS REPORT - CHEMICAL DEVELOPMENT

SEPARATIONS TECHNOLOGY UNIT

July, 1952

F. W. Woodfield
Chemical Development

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By Authority of C. H. Henager

CG-PR-2, 4-21-92

By J. E. Spuehly 9-27-94

Verified By Jerri Maley

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July 31, 1952

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PROGRESS REPORT - CHEMICAL DEVELOPMENT
JULY, 1952

REDOX PLANT ASSISTANCE

Plant Performance

The Redox Plant operated at a 76.7% time efficiency (IAF basis) and averaged 3.16 tons of uranium per operating day during the month. During the period from July 4 through July 13, 38,000 gallons of 72% ANN from Tygon-lined storage tank SS-112 were used in aqueous salt solution make-ups with some emulsion difficulty and fission-product carry-over resulting. New ANN, from stainless-steel tanks, was used for the balance of the month. An extensive (thermally) hot 60% HNO₃ cleanout was given to the IA, IB, 2D, 3D, 2A, and 3A Columns and associated salt solution head tanks to prepare the solvent-extraction battery for operation using 90-day "cooled" uranium. Continuous cross-over oxidation to improve 2A Column waste losses, and employment of KOH (vice NaOH) in 2DS, ICU, and 2EU butt additions to minimize Na in 3EU, were begun concurrently on July 20. Aged (90-day) uranium in head-end treated feed batches, was started to the IA Column on July 21, with early data indicating adequate two-cycle decontamination for both uranium and plutonium. The following is an over-all summary of plant production performance:

	<u>Approximate</u>
Tons of Uranium Processed	77.2
Plutonium Processed (Batch Equivalents)	119.1
Per Cent Uranium to Waste	1.4
Per Cent Plutonium to Waste	1.1

Operating Performance

A lapse in IA feed processing was scheduled between batches S-2-6-HE-10 (last of June, 1952; feed possibly contaminated with "extractable" radioruthenium during dissolving) and S-2-6-HE-11 to permit segregation of feeds through the Silo. Feed batches S-2-6-HE-11 through 15 (0.03M KMnO₄ oxidized) were then processed at nominal 2.5 tons per day from June 26 through July 1, when the first of twelve successive July feed batches oxidized with 0.2M Cr VI were started to the IA Column. The above-mentioned June 26 through July 1 processing was marked by (a) an initial high 3EU (uranium product stream) gamma activity, (12 times that of natural uranium), resulting in 0.7 ton of uranium being routed to rework; (b) some emulsification in 2A, and 2B Columns, requiring shut-down and organic flushing; and (c) over 1% Pu waste loss to 2AW. The initial high 3EU gamma count was probably due to residual activity remaining in process equipment from earlier operation.

A nominal 3 ton/day rate was established on July 3 (changed from 2.5) and maintained, except as indicated below for the period July 4 through 11, during which time ANN from Tygon-lined tanks was used:

July 7 - Reduced IAF rate to 2 tons/day when activity in uranium product streams increased.

July 9 - Shut down IA Column due to high organic density (emulsion), high differential pressure (extraction section pluggage) and hexone in D Cell via IAW.

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July 10 - Reduced rates to 2 tons/day to permit clean-up of 2A and 2B Column emulsions.

The above test demonstrated that Tygon-contaminated ANN can be used at a reduced average production rate. The test operation was of too short duration to result in any serious problems but evidence indicates that continued use of this material, without treatment, could result in difficulties similar to those experienced in March of 1952.

After completion of the test operation employing Tygon-contaminated ANN, processing of IAF was continued until July 16 using clean ANN, at nominal 3.5 ton/day rates except for a short-duration (4 hr.) "crash" shut-down due to water in hexone July 14 and an 8 hr. IA Column shutdown July 15 to clean the IA Column interface and extraction section.

During this 3.5 ton/day operation a 3EU rework (June E-12 Batch No. 25) was successfully carried out through the 2D Column (operating at 3.75 tons/day) by blending with normal production.

From July 16 to 20 the IA, IB, 2D, 3D, 2A, and 3A Columns were soaked, with air sparging, in (thermally) hot 60% HNO_3 for 12 hr. The IB Column flush was passed through the IBP Receiver (Tank E-6), Cross-Over Oxidizer (Z-7), 2AF Tank (E-5), and thence to the Waste Header Receiver (D-13) via the 2AW letdown valve. 32 units of plutonium were found in the combined flushes and transferred to the Rework Adjustment Tank (D-7) for subsequent rework through the IS Column. The ANN solution head tanks (8th level) were used to introduce the hot HNO_3 flush and later for introduction of water flushes, and thus were cleaned along with the columns.

On July 20, production was resumed to complete processing approximately 3 tons of feed uranium remaining before 90-day "cooled" material. This feed, from dissolver heels, was 0.03M permanganate-oxidized to minimize possible difficulties due to long dissolution times.

The IA Column was again shut down for approximately 6 hr. on July 21 to permit a lapse between dissolver heel processing and 90-day "cooled" test feed.

On July 21, IAF was restarted processing Batch HE 15 (the first 90-day "cooled" feed) at nominal 3 tons/day. Subsequent batches 16 through 18, permanganate-oxidized, were processed at 3 tons/day until July 25, when the processing rate was changed to 3.5 tons/day and 0.2M dichromate oxidized 90-day "cooled" feed was started to the IA Column.

The 3DF rotameter (F-4 FRC) failed on July 28. The cause is unknown at present. The 3DF flow controller is being operated manually on the basis of weight factor measurements. Some transient high uranium losses from the 3D Column first experienced on July 12, may have been caused by incipient failure of this control instrument.

The H-2 (Centrifuge) plow hydraulic system is leaking badly at month end and may not be adequate for essential plow operation if additional MnO_2 cake washes are required.

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During July, activity discharged through the ventilation stack averaged 0.5 curie/day of I^{131} and 0.001 curie/day of Ru. Maximums of 1.3 curies/day of I^{131} and 4.1 curies/day of Ru were observed during this period. Little change was observed in the rate of deposition of radioactive particles over the surrounding area during July as compared with June and no unusually large radioactive particles were observed. Weekly stack flushes are continuing with qualitative evidence of ammonium ion present in the sump sample taken at the month's end, July 28.

The following tables summarize decontamination and waste loss values by solvent-extraction cycle for selected periods during the month of July:

- Period covering 6-27-52 through 7-4-52; nominal production rate 2.5 tons U/day, processing head-end treated batches S-2-6-HE-11 through 14.

Cycle	Decontamination Factors (dF)						% to Waste	
	U			Pu			U	Pu
	Gamma	Beta	Ru	Gamma	Beta	Ru		
Feed	--	--	--	--	--	--	0.01	0.10
1st Cycle	3.8	3.6	--	3.9	4.5	--	0.22	0.26
2nd U	2.3	2.7	--	--	--	--	0.01	--
3rd U	0.4	0.3	--	--	--	--	0.03-1.8	--
2nd Pu	--	--	--	2.1	2.0	--	--	1.55
3rd Pu	--	--	--	1.5	0.9	--	--	0.06
Overall	6.5	6.6	5.1	7.5	7.4	6.5	0.27 to 2.12	1.95

Period covering 7-5-52 through 7-11-52; nominal production rate 3 tons U/day, processing 0.2M Cr^{6+} oxidized batches, using ANN from Tygon-lined storage tanks:

Cycle	Decontamination Factors (dF)						% to Waste	
	U			Pu			U	Pu
	Gamma	Beta	Ru	Gamma	Beta	Ru		
1st	3.6	3.8	--	3.9	4.4	--	0.04-1.5	0.5
2nd U	2.2	2.0	--	--	--	--	0.03	--
3rd U	0.7	0.6	--	--	--	--	0.01	--
2nd Pu	--	--	--	1.9	1.7	--	--	0.04-2.8
3rd Pu	--	--	--	1.0	0.8	--	--	0.05
Overall	6.5	6.4	5.3	6.8	6.9	5.6	0.08 to 1.6	0.6 to 3.4

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Period 7-21-52 through 7-25-52; nominal production rate 3 tons U/day, processing head-end treated batches S-2-7-HE 15 through 18.

Cycle	Decontamination Factors (dF)						% to Waste	
	U			Pu			U	Pu
	Gamma	Beta	Ru	Gamma	Beta	Ru		
Feed(a)	0.7	0.1	0.6	0.7	0.1	0.6	0.18	0.28
1st	3.3	4.6	3.3	3.2	4.5	3.6	0.35	0.04
2nd U	2.3	2.0	2.4	--	--	--	0.02	--
3rd U	(b)	(b)	(b)	--	--	--	0.013	--
2nd Pu	--	--	--	2.9	2.3	1.7	--	0.043
3rd Pu	--	--	--	1.1	0.4	0.5	--	0.015
Overall	6.3	6.7	6.3	7.9	7.3	6.4	0.56	0.38

Notes:

- (a) H-4 (Oxidizer) flushed with 2% H₂O₂ before these runs.
- (b) Negative dF experienced. Probable recontamination by solids in 3D Column. Possible source, interface emulsion left from earlier operation remained clinging to sides of top disengagement section during packing flush.

Feed Preparation

The dissolvers were charged during the month with fifteen 4.4-ton charges of uranium with an average pile exposure of 594 MWD/T. Heels of 1.62, and 1.13 tons were removed from Dissolvers A-2 and B-2, respectively, by incremental HNO₃ addition over 41 and 39-hr. periods. The average age of the 26 feed batches prepared as IAF was 73 days for Batches S-2-6-HE-11 to 15, and through S-2-7 HE-14 and 102 days for S-2-7-HE-15 through 21. The following table summarizes the treatments each batch received and includes pertinent annotations:

All batches 0.2M in Cr VI, sparge 4 hr. at 95°C. with 200 cu. ft./min. of air:

Batch	KMnO ₄ M	dF's			Waste Losses %		Pu IV, % in IAF
		Gamma	Beta	Ru	U	Pu	
S-2-6-HE 11	0.03	--	--	--	0.022	0.41	0.28
12	0.03	--	--	0.15	0.013	0.14	0.31
13	0.03	0.2	0	0.18	0.006	0.091	0.25
14	0.03	0	0.24	0	0.003	0.19	0.14
15	0.03	0.2	0	--	0.013	0.064	0.29

All batches 0.2M Cr VI, heat 4 hr. at 95°C.:

Batch	Average Waste Loss %		Pu IV % in IAF
	U	Pu	
S-2-7-HE-1 through 12	0	0	0.25
and 19 through 21	0.013	0.17	0.14

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S-2-7-HE Batch	KMnO ₄ M	dF's				Losses %		Pu IV% in IAF	Notes
		Gamma	Beta	Ru	Nb-Zr	U	Pu		
13	0.03	0.11	0.04	0	--	0.046	0.44	0.10	Dissolver heel
14	0.06	0	0	0	0	0.068	0.32	0.19	1/2 batch, dis- solver heel.
15*	0.06	0.63	0.08	0.62	1.6	0.08	0.16	0.085	
16	0.06	0.62	0	0.62	--	0.07	0.20	0.12	Concentrated before Mn VII addition.
17	0.08	0.59	0.09	0.95	--	0.37	0.11	0.11	
18	0.08	0.88	0.08	0.96	--	0.12	0.18	0.06	No sparge.

*Runs 15 through 18 employed manganous nitrate to complete the destruction of KMnO₄ after hot oxidation.

Uranium Extraction and Decontamination

In general, nominal conditions of the O.R.N.L. June, 1949 (acid-deficient) Flow-sheet (HW-22834) were employed throughout the uranium extraction cycle:

IAF:IAS:IAX:IBX:IBS:ICX = 1.0:1.0:4.0:0.4:2.0:2.0

2DF:2DS:2DX:2EX = 1.0:1.25:4.5:2.0 with 2DS modified to 1.8M ANN, 0.2M HNO₃-deficient, no ferrous sulfamate.

3DF:3DS:3DX:3EX = 1.0:1.0:4.0:2.0 with 3DS modified to neutral ANN, no ferrous sulfamate.

During the month a modification was made to the third uranium cycle flowsheet as follows:

June 27 through June 28: Used 2M ANN, 0.2M acid-deficient 3DS; then restored to neutral 2M ANN.

Average uranium losses were below the nominal flowsheet value for the month. Plutonium losses in the IAW ranged from 0.006 to 0.6% during the month. Adequate partitioning of U and Pu was achieved in the IB Column and through 2D-3D without Fe to maintain all uranium product batches within Pu in U specification. One uranium product (3EU) batch 5-2-7-E-12-20, with gamma activity about 9 times that of natural uranium, probably contaminated in third cycle equipment, was sent to rework. On July 20 KOH was substituted for NaOH in 2DF, 2DS, and 3DF acid-deficiency adjustment. Preliminary data indicate 300 parts K/10⁶ parts U in 3EU but Na values are still being reported at approx. 200 parts Na/10⁶ parts U.

Plutonium Extraction and Decontamination

All conditions of the HW#4 Flowsheet (except the 2AS and 3AS flow ratios) were maintained for Plutonium Cycle operation during the month. During operation at nominal rates of 3 tons/day or less, the 2AS and 3AS flow ratios were twice the flowsheet value; during operation at higher rates, the 2AS and 3AS flows were

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twice the 3 ton/day flowsheet rates. Following a series of exceptionally high 2AW Pu losses (1.5% of IAF) the IBS HNO_3 concentration was changed on July 4 from 0.01M to 0.015M. At the same time the cross-over oxidation procedure was changed so that IBP was acidified and oxidized after collection of one-half of the previous batch size; thus, the solution was butted within a shorter period. The 2AW Pu losses showed an immediate drop to flowsheet loss. On July 20, a continuous IBP butt via proportioning pump was instituted and was in satisfactory operation by July 22.

On July 14, the solvent-extraction battery was upset by water in the hexone supply, with the IB Column being most seriously affected. The "crash" shut-down and following start-up resulted in an excessive amount of uranium and fission products in the IBP solution. PR Batches S-2-7-L-62 through 65 were shipped to 224-T Building for processing because of high uranium content, ranging from 0.74 to 0.07 g.U/g.Pu, respectively. PR Batches S-2-7-L-66 and 67, containing 0.04 to 0.02 g.U/g.Pu, were shipped to 231 Building for processing. 231 Building processing has been normal, oxalate cake being shipped to 234-5 on boats. PR Batch S-2-7-L-73, collected immediately following the acid-soak, was shipped to 224-T because of solids in the solution. S-2-7-L-74 was processed in two parts because of high acidity caused by low AT concentration in 3BP.

Solvent Treatment and Recycle

The hexone losses for the period averaged 270 gal./day. The Hexone Still (G-3) and the IO Column operated with water as scrub during the entire period and all of the hexone recycled to the columns was treated in 276-S Building with a 5% NaOH wash followed by a water wash. 8000 gal. of new hexone were brought into the system and given a standard acid-dichromate pretreatment on July 25. (This represents about 1.5% of hexone to the columns.)

Forty per cent of current hexone is of Shell, and 60 per cent of Carbide and Carbon origin.

Rework

One 0.7-ton batch of uranium product (3EU) solution was reworked (see under Operating Performance) during the month. At month end three rework jobs are planned before continuing on with the test program for Pu flowsheet change evaluation. These reworks are: (a) 1/2 batch of S-2-6-HE-7 in the IS Feed Tank (F-8) to go to reoxidation in the Oxidizer (E-4) for IAF preparation; (b) 1500 gal. of salt waste in the Rework Adjustment Tank (D-7) to go to the IS Feed Tank (F-8) as ISF; and (c) 3EU Batch S-2-7-E-12-20 in the Rework 2DF Tank (E-13) to blend off with 2DF. It is planned to process these three off-standard feeds simultaneously.

Waste Processing

Ninety-five waste batches were neutralized and sent to underground storage and one batch was sent to the Rework Adjustment Tank (D-7) for recovery of 32 units of Pu. Average volumes were 2100 gal. concentrated salt waste, and 3900 gal. neutralized salt waste (before jetting to underground storage) per ton of U. No unusual difficulty was experienced in D-Cell (waste system) operation except for some waste condensate stripper upsets during the time when hexone entered the salt waste receiver via IAW.

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Rotating Equipment

On July 11, the Waste Concentrator agitator, A-D-9, a 5-horsepower unit operating at 125 rev./min., developed an increased seal leakage rate from 25 cc./hr. to 3 gal./min. The agitator will be run until complete failure (inability to maintain any seal cooling water in the seal housing) occurs.

A-H-4, the Oxidizer agitator, a 5-horsepower, 125-rev./min. unit, is being provided with a continuous flow of seal water through the seal housing to the sump through an overflow line in the housing.

Four regenerative turbine pumps have been ordered by the Manufacturing Department as identical spares for existing "hot" pumps. It is estimated that an additional 10 spare pumps will be ordered, which may be deepwell turbine type pumps similar in design to the Metal Recovery Plant "hot" pumps. Final design specifications for these pumps await the results of measurements of inert gas leakage through labyrinth-type seals. In the event gas leakage is low (a fractional part of the inert gas generator capacity of 200 cu.ft./min. per unit) the simplified deepwell turbine pump design coupled with a simple double labyrinth, inert gas purged vapor seal, is an attractive substitute for the more complicated present design of pump with rotary mechanical seal.

Instrumentation

The installation of a Merriam check valve at the canyon wall in the instrument air line to each "hot" bellows sealed flow control valve was recommended to the Manufacturing Department after failure of the bellows on the IAF flow control valve allowed contamination to enter the operating gallery.

ANN Clean-up

Filtration. The 250-sq.ft., 36-in.-diam., vertical-leaf Enzinger Union Corporation filter was received early in July. Installation of the filter and associated equipment is scheduled for completion on August 4.

Clarification by Settling. The results of a laboratory settling test employing a glass column approximately 1½ in. in diameter and 8 ft. tall showed that 8 to 10 days' settling time in the plant ANN storage tanks should be sufficient to produce a clarity of 92% (as compared with 97% for C.P. ANN) in a layer of solution 7 to 8 feet deep. This assumes that the solution remains undisturbed during settling, and that the initial volume per cent Al₂O₃ solids is 0.07% (clarity 50%).

Clean-Up of Tygon-Contaminated ANN. The effects of Darco G-60 and polythene as adsorbents for the Tygon contamination, were further studied. No really satisfactory adsorbents were found which could easily be removed by centrifugation. Of those which require filtration for removal, Darco G-60 has proved the most successful.

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- (a) Adsorption on Darco. The use of 3 to 5 grams of Darco G-60 per liter (of 62% ANN) at 25°C. for 2 to 5 hours' contact followed by 4 days' settling and subsequent filtration represent the optimum conditions for Darco.
- (b) Adsorption on Polythene. The use of 1/8" polythene cubes as an adsorption bed in a column appeared promising. However, since successfully processing approximately 50 liters of 62% ANN solution through a 1-inch I.D. column 12 feet long (packed with 1.5 pounds of polythene) at an optimum rate of 300 to 500 gallons/(hour)(sq.ft.), no means has been found for the regeneration of the polythene.

Process Chemistry

Head-End Treatment. Further investigation of the cause for the high rate of KMnO_4 reduction during plant head-end runs, led to the study of nitrite ion and total reducing molarity in metal solution samples. Five H-7 samples (plant metal solution storage tank) and two dissolver solution samples gave an average NO_2^- concentration of $7 \times 10^{-4}\text{M}$. The values for total reducing potential gave an average of $3 \times 10^{-3}\text{M}$, which leads to the conclusion that this reducing potential cannot account for the nearly 100% KMnO_4 reduction in plant head-end studies.

Laboratory studies showed that MnO_2 deposited on the walls of the H-4 (Oxidizer) tank could be readily removed by washing with a solution of 2% H_2O_2 in 1M HNO_3 . This was done in the plant prior to the current series of head-end runs, but did not prevent the usual reduction (approaching 100%) of KMnO_4 .

The laboratory-scale studies so far completed would account for no more than approximately 50 per cent reduction of KMnO_4 due to the combined effects of radiation, heat, surface condition, and reducing agents.

The effect of sparging in H-4 (Oxidizer) during the oxidation was unknown, and for this reason the final head-end batch (HE-18) in the plant was run without sparge, all other run conditions remaining unchanged. In this case, also, the permanganate reduction was essentially complete at the end of the 4-hour cycle.

Laboratory experiments on the dissolving of MnO_2 fines passing through the plant centrifuge have been completed. It was shown that the equivalent of 13 lb. of $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ added to 1600 gallons of centrifuged feed and digested at 70°C. for 2 hours, was sufficient to dissolve the maximum amount of MnO_2 (0.002M) to be expected, with no adverse effect on Pu valence in the IAF feed solution.

Solvent Extraction. Studies of the loss of 2nd and 3rd U cycle decontamination have been completed. The fault was found to lie in the aqueous feed to the solvent-extraction battery rather than with the solvent as was originally suspected. The cause was found to be the result of a "cold" dissolving in which the metal in the dissolver remained in contact with the HNO_3 for an unusually long period of

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time (18 hr.) at a much lower-than-average temperature (40 to 50°C.). The laboratory experiments indicated that the offending fission product, primarily Ru, could be removed by a KMnO_4 oxidation treatment followed by an air sparge, thus producing an aqueous solution of normal extraction characteristics.

Studies have been continued using the 3/8-inch 12-stage Mini (miniature mixer-settler) as a 2D (or 3D) Column, testing modifications to the Redox flowsheet intended to reduce the Na, Al, and Fe content of recovered uranium. The 20 runs originally planned have been completed and the results are being compiled in a report. Four additional modified O.R.N.L. flowsheet runs employed: (1) acid-deficient scrub prepared by dissolving Al_2O_3 in HNO_3 ; (2) acid-deficient scrub prepared with NH_4OH ; (3) acid-deficient scrub (NaOH) with Fe absent; and (4) neutral scrub with Fe absent. The data so far available are given in Table I. The acid-deficiency of 2DS in Run 1 was not as great as desired, and this run is being repeated. The use of NH_4OH appears very promising and will be studied further. The use of KOH in place of NaOH , will also be tried, and its effect on reactivity of the final UO_3 product will be determined.

Solvent Treatment. The plant spare hexone distillation column (G-3) is being modified by removing all of the bubble cap plates, in order to permit distillation of the plant hexone from a caustic heel. Laboratory runs are in progress to determine the effect of such a still on the fission-product decontamination of the solvent. The results are shown in Table II. The equipment used in the laboratory was not quite to scale, and as a result the vapor velocity through the column was estimated to be approximately three times that of the plant still, in spite of the fact that the maximum feed rates obtainable were only 40 to 70 per cent of plant rates. The beta and gamma content of the distillate was at or below the limit of detection, as a result of which it is predicted that this method will give satisfactory results.

Preparation of ANN from Al_2O_3 . A laboratory study of the preparation of an acid-deficient solution of ANN from Al_2O_3 is currently under way. A successful procedure has been demonstrated in the laboratory, and further studies are in progress to find the maximum rate and optimum initial HNO_3 concentration, which will give the minimum amount of residual undissolved Al_2O_3 . The results to date are given in Table III.

URANIUM RECOVERY PLANT ASSISTANCE

241-U Tank Farm Operation

Replacement of the slurry jets to the blend tanks (from designed 25 gpm. to 100 gpm. jets which deliver 60 gpm.) permitted renewal of blending operations early in the month. Intermittent sluicing and blending operations proceeded uninterrupted for about two weeks. Although mist in the tank prevented visual inspection, sluicing results during this period were the best obtained to date, and approximately 2 to 3 tons of uranium were removed per day. However, since that time sluicing operations have been hampered by mechanical difficulties.

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The most serious of these were: (a) failure of the sluice pump starting switch; (b) failure of the 101 sludge pump; and (c) rupture of the sluice nozzle hoses. Even after repair of these items, effective sluicing has not as yet been realized, and the uranium removal has approximated one ton per day.

The average of the analytical results from the first ten blend batches transferred to 241-WR Process Vault indicate that the blends were made from a slurry somewhat high in supernate. In addition, the blended feed has been diluted with water by one or more of the following means: (a) excessive jet dilution; (b) dilute nitric acid; or (c) water added from the vault sumps. The following table gives the average of the analytical results for the batches transferred, the calculated analysis of this material when concentrated to flowsheet uranium concentration, and the values for the TBP HW#4 Flowsheet.

RAF Composition

	Sp.Gr.	U	Concentration in Moles/Liter			
			H ⁺ (a)	Na ⁺	PO ₄ ³⁻	SO ₄ ²⁻
Average of blend batches	1.196	0.131	1.43	2.22	0.124	0.154
Blend concentrated to RAF uranium concentration	1.274	0.184	2.18	3.12	0.174	0.216
TBP HW#4 Flowsheet RAF	1.240	0.184	2.01	2.78	0.177	0.177

(a) Includes all H⁺; not titratable acid.

Since the installation of the new larger-throated jets and with continued blending operations, some reduction in the accumulation of sludge "rocks" in the slurry accumulator (described first in last month's report) has been noted. The attempt to remove the "rocks" by dissolution with water was not successful because an insufficient amount of water was used. Water dissolution may be tried again in the future if the accumulation does not continue to decrease.

Testing of the two new turbidimeters, operating on the principle of reflected light, during actual blending operations proved them to be entirely satisfactory. The failure of one instrument, after a short period of time, was traced to the widely varying voltage generated by the emergency generator. To prevent recurrence of this failure, the turbidimeters were disconnected from the emergency power generator circuit.

Tank Farm Construction Status

The approximate over-all tank farm construction status of the uranium removal facilities in the various areas is as follows:

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241-U	99.5%
241-C	99%
241-B-BX-BY	82%
241-T-TX	90%
Cross country pipe line vent station	99%.

The 241-CR control building and recovery vault were accepted from construction during the month, and the remainder of the facilities should be completed by about August 1, 1952. Preliminary operation of equipment, flushing, and tank calibrations are in progress.

221-U Plant - TBP Extraction

Operations in the TBP Plant during the past month were directed primarily toward discovering and correcting the cause of the excessive uranium loss in the RAW (RA Column waste) stream. Twenty-nine runs were completed with no serious equipment difficulties. Rates varied from 1.8 to 6.25 tons of uranium per day and RA Column pulse frequencies ranged from 40 to 93 cycles/min. After it became apparent that varying the pulse frequency and flow ratios would not produce an RAW with a uranium concentration approaching the 0.5% flowsheet uranium loss, various modifications were made in the original RA Column piping to minimize possible loss of pulse due to air pockets and produce more effective distribution of RAX. When the following modifications had been made on the spare column: (a) installation of an RAI distributor; (b) relocation of the RAX control valve from the operating gallery to a position adjacent to the column; (c) relocation of the RAW control valve to point closer to the column; and (d) installation of various air vents on the pulse leg and column; RA Column uranium losses as low as 0.4% were achieved at a 2-1/2 ton U/day rate. At 5 tons U/day, the early 3 to 30% RA Column uranium losses were reduced to about 1.5 to 2%. At the present time the necessary modifications required for the production line RA Columns are being determined by a series of elimination runs. As soon as the required modifications have been determined, the equipment changes will be made, and the "cold" shakedown program will begin. The RC Column continued to function satisfactorily, with uranium losses ranging typically from 0.2 to 0.5%. Table IV summarizes the runs completed during the month and lists the modifications completed prior to each run as well as the operating conditions and waste losses.

During shakedown and subsequent operation for column runs the following difficulties were encountered in the operation of the feed and waste evaporators: (a) the corroded shafts on the feed concentrator cooler agitators were replaced with stainless steel shafts; (b) all concentrator steam traps required resetting; and (c) plugged tubes in both the feed and waste evaporators were evidenced.

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The maximum capacity for the concentrators was determined in all cases by the maximum capacity of the feed pump or the valve in the feed line to the concentrator and in most cases was equivalent to a production rate of 9 to 10 tons U/day per processing line. The condensate decontamination factors obtained during shakedown, based on sodium decontamination, approached the 3.2×10^5 which has been estimated to be the D.F. required during normal plant operation. Tables V and VI respectively indicate the shakedown operating conditions and D.F.'s for the feed and waste concentrators.

The continuous pH monitor for the waste neutralizer was tested and put into service. However, the reliability of the results from this instrument still must be shown.

The following informal reports were issued during the month:

HW-24828 TBP Process Discussion Committee Meeting Minutes, by G. C. Oberg, dated June 24, 1952.

Cavitation in TBP Plant RA Columns with RAW and with RAX in the Pulse Leg, by G. Sege, dated July 16, 1952. (The calculations reported indicated the cavitation threshold to be higher than 95 cyc./min., the maximum frequency of the plant pulse generators.)

224-U Plant - UO₂ Conversion

Twelve and one-half lots (100 drums) of UO₃ were calcined in the 224-U Building pots during the period. Table VII summarizes the analyses of the UO₃ produced. All but approximately 7 drums of this UO₃ was produced from RCU which originally was Redox UNH solution but had been used for "cold" shakedown runs in the 221-U Building equipment. The remaining 7 drums were calcined from UNH received directly from Redox.

Considerable difficulty was encountered in the processing of the RCU. In addition to the foaming experienced in the final concentrator (which concentrates 60% UNH to 100% UNH) approximately 7% of the pots boiled over during calcination. To prevent the pot boil-overs which occurred between temperatures of 125 and 150°C., the starting furnace temperature was reduced from 625°C. to 400°C. When the pot charge temperature reached 160°C., the furnace temperature was increased to 625°C. and normal calcination was continued. Sixty per cent nitric acid was used as an anti-foam agent and 3 to 10 gallons were added to each pot in an attempt to eliminate the boil-overs. Although these two precautions (lower heat plus acid addition) greatly reduced the amount of foaming, pot boilovers were never entirely eliminated.

The foaming difficulties experienced with the RCU processed to date are believed to have been aggravated as a result of the previous history of the RCU solution. In order to provide storage space for continued 221-U Building operations, the RCU was repeatedly concentrated and continually recycled to the feed

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tank. In addition to this operation, which may have produced foaming agents by the decomposition of TBP, several high-uranium-content sump liquors containing building floor drainings were added to the RCU tank.

During the month approximately 7% of the 264 pots charged with RCU boiled over. About 50% of these boil-overs were mild (contamination confined to the top of the pot). Pot charges varied from 45 to 57 gallons in volume, and the pot feed ranged from 55 to 100% UNH solutions. The reduced heat at the beginning of the cycle increased the pot time cycle, which had been reduced to less than 8 hours when calcining Redox UNH, to 8-1/2 to 13 hours. These long time cycles coupled with the reduced pot charge of approximately 300 to 350 lb. of UO_3 slowed down production materially.

Indications of foaming in the large concentrators (concentrate from 6 to 60% UNH), as reported last month, have not recurred and the addition of an anti-foam agent to these concentrators has not been required.

In addition to the above-mentioned pot foaming the following operational and equipment difficulties were experienced in 224-U Building during the month: (a) freeze-up of 100% UNH solution in the dump valve of the final concentrator; (b) foaming in the final concentrator; (c) rupture of two bags in unloading system bag filter; and (d) failure (due to shearing of a pin) of the rotary valve located beneath the unloading bag filter.

Rotating Equipment

Replacement cast iron pumps with cast iron bearings were installed in the 241-WR-001 tanks to replace the pumps which had previously failed after inadvertent exposure to HNO_3 . Initial operation was satisfactory.

On July 20, RA Column pulse generator 19-8 developed a loud hammering and squealing while operating at 92 cycles/min. Inspection revealed insufficient oil in the motion box case with subsequent starvation of the lower thrust bearing on the worm gear. No visible damage was evident to the mechanism and operation was smooth after addition of oil to the proper level.

Tank X-19 (100% UNH tank) was inadvertently filled to the overflow which allowed solution to rise up around the shaft and coupling of the X-19 pump and resulted in failure of the mild steel coupling bolts. After installation of stainless steel coupling bolts and resetting of the pump clearances, operation was uneventful.

Instrumentation

All "hot" and "cold" flow meters in 221-U Building have been calibrated except the ROS rotameters on both "A" and "B" lines, and the ROF rotameter on the "A" line. The existing ROS rotameters are too large and are being replaced with two Redox prototype Schutte and Koerting, 0.3 gal./min. all stainless steel meters. A larger Hammel-Dahl valve is required on the "A" line ROF system.

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Larger plugs and seats have been installed in the B-1 and D-2 concentrator let-down valves (3/4 in. on B-1 and 1/2 in. on D-2) to give adequate flow out of the concentrator. The d/p cell on the D-2 concentrator feed line orifice meter system plugged and was replaced.

Process Chemistry

Sludge Removal, Blending, and Acidification. Plug-type valves in the slurry tank lines have been found lately to be almost inoperable. Their freezing was suspected to be the result of possible dissolution of the lubricant in the highly basic slurry. Three types of Nordstrom lubricants were therefore tested in the laboratory for their solubility in synthetic slurry. After 24 hours' immersion in an agitated sludge-supernate mixture at 25°C., the following weight loss results were obtained:

Nordstrom Lubricant	Black 147	White 147	147-S
Initial Wt., Grams	1.2948	0.8632	0.8943
Final Wt., Grams	1.2901	0.8499	0.8929
% Loss	0.36	1.54	2.34

From these data, it seems improbable that the difficulty can be attributed to the solubility of the lubricant in waste slurries.

Comparison of blended slurries has indicated no significant differences in beta and gamma extraction coefficients as a function of length of storage period following blending. This is clearly shown by the organic/aqueous distribution ratios listed below, which were calculated from analyses of aqueous and organic phases after equal-volume contacts between the slurry sample and RAX.

Four-year-old sludge blended in April, 1952:

$$\begin{aligned} E_B^{o/a} &= 1.31 \times 10^{-3} \\ E_G^{o/a} &= 1.50 \times 10^{-4} \end{aligned}$$

Four-year-old sludge blended July 1, 1952:

$$\begin{aligned} E_B^{o/a} &= 1.11 \times 10^{-3} \\ E_G^{o/a} &= 1.95 \times 10^{-4} \end{aligned}$$

RAF Preparation from Redox 3EU Solution. Four laboratory countercurrent batch runs (3 extraction, 1 scrub, 3 strip contacts) were made to determine the feasibility of using Redox 3EU solution having a gamma ratio (exclusive of U²³⁷) of 23 to 55 times natural uranium as feed to the U.R. Plant. The results of these runs are listed below:

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Run 1 TBP-HW No. 4 Flowsheet. RAF contains PO_4^{3-} and SO_4^{2-}

	U	B Ratio	G Ratio	
RAF	0.29 M	1.20	54.2	○
RCU	0.11 M	0.17	7.74	

Run 2 TBP-HW No. 4 Flowsheet. RAF contains no SO_4^{2-} or PO_4^{3-}

	U	B Ratio	G Ratio
RAF	0.26 M	1.18	60.6
RCU	0.12 M	0.14	2.19

Run 3 Modified Purex Flowsheet. RAX 12.5% TBP in hydrocarbon, RAS 2.0 M HNO_3 , RCU 0.01 M HNO_3

	U	B Ratio	G Ratio
RAF	0.28 M	0.62	23.4
RCU	0.13 M	<0.05	<0.5

Run 4 Purex Flowsheet. RAX 12.5% TBP in hydrocarbon, RAS 3.0 M HNO_3 , RAX 0.01 M HNO_3 . Flow ratios RAF:RAS:RAX:RCS = 100:50:700:700

	U	B Ratio	G Ratio
RAF	1.35 M	0.62	23.4
RCU	0.10 M	<0.05	1.09

These data indicate that 3EU solutions with gamma ratios higher than specifications can be successfully decontaminated in the TBP Plant by using a modified Purex-type flowsheet.

Solvent Extraction. Excessively high uranium losses (up to 30%) in the RAW stream have been shown to be the result of mechanical difficulties rather than inextractable uranium in the RAF, on the basis of single batch extraction contacts of high uranium RAW samples with new RAX. On equal volume contacts, the organic/aqueous uranium distribution ratios ($E_{\text{U}^{+4}}^{\text{O/a}}$) ranged from 3.4 to 4.9, and on organic/aqueous volume ratios of 2.5/1, the $E_{\text{U}^{+4}}^{\text{O/a}}$ rose to 7.7.

UNH Calcination and UO_3 Reactivity. Assistance to 224-U operations involved 24 laboratory calcination pot runs, a large number of small-scale calcinations, and 36 reactivity test runs on both laboratory and plant-produced UO_3 powder, comprising 144 samples for analysis. Three main approaches constitute the bulk of this laboratory investigation, viz., (1) the cause of foaming in calcining TBP UNH solutions, (2) its prevention, (3) the effect of the foaming agent(s) and anti-foam techniques on the UO_3 -to- UF_4 reactivity. Foaming in 100% UNH solution during small-scale (50 ml.) calcining was caused by TBP and DBP (1 g./l.), but not by MBP or Shell Spray Base. The minimum amount of TBP which will cause foaming was found to lie between 0.1 and 0.5 g./l. In small-scale experiments, anti-foam agents

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(such as heptadecanol) and HNO_3 (as little as 1 volume of 60% acid for 60 volumes of UNH solution) were found to be effective foam preventives. In the plant, however, the addition of these agents directly to the calcining pots has met with indifferent success. The volume ratio of 60% HNO_3 to UNH solution was increased to 1:9 with noticeable beneficial results, but even so has not been completely effective. In laboratory experiments, adding the acid prior to the final UNH concentration step (from 60% to 100% UNH) has been completely successful in eliminating the foaming during subsequent calcination. This method has not been proved out in the plant.

The reactivities (in laboratory hydrofluorination) of the most recent batches of TBP UO_3 have shown steady improvement to values up to 80 to 85% conversion to UF_4 . This is believed to be due partially to the HNO_3 which is added to control foaming and partially to the fact that the contamination accompanying the initial lot of material processed has been largely flushed out of the system.

Studies are currently under way to evaluate the effect of the water treatment (calcine, cool, add H_2O , and re-dry) proposed by personnel at K-25 for improving reactivity.

REDOX AND URANIUM RECOVERY DEVELOPMENT

Process Studies

Alterations to Uranium Recovery Process. Three methods of achieving additional decontamination have been partially demonstrated at this and other sites, and were evaluated tentatively from an economic viewpoint. The expedient of increasing the acidity of the feed to the RA Column from 3M to 4.5M and substituting NH_4NO_3 for nitric acid in the scrub is attractive in that additional decontamination is believed certain without adverse effects on uranium purity. However, the additional chemical and waste storage costs of operating in this manner would amount to about \$3,500,000 per year. Alternatives to this path include the construction of additional facilities to decontaminate further the concentrated uranyl nitrate from the solvent-extraction battery. One suggested process involves the batch precipitation and removal of copper sulfide for the removal of ruthenium, followed by passing the solution through a silica gel bed for the removal of zirconium-niobium activity (estimated construction cost approx. \$1 million). Another possibility lies in the construction of a second solvent-extraction cycle employing operating conditions similar to the Purex process, and making provisions for acid recovery, waste disposal, and solvent handling largely by modification of existing equipment (estimated construction cost approx. \$2.5 million). It is believed that either of the latter two processes could be operated for about \$250,000 per year. The first alternative could be incorporated with very little delay, while the latter two alternatives would require some delay for design and procurement of the necessary equipment. No decision has been reached pending an evaluation of actual plant performance.

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Purex Flowsheet. An addendum was issued to Purex Chemical Flowsheet HW#1 as HW-25039. This addendum recommends adopting the conventional plutonium cycle for the Purex Plant design basis, with minimum jumpers to be provided so that the later adoption of the reflux flowsheet is not altogether precluded.

Dissolver Studies. A decision has been made to design the Purex dissolvers so that two dissolvers will normally service the Purex plant (a third unit will be supplied as a spare), and that the dissolver deentrainment towers will also function as acid recovery units. Since these units will be of larger and different design than any currently in use, a test program involving the 321 Building and BiPO₄ plant dissolvers has been formulated and is in progress.

Slug Modifications. In answer to queries from the Pile Technology Unit, the effects of various slug modifications on separations processes were postulated and reported in Document HW-25040. It was predicted that the use of 63S aluminum jackets would have no effect on separations processing. The use of thin metallic sheaths between the uranium and aluminum jackets would probably have an adverse effect on dissolution time cycles, especially the postulated zirconium or niobium sheaths. The incorporation of various metals as uranium-metal alloys would probably not affect the dissolution cycles, but could overtax the solids separation equipment. Samples of the projected modified slugs will be dissolved as received to observe the magnitude of these and other effects.

Chemical Engineering Development

Purex Phase Disengagement Studies. An experimental program has been initiated for the purpose of developing disengaging sections for the hydrocarbon-diluent Purex IB Scrub, 2A, and 2B pulse columns, which would be safe from a slow-neutron chain reaction by virtue of their geometry. In the course of this work it is planned to test the effectiveness of various packings (e.g., Raschig rings and screens of various designs), inserted into the disengaging sections with a view to mechanically aiding deentrainment of the wrong liquid phase from the effluent streams. During the month twelve runs were made in 321 Building in a 3-in. glass pulse column, with 3-in.-diameter, vertical, unpacked phase disengaging sections. These runs were designed primarily as controls, with reference to which the effectiveness of the packings to be tested could be evaluated. The studies were made under the conventional-type 2A Column extraction section and 2B Column conditions of the Purex Chemical Flowsheet HW#1 (Documents HW-24764 and HW-25039), with plutonium and fission products omitted. The perforated plates employed in the column plate section were "dual-face" (stainless-steel plates undercoated with Kel-F NW-25) with 0.06-in.-diameter holes, 21% free area, spaced 1-in. apart. The organic phase was continuous in the 2A Column runs, the aqueous in the 2B runs. The specific conditions and results of each of these runs are presented in Table VIII. The highlights of the findings are as follows:

1. Entrainment was considerably more severe in the top effluent than in the bottom effluent, as indicated by the following ranges of values determined with disengaging section "equivalent lengths**" of 1.2 to 3 ft.:

*) Defined as disengaging section volume divided by the cross-sectional area of the plate section.

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Column	Top Disengagement Section		Bottom Disengagement Section	
	Holdup Time, Min.	Vol. % Entrainment	Holdup Time, Min.	Vol. % Entrainment
2A	2 to 13	0.04 to 2	0.5 to 2	<0.01 to 0.1
2B	1.1 to 5	0.1 to 1.5	2 to 12	<0.01 to 0.03

2. The severity of entrainment increased sharply with increasing frequency and volume velocity, as illustrated by the following typical 2A Column top disengagement section data:

Volume Velocity, GPH/Sq. Ft., Sum of Phases	Amplitude x Frequency In./Min.	Equivalent Length of Disengaging Section, Ft.*	Holdup Time, Min.	Vol. % Entrainment
1200	60	1.3	2	0.10
		3.0	6	0.10
1200	80	1.3	2	2
		3.0	5	0.3
600	80	1.3	6	0.09
		3.0	13	0.08

The following specification letter and informal report were issued during the month:

HW-25020 Revised Preliminary Specifications for Purex Plant Pulse Columns, by F. W. Woodfield and G. Sege, dated July 14, 1952. (These specifications involve the reduction of the over-all column cascade height from the previously-specified 39 ft. to 33 ft., with a special "penthouse"-type design to accommodate the top of a 40-ft. high 2A Column.)

HW-25105 Estimated Apparent Specific Gravities in Purex Pulse Columns, by G. Sege, dated July 22, 1952.

*) Defined as disengaging section volume divided by the cross-sectional area of the plate section.

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Mechanical Development

Centrifuge Baffle Development for the Hot Semiworks. Two types of baffles were evaluated in the 321 Building 26-inch bowl centrifuge to determine which design would produce optimum centrifugate clarity when operating under simulated Redox head-end conditions. The inside diameter of both baffles was 1 inch less than the diameter of the bowl overflow lip. Liquid passage was provided in the first baffle by eight 1/2-inch holes (4 on a circle 1-3/4 inches from the bowl wall and 4 on a circle 3 inches from the bowl wall), and in the second baffle by two 1 inch by 3 inch radial slots in the baffle, extending outward from the bowl wall. Three runs were made with the first baffle and two runs with the second baffle. Co-formed MnO₂ was prepared in "cold" dissolver solution by reduction of KMnO₄ by the standard Redox procedure (HW-21630 and HW-24221). Centrifugation was carried out at 1100 G with an average bowl residence time of either 10 or 20 minutes. All centrifugate samples were visually clear, but showed an improvement of clarity when filtered through a very fine sintered glass filter. The actual MnO₂ content of the centrifugate ranged from 0.003 g./l. to 0.041 g./l. Results from the two baffles were almost identical. The second baffle, containing the two slots, was recommended for the Hot Semiworks centrifuge because the design allows the centrifuge bowl to be cleaned and drained more readily. Details of these tests are tabulated in Table IX.

Bearing Development. Carbon filled fluorothene, fiber glass filled Teflon, and clear mica filled Teflon bearings have been evaluated in the bearing test machines using RAX (Spray Base - 12.5% TBP) as a lubricant. Running against Stellite journals carbon filled fluorothene and fiber glass filled Teflon had the lowest coefficients of friction while clear mica filled Teflon and carbon filled fluorothene had the greatest load carrying capacity.

When Teflon and fluorothene are used as bearing materials the diametral clearance must be increased to give good performance (clearances of 0.006 to 0.008 in. as compared to clearances of about 0.002 inch for a hard bearing material with a 1-inch shaft). The relatively high coefficients of thermal expansion for Teflon and fluorothene make these greater clearances necessary.

Samples of No. 7740 Pyrex glass tubing have been received from Corning Glass Company for possible use as a bearing. One bearing is being machined for test. A fiber glass filled Teflon journal is being fabricated for test with Teflon bearings now on hand to investigate the feasibility of using Teflon to Teflon bearings and journals.

Pulse Generator Development

A 3-in. diameter Teflon bellows with a 4-in. face to face dimension has operated, pulsing water, for a total of 4,614,324 cycles at a pressure of 14 to 18 lb./sq.in.g.a. The history of the bellows to date is shown below:

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<u>No. of Cycles in Operation</u>	<u>Frequency, Cycles/Min.</u>	<u>Stroke, In.</u>
172,364	41	1.37
655,000	100	1.37
<u>3,786,960</u>	100	1.50
Total 4,614,324		

A second test stand for 3-inch bellows has been installed. The cam on the second test stand will produce a fixed stroke of 1/2 inch.

Two 10-in. diameter Teflon bellows have been procured from excess and a work order has been issued to install them on the two push rods of the 16-inch column pulse generator. These bellows may be compressed 2 inches and extended 1 inch. It is planned to test the bellows at a 1.3-inch stroke which will give at least a 1 inch amplitude in the 16-inch column.

A piston ring test stand for evaluation of alternate piston and piston ring materials for pulse generators is being constructed. The first tests will be made with Teflon rings.

Pump Development

Small Pump Development. A model PP-1 Eco Engineering Co. pump has completed a life test. The pump is a semi-positive-displacement pump consisting of a stainless steel housing and double acting eccentric Teflon impellers which rub against the housing, rolling the solution through the pump. The stainless steel shaft turns in Teflon bearings and Teflon packing is used in the stuffing box as a seal on the shaft. The pump completed 140 hours of test, first with water and later with 10% nitric acid. The results of the test are summarized below:

1. Below 25 lb./sq.in.ga. discharge head, delivery was constant, but at high discharge pressures, above 25 lb./sq.in.ga., considerable pulsation occurred in the discharge line.
2. At a constant head the discharge rate is essentially proportional to the speed, between speeds of 600 and 300 rev./min.
3. Packing gland leakage could not entirely be stopped.
4. The discharge rate against a 110 ft. head at the beginning of the test was 7650 ml./min. and after 130 hours of operation was 5650 ml./min. This is equivalent to a 26% loss of capacity over a 140 hour operating period.
5. Wear on the Teflon bearings after 140 hours was negligible. Wear on the journals after 140 hours was as follows:

Outboard	0.0161 in.
Inboard	0.0062 in.
Center	0.0047 in.

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The eccentrics which drive the impellers are made in the form of a clip which fits over a flattened portion of the shaft. Wear on the groove in the eccentric and on the mating portion of the shaft was excessive. This wear was the probable cause for the reduced discharge rate at the end of the test.

6. It is concluded that the Eco pump might be suitable for intermittent use as a transfer pump, but would be unsuitable for use as a metering pump.

Materials Testing

A. Coatings

The following paint-type protective coatings were applied to mild steel coupons by the manufacturers' recommended methods and immersed for 14 days in 50% NaOH, 10% HNO₃, 60% HNO₃, hexone, CCl₄ containing 30% TBP, and deobase containing 12.5% TBP.

Amercoat 1577, an Amercoat Corporation product of unknown composition, failed in 60% HNO₃ within 3 hours, failed in 10% HNO₃ in 12 days, but resisted all other solutions.

Modified Phenoline #300, produced by the Carboline Co., resisted all solutions with the exception of 60% HNO₃.

Liquid Neoprene, a product of the Gates Engineering Co., was not attacked by 50% NaOH, resisted 10% HNO₃ for 10 days, but failed in the other solutions within several hours.

B. Decontamination Studies

Redox 2DW on Stainless Steel (347). Six coupons of Type 347 stainless steel were contaminated with Redox 2DW, dried under a heat lamp, and sprayed three times (500 ml. per wash) with one of the following solutions: water, 5% Versene Fe-3, 5% Versene (Regular), 5% Versene Acid containing 1-3/4% NaOH, 10% HNO₃, and 3% HF-20% HNO₃. The amount of contamination on the coupons varied from 38,000 to 200,000 c/m (beta plus gamma). The decontamination factors varied from 13 to 258, but are not considered significant for comparison purposes because of the large variation in the initial contamination on the coupons. The data are presented in Table X.

Effect of Drying Time on Decontamination. In order to evaluate the effect of drying time on decontamination, six identical coupons of type 347 stainless steel were contaminated with equal amounts of Redox 2DW, dried for varying periods under the same heat lamp and then sprayed with three successive 500 ml. water washes. The drying time was varied from 8 to 30 minutes. After three washes the amount of contamination remaining on the coupons was about equal, but there was a large difference in the residual contamination after only one wash. The decontamination factors ranged from 48 to 110, and no correlation appeared to exist between the D.F. and the drying time. The data from this test are presented in Table XI.

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C. Corrosion Studies

Studies defining the corrosion of SAE 1010 mild steel in Redox Waste solutions as outlined in Documents HW-24407 and HW-24442 are approximately 80% complete. Corrosion rates after 1000 hours immersion are listed in Table XIII. Rates in the vapor phase were found to exceed those in the liquid phase at all test pH's at 180° and 200°F., but in all cases the corrosion rates were less than 0.0003 in./mo.

Effect of HNO₃ Concentration on Corrosion by Cl⁻. Samples of 304 ELC stainless steel exposed to boiling 3M HNO₃ spiked with 1000, 3000, and 10,000 ppm Cl⁻ corroded at rates of less than 0.0004 in./month over a 200 hour exposure period.

Tests in boiling 5M and 8M HNO₃ have not been run since it was found that in these solutions most of the Cl⁻ is rapidly oxidized to NOCl. Tests are under way to determine whether Cl⁻ concentration can be maintained in these solutions at room temperature.

D. Radiation Exposure Tests

Samples of polythene, Teflon, and fluorothene were exposed to gamma radiation in the 105-F cooling basin to determine the suitability of these materials for fabricating dual-faced plates for the Purex process pulse columns.

After a total exposure of about 10⁷ roentgens, Teflon had become very brittle. The test was continued to a total estimated exposure of 1.5 x 10⁶ roentgens, at which time fluorothene was beginning to form thin cracks when bent. The only noticeable damage to the polythene was a relatively slight discoloration.

Potassium Permanganate. Acid solutions of KMnO₄ were exposed to gamma radiation in the 105-F cooling basin. The permanganate concentration decreased with increased radiation exposure as follows:

<u>Exposure, Roentgens</u>	<u>MnO₄⁻ Concentration M</u>	
	<u>Exposed</u>	<u>Unexposed</u>
0	0.012	0.012
1 x 10 ⁶	0.010	0.012
3 x 10 ⁶	0.009	0.011
2 x 10 ⁷	nil	0.008

Dissolver Studies

A series of dissolver studies, employing the 321 Building dissolver, have been initiated to obtain information on dissolver performance, capacity, and modified or new dissolving techniques. Reaction rates in the dissolver were determined by taking frequent samples during dissolution. For the first run 600 lb. of 8-inch bare slugs were charged to the dissolver. A first cut and a heel cut, each of 150 lb. U, were taken with 60% nitric acid. A second cut, of 300 lb. U, was tried

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with 45% nitric acid. The results of these dissolving studies are being correlated.

A condenser-absorber is being fabricated for insertion in the dissolver off-gas line to study absorption of the oxides of nitrogen.

321 Building Operations

Mechanical and chemical engineering development studies are continuing in 321 Building. These studies embrace the development and testing of pumps, instruments, and pulse generators. Dissolving studies in the B Cell dissolver are in progress, and the F Cell centrifuge has been used to study centrifuge baffle design. Uranium waste solutions from 321 Bldg., containing 800 pounds of uranium metal, were transferred by trailer truck to the 200 area tank farms and 4939 pounds of uranium metal as UO_3 powder have been prepared for shipment to Mallinckrodt.

Hot Semiworks

Construction of the Hot Semiworks is 99 per cent complete. All equipment has been received and installed except five transfer jets, one air operated three-way valve and one explosion proof agitator motor. All buildings except the Hot Process Building have been accepted for beneficial occupancy.

The Hot Semiworks Manual is 98 per cent complete in rough draft and 75 per cent complete in final copy. Part I, a description of the facilities, has been completed except for three subsections. This has been issued as Document HW-22955 and the remaining subsections will be added later.

Calibration has been completed for 15 tanks in the Solvent Handling Building and 5 tanks in Aqueous Make-Up Building, i.e., a total of 20 of the 64 tanks.

The five Roth submerged pumps were run in for two hours each. Seal leakage was checked and found excessive on two of the pumps, P-5 at 31.5 ml./hr. and P-12 at 63.0 ml./hr. The pumps will be run continuously in an attempt to reduce the seal water leakage rate to 15 ml./hr. The 20 Class I agitators in the Cells were run in for four hours each. Eight of these heated excessively at the Mechanical seal and will be slowly run in. No seal leakage test could be made as the graphitar to stellite seals were cooled by a continuous water purge. Flow rates through the 18 Hammel Dahl valves were checked. Six of these would not deliver the specified rates and the subcontractor has requested a factory representative to determine the fault. Functional tests on the sample jets revealed that fluid would not flow through the 1/2-in. tubing discharge line. This is attributed to a design error which will be corrected by installation of 1/2-in. discharge lines of Schedule 10 pipe. A leak test was performed on the east bank of the ventilation air exhaust filter after calking the cartridges. No leaks were found and the ventilation filters were accepted. A decanter for the hexone still condensate has been fabricated and installed in the Solvent Handling Building. Decant sight glasses have been installed on outlets to five tanks on the first floor of the Solvent Handling Building. Safety showers have been installed in the cells. An additional transfer

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jet has been installed on the waste accumulator, Tank TK-54, in A Cell to permit recycle of waste through the first cycle. The 45 foot telescoping Hi Lift elevator platform for B Cell maintenance is scheduled for delivery toward the end of September. Temporary scaffolding left by the contractor in B Cell has been procured for current use. Bayonet sampling equipment and the sample carrying truck have been procured. All spare parts lists have been prepared and many items (including Amercoat 1574 and Koncentrik fittings) have been made Stores items.

The design of B Cell shielding has been completed and fabrication of lead sections to surround the IA Column has been initiated. A 400-gal. tank for transporting Uranium Recovery Plant solutions has been procured and is being modified for this use. A PR can and jacket have been procured for product removal.

Under the present schedule, the construction contractor will leave the Hot Semiworks area on August 1. The area will become an exclusion area on August 4. Four-shift operation will start on August 4.

The following informal report was issued during the month:

HW-24953 Potential Hot Semiwork Redox Tests - Minutes of Hot Semi-
works Committee Meeting, July 3, 1952. By G. Sege, dated
July 7, 1952.

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TABLE I

RECENT 2D COLUMN RUNS
FLOWSHEET VARIATIONS

3/8-In. Mini (Miniature Mixer Settler). 8 Extraction and 4 Scrub Stages.

Run	Stream Composition	D.F. (a)			2DW Loss (a) (% U in 2DF)	Contamination in 2DW (b)		
		AT	B	G		Na	Fe	Al
1 Acid-deficient 2DS from Al_2O_3 dissolved in HNO_3	2DS 2.06 M ANN							
	-0.01 M HNO_3							
	0.05 M $\text{Fe}(\text{NH}_2\text{SO}_3)_2$	.4.4	51.0	61.9	12.6	340	0.8	260
	2DF 0.2 M NaOH							
2 Acid-deficient 2DS with 28% NH_3 soln.	2DS -0.2 M HNO_3							
	0.05 M $\text{Fe}(\text{NH}_2\text{SO}_3)_2$	12.0	49.5	72.3	0.60	115	86	600
	2DF 0.2 M NaOH							
3 No Fe in 2DS or 2DF	2DS -0.2 M HNO_3 (NaOH)	2.7	42.3	66.0	1.2	2300	<1	
	2DF 0.2 M NaOH							
4 No Fe in 2DS or 2DF	2DS Neutral							
	2DF 0.2 M NaOH	3.3	28.5	60.0	0.88			

Notes:

(a) These are the averages of 3 or 4 end stream samples taken after steady state had been reached.

(b) Parts per million parts of uranium metal.

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TABLE II

LABORATORY DISTILLATION OF ICW (HEXONE)
IN PLATE-FREE DISTILLATION COLUMN^(a)

Feed Rate, Ml./Min.	Distl. Rate Ml./Min.	Beta c/m/gal.	Beta D.F.	Gamma Mc/Gal.	Gamma D.F.	Diketone, %	MIBC ^(b) %	Mesityl Oxide, %	Feed Point	Run No.
---	---	1.9×10^7	---	6.7×10^2	---	0.046	0.017	0.079	---	Feed
25	27	4×10^4	4.8×10^2	<1	$>6.7 \times 10^2$	0.020	0.096	0.059	Bottom	1
41	45	$<2 \times 10^4$	$>9.5 \times 10^2$	<1	$>6.7 \times 10^2$	0.016	0.075	0.037	Bottom	2
25	30	$<1 \times 10^4$	$>1.9 \times 10^3$	<1	$>6.7 \times 10^2$	0.019	0.12	0.045	Bottom	3
35	39	$<1 \times 10^4$	$>1.9 \times 10^3$	<1	$>6.7 \times 10^2$	0.019	0.080	0.053	Bottom	4
31	41	$<1 \times 10^4$	$>1.9 \times 10^3$	1	$>6.7 \times 10^2$	0.016	0.048	0.037	Top	1-a
41	53	$<1 \times 10^4$	$>1.9 \times 10^3$	<1	$>6.7 \times 10^2$	0.018	0.090	0.01	Top	2-a
65	70	$<1 \times 10^4$	$>1.9 \times 10^3$	<1	$>6.7 \times 10^2$	0.014	0.120	0.069	Top	3-a
19	29	$<1 \times 10^4$	$>1.9 \times 10^3$	<1	$>6.7 \times 10^2$	0.017	0.072	0.051	Top	4-a

Notes: (a) Laboratory equipment consisted of a 500 cc. glass, round-bottom flask containing approximately 200 cc. of 5 wt. % aqueous NaOH, surmounted by an empty glass column approximately $1\frac{1}{2}$ in. in diameter and 6 in. tall.

(b) The analytical method is not specific for MIBC, and it has been shown that this material is largely non-volatile and thus not MIBC. It is believed to be an aldol condensation product of hexone, the formation of which is accelerated by distillation from a caustic heel, but not by distillation from a water heel.

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TABLE III

LABORATORY PREPARATION OF ACID-DEFICIENT ANN

<u>Run</u>	<u>Grams Alumina</u>	<u>Grams HNO₃</u>	<u>Grams Residue</u>	<u>% Residue</u>	<u>Time, Hours</u>	<u>Initial Conc. HNO₃, %</u>	<u>Final HNO₃ Conc., M</u>	<u>Final ANN Conc., M</u>
C	42.0	82.2	0.54	1.29	24	60	-0.89	1.38
D	42.0	90.4	3.42	8.10	22	27.2	-0.06	2.15
E	35.2	82.1	0.697	1.98	10	40	+0.097	2.02
F	35.5	82.1	0.93	2.62	7.5	42.8	-0.011	2.26
G	36.5	85.94	0.91	2.49	9.0	44.5	-0.086	2.4

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TABLE IV
TBP PLANT SOLVENT-EXTRACTION RUNS

Date	Run No.	Changes in Equipment Prior to Operation of the Run	Production Rate, Tons U/Day	Pulse Frequency Cycles/Min.		Average Waste Losses (% Feed Uranium)		Remarks
				RA	RC	RAW	RCW	
6/24/52	U-4A-1	Original design.	5.0	40	70	23.8	0.018	Pulse frequency investigation
6/25/52	U-4A-2	Original design.	5.0	60	70	5.93	0.18	Pulse frequency investigation
6/25/52	U-4A-3	Original design.	5.0	70	70	28.0	0.045	Pulse frequency investigation
6/25/52	U-4A-4	Original design.	5.0	70	60	18.8	0.18	Pulse frequency investigation
6/26/52	U-4A-5	Original design.	5.0	70	80	21.5	0.17	Pulse frequency investigation
6/27/52	U-4A-6	Original design	5.0	60	90	18.2	0.38	Pulse frequency investigation
6/27/52	U-4A-7	Original design	5.0	60	70	2.7	0.23	Pulse frequency investigation
6/30/52	U-3A	Removed RAX syphon breaker for straight run to column. Installed monometer to measure RA pulse amplitude.	5.0	55	75	29.7	1.48	
7/1/52	U-5B-1	Same changes as above	6.25	55	75	1.09	3.9	Maximum capacity at 110% RAX
7/1/52	U-7A	Installed sight glass in RAX line to spot air bubbles. In-line sampler for RAW installed and two vents put in high points of bottom disengaging section box. Installed saran piston overflow line on RA. Turned off RA static pressure bubbler.	2.5	55	75	30.6	0.24	

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TABLE IV (Cont'd)
TBP PLANT SOLVENT-EXTRACTION RUNS

Date	Run No.	Changes in Equipment Prior to Operation of the Run	Production Rate, Tons U/Day	Pulse Frequency Cycles/Min.		Average Waste Losses (% Feed Uranium)		Remarks
				RA	RC	RAW	RCW	
7/3/52	U-5B-2	Same as above. These equipment changes in addition to those for U-3A.	2.5	55	75	18.6	0.43	
7/5/52	U-5B-3	No further changes in equipment.	2.5	55	75	8.2	0.52	(125% RAX)
7/6/52	U-5B-4	This series made to study effect of	2.5	55	75	4.2	0.33	(175% RAX)
7/7/52	U-5B-5	varying RAX to RAF ratios.	2.5	55	75	4.9	--	(250% RAX)
7/8/52	U-5B-6	Emulsification test in RA.	1.8	95	75	17.0	0.35	Did <u>not</u> emulsify
7/13/52	U-7B-2	Installed vent below RA pulser piston. Installed vent on horizontal run of RA pulser leg. Installed vent on vertical run of RA pulser leg. Connected RAX vent line with sight glass to 387 return line.	2.5	58	75	21.1	--	
7/13/52	U-7B-3	RA emulsification run. No change from U-7B-2.	2.5	95	75	15.0	--	Did <u>not</u> emulsify
7/13/52	U-7B-4	Comparison with Run U-4A-4 which was made under same conditions without the mechanical changes.	5.0	70	75	9.8	--	
7/16/52	U-7B-5	Pickled RA Column with hot 60% HNO ₃ and used startup procedure calling for fill-up of column with aqueous rather than organic. All possible air bled off RAX line.	2.5	55	75	25	.27	
7/18/52	U-8B-1	Moved RAX control valve from operating gallery to RAX line in cell. Installed swing check downstream of control valve.	2.5	55	75	15.4	0.47	

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TABLE IV (Cont'd)
TBP PLANT SOLVENT-EXTRACTION RUNS

Date	Run No.	Changes in Equipment Prior to Operation of the Run	Production Rate, Tons U/Day	Pulse Frequency Cycles/Min.		Average Waste Losses (% Feed Uranium)		Remarks
				RA	RC	RAW	RCW	
7/19/52	U-8A-1	Modified spare RA Column. Added RAX distributor. Installed check valve in RAX line near column before run. No pulser vents installed.	2.5	55	75	13.2	0.67	
7/20/52	U-8A-2	No change from U-8A-1	2.5	92	75	1.58	2.76	One sample.
7/22/52	U-8A-3	Relocated RAX & RAW control valves in lines near column. Installed "soda straw" vent* plus additional pulser vents on RA system.	2.5	55	75	3.7	0.37	
7/22/52	U-8A-3A	No change from U-8A-3	2.5	70	75	0.4	0.52	
7/22/52	U-8A-4	No change from U-8A-3	5.0	55	75	2.74	0.28	115% RAX
7/23/52	U-8A-5	No change from U-8A-3	5.0	70	75	5.2	--	
7/24/52	U-8A-6	No change from U-8A-3 except RAW valve restored to original location.	5.0	55	75	3.4	--	115% RAX
7/25/52	U-8B-2	Same as U-8B-1 except for removal of check valve and relocation of RAW control valve near column.	5.0	55	75	7.7		
7/26/52	U-8B-3	Same as U-8B-2 except for installation of single point RAX distributor and relocation of RAX control valve near the column.	5.0	55	75	1.4	--	115% RAX

*) Located so as to remove any air trapped under RA pulser piston.

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TABLE V

TBP PLANT RAF FEED CONCENTRATOR PERFORMANCE

Run No.	Rate		Concentrate/ Feed Ratio	Tube Liquid Level		U D.F. Feed Condensate
	Feed, Gal./Min.	Equiv. Tons U		In.	%	
FC-6-1 (a)	19.3	5.07	0.64	67	37	—
(b)	20.1	5.28	0.71	90	50	8×10^3
(c)	20.1	5.28	0.70	117	64	—
(d)	20.1	5.28	0.73	152	84	$> 8.6 \times 10^5$
FC-7-1 (a)	36.3	9.55	0.64	68	37	66×10^4
(b)	36.3	9.55	0.61	93	51	9.4×10^4
(c)	36.0	9.46	0.59	115	63	1.3×10^5
(d)	35.2	9.25	0.55	152	84	—
FC-8-1 (a)	19.6	5.15	0.64	106	58	$> 8.9 \times 10^5$
(b)	29.1	7.65	0.71	109	60	1×10^6
(c)	43.0	11.30	0.65	109	60	$> 7.8 \times 10^5$

TABLE VI

TBP PLANT WASTE CONCENTRATOR PERFORMANCE

WC-8-1 (a)	38	8.26	Not Operable	60	33	Not Operable
(b)	38.2	8.30	0.52	89	49	$> 15 \times 10^5$
(c)	38.6	8.40	0.55	116	64	$> 1.9 \times 10^5$
(d)	38.6	8.40	0.57	156	86	$> 1.7 \times 10^5$
WC-9-1 (a)	23.0	5.0	0.54	104	57	2.2×10^4
(b)	32.5	7.06	0.54	112	62	$\sim 1.6 \times 10^5$
(c)	42.8	9.30	0.57	114	63	4.2×10^4
WC-9-2 (a)	14.0	3.03	0.66	121	67	5.4×10^4
(b)	22.6	4.89	0.57	118	65	—
(c)	34.3	7.43	0.45	118	65	1.4×10^4
(d)	42.3	9.18	0.50	117	64	—
WC-10-1 (a)	37.0	8.05	0.65	68	37	—
(b)	37.0	8.05	0.55	87	48	1.4×10^5
(c)	37.0	8.05	0.52	119	66	—
(d)	37.0	8.05	0.48	153	84	$> 1.4 \times 10^6$
WC-10-2** (a)	41.5	9.02	0.50	64	35	3.2×10^4
(b)	41.5	9.02	0.56	90	50	—
(c)	39.6	8.60	0.53	103	57	2.3×10^4
(d)	41.5	9.02	0.65	156	86	2.3×10^4

*Per cent of tube height, calculated from weight-factor instrument readings.

**This run was made in Section 9 equipment.

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TABLE VII
UO₃ ANALYSES BY LOTS

Analyses	007-T	008-T	67	68	69	70	71	72	73	74	75	76	Carload (Lots 007-T, 008-T, and 67 - 76)
UO ₃ , wt. %	98.0	97.8	98.1	97.8	98.0	98.0	97.9	98.4	98.2	97.8	98.9	97.8	
U ₃ O ₈ , wt. %	.08	.04	.11	.09	.15	.02	.14	.33	.38	.31	.016	.08	
H ₂ O, wt. %	.80	.89	.37	.32	.40	1.00	.71	.65	.43	.31	.47	.54	
HNO ₃ , wt. %	.84	.77	.66	.52	.67	.66	.68	.47	.52	.47	.41	.53	
Particle size % thru 80 mesh	99.2	99.4	99.4	99.5	99.5	99.5	99.3	96.8	97.5	96.4	99.3		99.8
Bulk density, g/cc.	2.04	1.94	2.10	2.08	1.96	1.88	1.87	1.83	1.87	1.94	2.07		1.93
Surface area, sq. meters/g.	1.1	1.2	1.2	1.5	1.6	1.6	1.30	2.10	1.97	2.19	1.83		1.64
Fission product beta, % natural uranium beta	10.8	11.3	4	6	<3	<3	9						<3
Fission product gamma, % natural uranium gamma	33.2	32.1	22	20	22	31	23.4						35
Pu, ppb. parts U	<5	<5	<5	<5	<5	<5	<5						<5
Impurities, ppm. parts U													
Na	800	500	1000	1000	1000	1000	1000	1000	1000	1000	1000		2000
P	200	200	200	200	100	200	200	100	100	100	100		200
Fe	299	311	311	318	318	624	482	760	656	690	690		672
Ni	50	50	50	50	50	50	50	50	50	50	100		100
Mo	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2		<2
Cr	20	20	100	50	200	160	50	10	10	100	200		50
W	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10		<10
Si	50	20	50	10	10	20	21	20	20	200	200		50
B	1	1	2	2	2	2	2	2	2	2	2		5
S	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100		<100
Al	1000	800	100	50	100	100	100	50	50	100	100		20
Isotopic ratio, % U-235	.64		.64	.64	.64	.64	.64	.64				.64	

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Run No.	Interface Position	Pulse			Volume Velocity, Gal./ (Hr.) (Sq. Ft.), Sum of Both Phases	L/V, Aq./Org. Volume Flow Ratio
		Ampl., In.	Freq., Crc./Min.	Ampl. x Freq., In./Min.		
3"-2A-D1a	Bottom	1.0	65 ± 5 (c)	65 ± 5 (c)	1220	4.2
3"-2A-D1b	Bottom	1.0	95 ± 5 (d)	95 ± 5 (d)	1220	4.2
3"-2A-D2	Bottom	1.0	80	80	1240	3.7
3"-2A-D3	Bottom	1.0	60	60	1170	4.1
3"-2A-D4	Bottom	1.0	70	70	1190	4.6
3"-2A-D5	Bottom	1.0	80	80	590	4.7
3"-2A-D6	Bottom	1.0	60	60	590	4.8

Run No.	Interface Position	Pulse			Volume Velocity, Gal./ (Hr.) (Sq. Ft.), Sum of Both Phases	L/V, Aq./Org. Volume Flow Ratio
		Ampl., In.	Freq., Crc./Min.	Ampl. x Freq., In./Min.		
3"-2B-D1a	Top	1.0	65 ± 5 (c)	65 ± 5 (c)	740	0.53
3"-2B-D1b	Top	1.0	75 ± 5 (d)	75 ± 5 (d)	740	0.53
3"-2B-D2	Top	1.0	70	70	740	0.52
3"-2B-D3	Top	1.0	60	60	720	0.52
3"-2B-D4	Top	1.0	50	50	720	0.53
3"-2B-D5	Top	1.0	50	50	330	0.62
3"-2B-D6	Top	1.0	70	70	300	0.54

- Notes: (a) Equipment: 3.0-in. i.d. precision bore glass column; 0.06-in. diameter holes, 21% free area; 1-in. plate sp through which the feed streams entered and in one of wh pipe and another sampler. The remainder of the top di the column. The part of the bottom disengaging section
- (b) The equivalent length of the disengagement section, L_D , section volume is taken as that between the effluent po
- (c) Cyclic local flooding occurred at and above the indicat
- (d) Complete Flooding occurred at and above the indicated f
- (e) Average results. Individual samples varied as much as
- (f) Volume per cent of aqueous phase entrained with the org
- (g) Volume per cent of organic phase entrained with the aqu

TABLE VIII

**PURAX 3-III. 2A & 2B PULSE COLUMN PHASE DISENGAGEMENT STUDIES
WITH UNPACKED VERTICAL DISENGAGING SECTIONS^(a)**

Flowsheet: Purax HW/L With Pu and P.P.'s omitted; Shell Deodorized Sugar Base diluent

2A EXTRACTION SECTION

Phase
Disengag-
ing Time,
Sec.

Top Phase Disengagement Section

Sec.	Holdup Time, Min.			% Entrainment ^(c)			L _D =
	L _D = 1.3 Ft. (b)	L _D = 1.8 Ft.	L _D = 2.0 Ft.	L _D = 1.3 Ft.	L _D = 1.8 Ft.	L _D = 2.0 Ft.	
36	(Flooding determination)						
36	(Flooding determination)						
36	2.2	3.1	5.0	1.65 ^(e)	0.43	0.29	0.
44	2.5	3.6	5.7	0.10	0.23	0.10	0.
44	2.7	3.9	6.2	0.36	0.31	0.18	0.
44	5.5	8.0	12.8	0.09	0.09	0.08	0.
44	5.6	8.2	13.1	0.06	0.06	0.04	0.

2B COLUMN

Phase
Disengag-
ing Time,
Sec.

Top Phase Disengagement Section

Sec.	Holdup Time, Min.			% Entrainment ^(c)			L _D =
	L _D = 0.6 Ft. (b)	L _D = 1.2 Ft.	L _D = 2.3 Ft.	L _D = 0.6 Ft.	L _D = 1.2 Ft.	L _D = 2.3 Ft.	
65	(Flooding determination)						
65	(Flooding determination)						
65	0.6	1.1	2.1	2.53	1.48	0.64	2.
65	0.6	1.1	2.2	0.69	0.72	0.36	2.
65	0.6	1.1	2.2	0.65	0.53	0.20	2.
65	1.3	2.6	5.0	0.25	0.20	0.10	4.
65	1.4	2.7	5.2	0.34	0.32	0.23	5.

ate section height: 3.9 ft.; perforated plates: dual face (stainless steel with the bottom surface coated with silver). The vertical disengaging sections each consisted of a 3-in. diameter 10-in. glass tee adjacent to the interface was maintained. Each tee was followed by a sampler which was followed by a 6-in. section of 3-in. glass pipe followed by a 10-in. glass tee through which the interface was maintained. Below the lower sampler consisted of a 12-in. length of 3-in. glass pipe through the bottom of which the interface was maintained.

is defined as the disengagement section volume divided by the cross-sectional area of the plate section. It is defined as that between the effluent point and the interface, whichever is nearest the interface.

frequency.

frequency.

x-fold.

is phase,

us phase.

Base diluent

Height (ft.)	Bottom Phase Disengagement Section					
	Holdup Time, Min.			% Entrainment (g)		
8 Ft. $L_D = 3.0$ Ft.	$L_D = 0.6$ Ft.	$L_D = 1.2$ Ft.	$L_D = 2.2$ Ft.	$L_D = 0.6$ Ft.	$L_D = 1.2$ Ft.	$L_D = 2.2$ Ft.
0.29	0.3	0.5	1.0	0.12	0.10	0.06
0.10	0.3	0.6	1.1	0.01	0.01	0.01
0.18	0.3	0.5	1.0	0.02	0.01	0.01
0.08	0.6	1.1	2.1	0.02	< 0.01	≤ 0.01
0.04	0.6	1.1	2.1	0.01	0.01	0.01

Height (ft.)	Bottom Phase Disengagement Section						
	Holdup Time, Min.			% Entrainment (g)			
1.2 Ft. $L_D = 2.3$ Ft.	$L_D = 1.2$ Ft.	$L_D = 1.8$ Ft.	$L_D = 2.9$ Ft.	$L_D = 1.2$ Ft.	$L_D = 1.8$ Ft.	$L_D = 2.9$ Ft.	
48	0.64	2.2	3.2	5.1	0.02	0.02	< 0.01
72	0.36	2.3	3.3	5.3	0.03	0.02	≤ 0.01
93	0.20	2.2	3.3	5.2	0.02	0.03	≤ 0.01
20	0.10	4.4	6.4	10.2	0.02	0.01	0.01
32	0.23	5.2	7.7	12.2	0.03	0.02	0.01

with the bottom surface coated with Kel-F HM-25), 3-in. glass tee adjacent to the plate section is followed by a 6-in. section of 3-in. glass glass tee through which the organic phase left through the bottom of which the aqueous phase exited.

area of the plate section. The disengagement at the interface, whichever is less.

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TABLE IX

CENTRIFUGATION OF MnO₂

Equipment: Bird, 26-inch bowl diameter, solid-bowl centrifuge, 1100 G at top speed of 1650 rev./min. Liquid bowl holdup 4.8 gal. Baffle was located in central horizontal plane of bowl and extended inward 1/2 inch beyond bowl overflow lip. The first baffle had eight 1/2 inch holes (4 on a circle 1 3/4 in. from bowl wall and 4 on a circle 3 in. from bowl wall), and the second baffle had two 1 inch x 3 inch radial slots extending outward from the bowl wall.

Solution: "Cold" dissolver solution was made 0.03M in KMnO₄, heated to 90°C., and sparged with air for 4 hours. Residual KMnO₄ was precipitated as MnO₂ with Cr(NO₃)₃. Typical composition of oxidized solution:

UNH	1100 g./l.
Na ₂ Cr ₂ O ₇	0.1M
HNO ₃	1.5M
MnO ₂	2.6 g./l. (precipitate)
Sp.Gr.	1.8 @ 50°C.

Procedure: Oxidizer solution was fed to the centrifuge at an average rate of 0.24 gal./min. and 0.48 gal./min. for 20 min. and 10 min. residence times, respectively.

Samples of the centrifugate were taken as desired.

Analytical: Clarity of the centrifugate was defined as per cent light transmission at 550 milli-micron wavelength. Clarity was determined for each sample as taken and again after filtration through a fine, sintered glass filter.

<u>Run</u>	<u>Baffle Design</u>	<u>Residence Time, Min.</u>	<u>Clarity as Sampled, %</u>	<u>Clarity After Filtration, %</u>	<u>MnO₂ in Sample, G./L.</u>
1	1/2 in. holes	20	15	20	---
1	"	20	22	24	---
2	"	20	24	26	0.003
3	"	10	21	28	0.041
3	"	10	24	28	---
4	1 x 3 in. slots	20	23	27	---
4	"	20	23	25	---
5	"	10	30	35	---

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TABLE X

DECONTAMINATION OF REDOX 2DW ON STAINLESS STEEL (347)
COMPARISON OF DECONTAMINANTS

	Decontaminant					
	Water	5% Versene Fe-3	5% Versene (Regular)	5% Versene Acid + 1 3/4% NaOH	10% HNO ₃	3% HF - 20% HNO ₃
Initial Contamination	67,000 c/m (B + G)	38,000 c/m (B + G)	51,000 c/m (B + G)	150,000 c/m (B + G)	20,600 c/m (B + G)	73,000 c/m (B + G)
After 1st Wash	2,000 c/m	25,000 c/m	9,000 c/m	47,000 c/m	4,000 c/m	9,000 c/m
After 2nd Wash	2,000 c/m	5,000 c/m	2,000 c/m	7,000 c/m	4,000 c/m	400 c/m
After 3rd Wash	1,000 c/m	3,000 c/m	700 c/m	600 c/m	800 c/m	---
D.F.	67	13	73	250	258	162

Note: The volume of each wash was 500 ml.

TABLE XI

DECONTAMINATION OF REDOX 2DW ON STAINLESS STEEL (347)
EFFECT OF DRYING TIME ON DECONTAMINATION

Sample	Drying Time	Initial Contam.	After 1st Wash	After 2nd Wash	After 3rd Wash	Decontamination Factor
1	8	65,000 c/m	2,000 c/m	1,900 c/m	1,800 c/m	61
2	10	70,000 c/m	3,000 c/m	2,500 c/m	2,200 c/m	48
3	15	72,000 c/m	58,000 c/m	1,700 c/m	1,400 c/m	110
4	20	72,000 c/m	20,000 c/m	3,000 c/m	2,000 c/m	57
5	25	70,000 c/m	70,000 c/m	1,800 c/m	1,800 c/m	66
6	30	56,000 c/m	28,000 c/m	2,400 c/m	1,800 c/m	53

Note: Each wash consisted of 500 ml. water.

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TABLE XII

CORROSION RATES OF SAE 1010 MILD STEEL IN REDOX WASTE STORAGE SOLUTIONS

(1000 hours exposure)

pH of solution: Temp: <u>180°F.</u>	Corrosion Rate, Inches/Month		
	11	12	13
Liquid	Gain Gain	1.1×10^{-5} 3.5×10^{-5}	Gain Gain
Interface	0.2×10^{-5} 0.5×10^{-5}	9.9×10^{-5} 1.5×10^{-5}	1.1×10^{-5} Gain
Vapor	23.7×10^{-5} 18.9×10^{-5}	20.8×10^{-5} 12.7×10^{-5}	5.7×10^{-5} 5.9×10^{-5}
Temp: <u>200°F.</u>			
Liquid	0.5×10^{-5} Gain	2.2×10^{-5} 3.9×10^{-5}	Gain Nil
Interface	1.0×10^{-5} 2.7×10^{-5}	2.6×10^{-5} 1.0×10^{-5}	Gain Gain
Vapor	28.1×10^{-5} 30.3×10^{-5}	24.9×10^{-5} 21.8×10^{-5}	13.7×10^{-5} 14.9×10^{-5}
Temp: <u>220°F. (Boiling)</u>			
Liquid	Gain 0.3×10^{-5}	Incomplete	Incomplete
Interface	2.0×10^{-5} 2.8×10^{-5}		
Vapor	13.2×10^{-5} 18.1×10^{-5}		

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