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PUREX PULSE-COLUMN STUDIES WITH UNIRRADIATED URANIUM

(Development of Specifications for the O.R.N.L. Pilot Plant)

CLASSIFICATION CANCELLED

DATE 3-28-60

For The Atomic Energy Commission

H. R. Canale

Chief, Declassification Branch *ml*

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February 20, 1951

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INTRODUCTION

Oak Ridge National Laboratory personnel requested the Hanford Works Chemical Development Section to provide pulse column specifications for the O.R.N.L. Purex pilot plant. To obtain operating and performance data using pulse columns to process Purex type feeds, a series of 19 IA simple extraction, and IC Column studies was carried out in the Demonstration Unit, 3-inch i.d. pulse column during the period September 23 to 25, 1950 and tentative O.R.N.L. pilot plant IA-IB-IC cascade pulse column battery specifications were developed. This report will summarize the data obtained from the Purex runs, and present the pilot plant specifications (Table IV) which were estimated from these Purex runs.

OBJECTIVE

The primary objective of the studies was to evaluate Purex IA and IC-type pulse column operation, employing a "standard cartridge" (perforated plates, 0.125-in. diam. holes, 2-in. face to face plate spacing, 23% open), at O.R.N.L. Tentative Purex Process Flowsheet (EW-18854) conditions to develop specifications for a Purex pilot-plant IA-IB-IC cascade pulse column battery to be constructed in the O.R.N.L. 205 Building pilot plant cells.

Secondary objectives were to obtain qualitative comparison of pulse column operation employing 30 vol.% TBP in hydrocarbon diluent solvent with operations carried out under TBP Waste Metal Recovery flowsheet conditions employing 12.5 vol.% TBP in hydrocarbon diluent solvent, and to determine the range of optimum throughput, pulse amplitude and frequency for the Purex IA and IC Columns.

Since the objectives of these studies were limited to the above, information relevant to other cartridge geometries, scale-up factor, and flowsheet variables was not obtained.

SUMMARY AND CONCLUSIONS

Nineteen IA Simple Extraction Section and IC Column runs were carried in 321 Building Demonstration Unit equipment (data in Tables I, II). No unexpected operating difficulties were encountered during these studies and evaluation of data from these runs led to the suggested O.R.N.L. pilot plant pulse column specifications presented in Table IV and Fig. 1. Highlights of information gained from these studies follow:

1. Detailed pilot plant specifications developed from these runs are presented in Table IV and Figure 1. The 27 ft. of headroom available in O.R.N.L. 205 Bldg. cells should be adequate with less than 5 ft. of the IC Column inserted in a pit. The overall column lengths and cascade height are approximately 50% of those required for packed columns (Table III) and are as follows:

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	Column Lengths			Overall Cascade Height IA Top to IC Bottom Ft.
	"Packed" Ft.	Unpacked Ft.	Total Ft.	
IA	16	5	21	
IB	16	5	21	
IC	12	4.5	16.5	30.7

2. The "standard cartridge" (0.125-in. holes, 2-in. face to face spacing, 23% open), developed in TBP Waste Metal Recovery studies (HW-19170), gave satisfactory performance under Purex flowsheet conditions and should prove adequate for use in Purex studies.
3. IA Simple Extraction Section run data (Table I) indicate that optimum uranium waste losses may be attained from 1000 to 1500 gal./hr. (sq. ft.), sum of both phases, at a pulse amplitude of 1 inch, and a pulse frequency of 70 cycles/min. No flooding or instability was experienced at the maximum rate (1560 gal./hr. (sq. ft.)) tested.
4. IC run data (Table II) indicate:
 - a. Optimum pulse frequency range = 70 to 95 cycles/min.
 - b. Optimum pulse amplitude = 1/2 inch.
 - c. optimum volume velocity range = 400 to 800 gal./hr. (sq. ft.), sum of both phases.
 - d. two-fold lower waste losses to ICW may be attained by increasing H_2SO_4 in ICX from 0.001 M to 0.01 M.
 - e. a maximum flooding capacity of 1200 gal./hr. (sq. ft.) was found at amplitudes of 1 inch and 0.25 inch, but the best extraction performance was observed at 0.5 inch amplitude for which the maximum flooding capacity was 900 gal./hr. per (sq. ft.).

DISCUSSION

Equipment and Operating Procedure: The 321 Building Demonstration Unit, 3-inch i.d. precision bore glass pulse column (HW-19170 Fig. 2) with an effective "packed height" of 9.11 ft. and its auxiliary pumps and instruments was used for the Purex scouting studies. The organic feed (IAX or ICF) was pulsed by the alternate compression and expansion of from one to six 4.5-in. i.d. stainless-steel bellows operating in parallel to give a pulse movement of from 0.25 to 1.5 inches in the 3-inch column. Aqueous feed (IAFS) was prepared by adjusting concentrated RCU from Waste Metal Recovery runs to Purex IAFS conditions while IAX was prepared from Scale-Up RAX (12.5 vol.% TBP, dilute RC H_2SO_4 less than 0.002) by addition

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of new TBP to give 30 vol.% TBP in Shell Deodorized Spray Base. IAX was washed with 1/3 volume 2% Na_2SO_4 followed by a 1/3 volume wash of 2% HNO_3 before use. ICF was prepared by butting IAU from IA runs to ICF flowsheet composition with additional IAX.

H.E.T.S. and H.T.U.: No equilibrium data were available for calculation of H.E.T.S. and H.T.U. values for these runs and uranium waste losses were used as a design criterion for the development of pilot plant specifications. The waste loss variation with amplitude and frequency in the IA Column (Table I) indicated that optimum operating conditions in the "standard cartridge" are approximately the same for the Purex flowsheet as for the RA Column operating under TBP Waste Metal Recovery flowsheet conditions. For both processes the range of optimum pulse conditions for the IA or RA column are at an amplitude times frequency product of 50 to 70 inches/minute, and at a volume velocity of 1000 to 1500 gal./hr. (sq.ft.), sum of both phases. The optimum waste losses and capacity of the Purex IC Column (Table II) are analogous to those obtained in the TBP RC Column with an amplitude times frequency product of 40 to 45 inches per minute with an amplitude of 0.5 inch giving the lowest waste losses over a volume velocity range of 400 to 800 gal./hr. (sq.ft.).

Flooding: No instability or flooding was experienced under the run conditions tested in IA runs. In the IC Column the maximum flooding capacity was found to be 1200 gal./hr. (sq.ft.) (runs 3.00-8 and 12a-CU (PRX)) as compared with a maximum flooding capacity of 2000 gal./hr. (sq.ft.) in the Waste Metal Recovery RC Column. The flooding experienced in the Purex IC Column was characterized by considerable emulsification and entrainment of ICF with the ICU stream. All floods originated in the lower 1/3 of the column.

Basis for Specifications:

Column Diameter: A minimum column diameter of 2 inches is considered to be as small as practicable for applicability to scale-up studies. To operate near the optimum range of flowrates the pilot plant should process 50 to 150 kg. of uranium per day. At the 100 kg./day processing rate the IA-IB-IC throughputs will be as follows:

Column	"Packed" Section	Pipe Size I.P.S. In.	Vol. Vel. Gal./hr. (Sq.ft.)	Pulse Conditions Amplitude	Freq.
IA	Top (Scrub)	2	590	1	50 to 70
	Lower (Extr.)	2	740	1	
IB	Top (Extr.)	2.5	540	0.7	40 to 70
	Lower (Scrub)	2	250	1	
IC	"Packed"	4	420	0.5	70 to 90

Packed Length: Data from the IA runs (Table I) indicated that 8-ft. of "packed" extraction section should consistently give uranium waste losses of less than 0.1% to the IAW, and IC run data (Table II) indicated that uranium losses of less than 0.5% to the ICW might be attained with 12 ft.

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of "packed" section. A IB Column scrub section length approximately equal to the IA Column extraction section length was assumed, while hydraulic and headroom considerations dictated the length of the IB extraction section.

Phase Disengagement and Influent Introduction: Provision of enlarged top-end sections was recommended on the basis of adequacy of the enlarged top-end section provided by the 3-inch Demonstration Unit Column in the IA and IC Purex runs. Bottom-end phase disengagement under conditions found operable in IA and IC runs indicated that provision of a 1.5 ft. "unpacked" section below the bottom plate should be sufficient to permit phase disengagement. Simple organic and aqueous feed introduction points consisting of single pipes discharging along the column axis were considered adequate for the small diameter pulse columns specified for the pilot plant.

Cascade Heights: The relative elevations of the IA, IB, and IC organic overflow points indicated in Figure 1 were based on the IA and IB organic overflow specific gravity being 0.8 and the specific gravity of the IB and IC Column contents being 1.1 with the IB and the IC interface at the overflow. An additional 2 ft. of H_2O head was provided to permit flexibility of operation if different flowsheets are employed.

Applicability of Data: A general evaluation of the Purex IA and IC runs made in Demonstration Unit equipment and comparison of the results obtained in these studies with results obtained in TBP Waste Metal Recovery RA and RC studies indicates that reasonably firm design data for pilot plant size Purex IA and IC Columns are available as a result of these runs. Some improvement in design of the IC Column to improve capacity is indicated as desirable and IB Column operating data are essential for firming up design specifications but the column design specified in Table IV and Fig. 1 should give adequate performance for evaluation of Purex flowsheets in the O.R.N.L. pilot plant.

TABLE I

PULSE COLUMN STUDIES: 3-INCH IA PUREX SIMPLE EXTRACTION COLUMN

Plates: $5/4$ pierced plates spaced 2.0-in. apart to give an effective "packed height" of 9.11 ft. Plates pierced with 0.125-in. diam. holes allowing approximately 23% perforated area.

Organic Phase Distributor: Single 1/2-in. IPS stainless-steel pipe discharging up, approx. 7-1/2 in. below the bottom plate in a 22-in. long by 3-in. i.d. bottom disengaging section.

Pulse: Organic feed (IAI) pulsed.

Compositions:

IAI: 31.6 (nominal 30) vol.% TBP in "Shell" Deodorized Spray base, prepared by adding new TBP to 12.5 vol.% RAI then washing with a 1/6 vol. 5% Na_2CO_3 wash, and a 1/6 vol. 2% $NaNO_3$ wash.

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IAPS: O.R.N.L. Tentative Purex Flowsheet, (HW-18854), 0.805 M UNE, 2.40 M HNO₃.

Volume Flow Ratio: IAPS:IAX = 1:2

Run No.	Pulse Amplitude In. (a)	Pulse Frequency Cycles/Min.	Volume Velocity (b)	% U Lost in IAW
3.00"-6-AU (PRX)	1.5	35	980	.009
3.00"-5-AU (PRX)	1.5	35	1510	.021
3.00"-4-AU (PRX)	1.0	70	960	.0004
3.00"-1-AU (PRX)	1.0	50	1560	.011
	1.0	70	1560	.003
3.00"-3-AU (PRX)	0.5	70	980	.021
3.00"-2-AU (PRX)	0.5	70	1500	.041

a) Pulse amplitude = pulse movement in the column, inches.

b) Volume Velocity = gal./ (hr.) (sq.ft.), sum of both phases.

TABLE II

PULSE COLUMN STUDIES: 3-INCH IC PUREX COLUMN

Plates: Same as described for 3-inch IA pierced plate column studies.

Organic Phase Distributor: Same as described for 3-inch IA pierced plate column studies.

Pulse: Organic feed (ICF) pulsed.

Compositions:

ICF: O.R.N.L. Tentative Purex Flowsheet (HW-18854), 0.32 M UN, 0.025 M HNO₃. IAU from runs 3.00-1 through 6-AU (PRX) adjusted to 0.32 M UN by addition of IAX. Solvent phase 31.6 (nominal 30) vol.% TBP in "Shell" Deodorized Spray Base.

ICX: 0.001 M H₂SO₄ in water, except runs 10c and 10d which used 0.01 M H₂SO₄ as ICX.

Volume Flow Ratio: ICX:ICF = 1.5/1.0, except runs 10b and 10c, which operated at ICX:ICF = 2.0/1.0

Run No.	Pulse Amplitude In. (a)	Pulse Frequency Cycles/Min.	Volume Velocity (b)	% U Lost In ICW
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Studies at O.R.N.L. Tentative Purex Flowsheet Conditions:

3.00"-6-CU (PRX)	1.0	40	400	5.0
-7-	1.0	40	800	3.3

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Run No.	Pulse Amplitude In. (a)	Pulse Frequency Cycles/Min.	Volume Velocity (b)	% U lost in ICW
Studies at O.R.N.L. Tentative Purex Flowsheet Conditions (Cont.)				
3.00"-8-CU	1.0	40	1000	1.1
-11-	1.0	46	800	0.54
-4-	0.5	60	800	8.7
-5-	0.5	60	1000	3.9
-10-	0.5	85	800	.43
-1-	0.5	90	400	.63
-9-	0.25	140	800	.87

Studies at O.R.N.L. Tentative Purex Flowsheet conditions modified to 0.01 M H_2SO_4 in ICW.

3.00"-10d-CU (PRX)	0.5	85	800	.29
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Studies at O.R.N.L. Tentative Purex Flowsheet conditions modified to ICX:ICF = 2.0/1.0

3.00"-10a-CU (PRX)	0.5	85	800	0.27
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Studies at O.R.N.L. Tentative Purex Flowsheet conditions modified to ICX:ICF = 2.0/1.0, and 0.01 M H_2SO_4 in ICW.

3.00"-10c-CU (PRX)	0.5	85	800	0.13
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Run No.	Pulse Amplitude In. (a)	Pulse Frequency Cycles/Min.	Volume Velocity (b)
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Flooding determinations at O.R.N.L. Tentative Purex Flowsheet conditions.

3.00"-8-CU (PRX)	1.0	40	1200 ± 150
-11-	1.0	50	800 ± 50
-13-	0.5	80	900 ± 100
-2-	0.5	90	800 ± 100 (c)
-10-	0.5	110(Emuls.) (d)	800 ± 100
-12a-	0.25	100	1200 ± 200
-12b-	0.25	140	1000 ± 100
-9-	0.25	160(Emuls.) (d)	800 ± 100

Flooding determinations at O.R.N.L. Tentative Purex Flowsheet conditions modified to ICX:ICF = 2.0/1.0.

3.00"-10b-CU (PRX)	0.5	85	800 ± 50
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*Note: Run 3.00"-3-CU (PRX) scheduled at a = 0.5, f = 90, vol. vel. = 1200 not made due to flooding experienced in Run 2.

a) Pulse amplitude = pulse movement in column, inches.

b) Volume velocity = gal./(hr.)(sq.ft.), sum of both phases. Rates given are nominal for IC runs and may differ from actual rates by ± 10%.

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- c) Two attempts to operate this run resulted in flooding when frequency was increased to 90 cyc./min. although a frequency of 110 cyc./min. was attained by a slow approach, ca. 1 cyc./min.², at 800 gal./(hr.)(sq.ft.)
- d) Emulsification frequency determined as the frequency at which emulsification of the countercurrent phases resulted in flooding.

TABLE III

ESTIMATED PACKED COLUMN DIMENSIONS FOR PUREX
STUDIES EMPLOYING 30 VOL. PERCENT TBP IN HYDROCARBON DILUENT^(a)

Column	I.D. In.	Packed Height Ft.	Volume Velocity Gal./(Hr.)(Sq.Ft.) ^(b)	Number of Stages Required	H.E.T.S. Ft.	Total Flooding Capacity Gal./(Hr.)(Sq.Ft.) ^(c)
IA	3	Scrub 20	140	7	2.9	610
		Extr. 20	175	6	3.3	
IB	3	Extr. 28	175	7	4(ave.)	320
		Scrub 13.5	58.5	5	2.7	
IBS	2	15	82.4	8	1.9	
IC	4	22	205	8	2.7	310
ID	3	Scrub 20	140	4	-	610
		Extr. 20	175	5	-	
IE	4	22	164	8	-	310

Notes to Table III:

- a) Table is copy of an informal, typed memo supplied by O.R.N.L. personnel.
- b) Sum of both phases in the indicated section.
- c) Based on flooding, in 1/4 by 1/4-in. Raschig rings in a 1-inch diameter glass column.

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TABLE IV

RECOMMENDED COLUMN SPECIFICATIONS FOR A PUREX FIRST CYCLE PULSER
COLUMN PILOT PLANT

	Sections		Plates ^(d)				
	Length (Feet)	Diam. (In.)	No.	Spacing (In.)	Thickness (In.)	Hole Diam. (In.)	% Open
<u>IA</u>							
Top (a)	3	4	-	-	-	-	-
Scrub Sect.	8	2	47	2	0.05	0.125	23
Mid-Feed Point (b)	0.5	2	-	-	-	-	-
Extr. Sect.	8	2	47	2	0.05	0.125	23
Bottom Sect. (c)	1.5	2	-	-	-	-	-
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Overall	21.0		94				
<u>IB</u>							
Top (a)	3	5	-	-	-	-	-
Extr. Sect.	8	2.5	47	2	0.05	0.125	23
Mid-Feed Point (b)	0.5	2.5	-	-	-	-	-
Scrub Sect.	8	2	47	2	0.05	0.125	23
Bottom Sect. (c)	1.5	2	-	-	-	-	-
<hr/>							
Overall	21.0		94				
<u>IC</u>							
Top (a)	3	8	-	-	-	-	-
"Packed" Sect.	12	4	71	2	0.05	0.125	23
Bottom Sect. (c)	1.5	4	-	-	-	-	-
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Overall	16.5		71				

Notes to Table

- (a) Top sections of approximately 4x column cross section and of the specified length should be provided to permit phase disengagement and reasonable sensitivity to interface level variations in the top sections. To gain headroom, these sections may be shortened if designed to allow the same organic holdup time provided in the specifications table, but in this case an additional 6 to 8 inch length of unpacked top column section of the same diameter as the "packed" top column section, in which the interface may be maintained, should be provided.
- (b) These sections may be spool pieces and should be unfilled except for the single feed line entering from the side and pointing up.

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- (c) Same as (b).
- (d) The number of plates is arbitrarily determined by assuming plates are 0.05 inch thick. Thus, for an 8 foot section 47 plates $\times$ 0.05 in./plate = 46 spaces $\times$ 2 in./space = 94.4 inches.

General Notes

Support Rods. Single 1/4 in. diameter stainless-steel rods of proper length to allow for plates, spacers, rod support and locknuts should be used.

Spacers. One-eighth inch I.P.S. stainless-steel pipe has been used successfully to make spacers for 3 inch and 16 inch columns.

Rod Support. A standard pipe flange with a single spoke may be used to support rods. This support may be installed to support the rod from below or from above.

Middle Feed. A single pipe introducing IAF or IBF upward along the column axis at approximately the center of the "unpacked" middle feed section should be adequate. This feed line should not occupy more than 30 to 40% of the column cross section (i.e., 1/2 in. I.P.S. pipe in a 2 in. I.P.S. column would be satisfactory).

Bottom Organic Feed. A single pipe discharging up approximately 6 inches below the bottom plate is recommended. This pipe should be as large as possible, to minimize acceleration pressure drop, consistent with maintenance of the maximum open area for phase disengagement.

Top Aqueous Feed. A single pipe discharging down approximately 4 inches above the top plate.

Interface Location. Six to ten inches above the top plate.

Pulse Conditions.

	<u>Ampl.</u> <u>(Inches)</u>	<u>Frequency Range</u> <u>(Cycles/Min.)</u>
1A	1	50 to 70
1B (Scrub)	1	40 to 70
(Extr.)	0.6	40 to 70
1C	0.5	70 to 95

Overflow Location. Six inches below column top. Since flow is pulsed it may be desirable to design the downcomer oversize to prevent aspiration.

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FIG.-1

Figure 1

ROUGH SKETCH OF IA-IB-IC CASCADE PUREX UNIT

NOTES

TOP DISENGAGING SECTION:

- (a) Top aqueous stream introduction by single pipe below interface.
- (b) First part of organic effluent line must be large enough to prevent aspiration.

MIDDLE FEED SECTION:

- (c) IA - Same diam. as column.
- (d) IB - Same diam. as extraction section.
- (e) IA+IB - Feed introduction: single pipe in center of column pointing upward. No plates in section.
- (f) Entering feed lines should be large enough to prevent excessive loss of head.

BOTTOM DISENGAGING SECTION:

- (g) Entering organic distributor: single pipe pointing upward.
- (h) 1'-6" section based on TBP waste metal recovery indications.

PLATES:

- (i) 0.05-in. thick, 0.125-in. diam. holes on 1/4-in. triangular spacing, not more than 0.01-in. clearance between wall and plate edge, 25% open area, plates supported by 1/4 inch rod through center, and 1/8-in. IPS pipe for spacers, spacing is 2" between plates (face to face).

GENERAL STATEMENTS:

- (j) Column elevations calculated on basis of worst conditions plus safety factor to allow for flexibility of conditions (high feed conc., etc.).
- (k) Instrumentation--can be based on static pressure at base of column. Bubble type recommended.
- (l) Location of ICU pulse generator should be as close to bottom as possible to insure positive pressure on piston.

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