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HW-19023

Technology - Hanford Processes

**SCOUTING STUDIES IN A 5.05-INCH
DIAMETER REDOX PULSE COLUMN**

by

W. S. Figg and J. G. Bradley
Chemical Development Section
Separations Technology Division

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-3-

HW-19023

SCOUTING STUDIES IN A 5.05-INCH
DIAMETER REDOX PULSE COLUMN

INTRODUCTION

In September of 1949, development and engineering studies leading to firm specifications (1) for a series of stainless-steel, Raschig-ring packed, liquid-liquid contactors for Redox Plant No. 1 were completed and detailed architectural and equipment design work started by the Kellex Corporation. Throughout the development program leading to this stage, architectural economy, among other considerations, had dictated that contactor lengths be held to a minimum.

Chemical Research Section studies (2), carried out in a 1-inch diameter glass column, indicated that additional savings in column length might be realized if pulse columns, originally described by W. J. D. Van Dijk (3), could be employed as Redox production plant contactors. Although the studies reported by the Chemical Research Section (2) looked promising, no data were available to demonstrate the performance of plant-size columns.

Since a second Redox Plant was then planned, the Chemical Development Section carried out a series of pilot-plant scouting studies in a 5-inch diameter stainless-steel pulse column to obtain preliminary extraction efficiency data for the IA, IB, and IC contactors. These studies were followed by the TBP Waste Metal Recovery process studies (4), in which the principles of pulse-column design and effects of operating variables were investigated more extensively.

OBJECTIVES

The objectives of these pulse column scouting studies were:

1. To scout the extraction performance of a 5-inch diameter pulse column (approximately full Redox plant 1A Column diameter), operating as a 1A Simple Extraction Section, a 1B Simple Scrub Section, and a 1C Column.
2. To make only a very limited number of runs (24) simulating each column to determine the flooding and extraction performance for comparison

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with 1-inch pulse column and plant-scale packed column performance.

3. To evaluate performance using only one column geometry arbitrarily selected on the basis of Chemical Research Section studies.

The scope of the pulse column program, discussed in this report, did not include development of an adequate pulse generating mechanism, determination of optimum contactor specifications, complete evaluation of scale-up factors, and a study of column rangeability with respect to capacity and flowsheet variables.

SUMMARY AND CONCLUSIONS

All studies described in this report were carried out in the 5.05-inch i.d. column (Fig. 5), using unirradiated uranium feeds adjusted to Redox No. 1 Flowsheet (7) conditions with the following modifications: a) no dichromate in 1A feeds; b) no ferrous sulfamate in 1B feeds; c) no nitric acid in 1CX. The hexone solvent stream (1AX, 1BS, or 1CF) was pulsed at amplitudes in the column from 0.1 to 0.45 in. (defined in Fig. 4), and frequencies from 50 to 200 cycles/min. The plate cartridge design chosen on the basis of Chemical Research Section studies (2) was:

Effective "packed height"	4.72 ft.
Number of plates	28
Spacing of plates (face to face)	2 in.
Plate hole diam.	0.04 in.
Plate thickness	0.025 in.
Perforated area	22 %

Complete data for these runs are summarized in Tables I, II, and III and are plotted in Figs. 1, 2, 3, and 4. Operation of the column as a 1A Simple Extraction Section, 1B Simple Scrub Section, and 1C Column led to the following new information:

1. H.T.U. values (for uranium transfer) obtained in these studies were 1/3 to 1/2 of corresponding values obtained in packed columns, with flooding capacities similar to those obtained with 1/2-in. Raschig rings and less than

3

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predicted capacities are as follows:

1A	100 cycles/min.
1B	125 cycles/min.
1C	100 cycles/min.

At these conditions, phase emulsification becomes severe. This has been observed in 3-in. i.d. glass pulse column TBP studies (4).

4. An approximate scale-up factor of 50 per cent increase in H. T. U. was observed for a diameter increase from 1 inch to 5 inches for both the 1A and the 1C Column.

5. Increased throughput might be attained by the use of other plate cartridge geometries and pulsing conditions as indicated by TBP Waste Metal Recovery studies (4), but it is not believed that the H. T. U. values can be significantly reduced.

DISCUSSION

Staff: The experimental studies were carried out by the Scale-Up Operations Group of the Chemical Development Section under the direction of A. R. Maguire and G. C. Oberg with J. C. Cobb, E. R. Irish, G. F. Jacky, G. E. Johnston, G. R. Kiel, R. B. Lemon, E. F. Riggs, W. J. Smith, Jr., and N. G. Whittenbrock assisting. Planning and data correlation were conducted by the Chemical Engineering Group. Chemical and process assistance was rendered by the Process Chemistry Group while assistance in solution of design and instrumentation problems was given by the Equipment Development Group.

Equipment and Operating Procedure: The Scale-Up solvent extraction equipment located in the 321 Building was employed for all studies described herein. The 5.05-in. i.d. stainless-steel column was the result of alteration of an existing Scale-Up packed column, and the pulse generating mechanism was obtained by alteration of an existing Demonstration Unit bellows-type pump and drive. The important details of the column and plate cartridge construction are given in Fig. 5, and a description of the pulse generating mechanism is given in Table I. Standard Worthington centrifugal pumps and flow controllers were found adequate for controlling the streams and no difficulty was encountered with the flow-con-

trol equipment. The pulse applied to the bottom organic feed was transmitted to the aqueous effluent stream and resulted in minor oscillation of both organic feed and aqueous effluent recorder-controller instruments.

At high pulse frequencies it was evident that the full, planned amplitude was not delivered to the column (See Fig. 4, 1A Flooding, Run No. 5"-32b-U). The factors causing lowered pulse amplitude were: 1) failure of the check valve in the organic feed line to seat properly due to inertia of the moving valve parts, and 2) failure of the bellows to follow the cam on the intake half of the pulse cycle. The organic feed was pumped from the Scale-Up column enclosure, through approximately 75 ft. of 1-1/2-in. i.p.s. pipe, in which was located an orifice-type flow meter and Hammel-Dahl flow control valve, then through a check valve into a 1-1/2-in. i.p.s. by 3-ft. long manifold. From the manifold the feed was supplied to the column through an additional 12-1/2 ft. of 1-1/2-in. i.p.s. pipe terminating in a 3-hole distributor. Side arms of 1-in. i.p.s. pipe were connected into the manifold and into each bellows. Compression and expansion of the bellows supplied a pulse to the feed stream. With no resistance to backward flow given by the flow-control valve and pump pressure only 14 per cent of the pulse would be absorbed in the 75-ft. long feed line from the pump to the bellows while under operating conditions (resistance of Hammel-Dahl valve and pump pressure) this effect is negligible and essentially the entire pulse displacement is applied to the column even with no check valve operating.

The force required to compress a bellows was supplied through a push-rod located over a cam-actuated lever arm. The force for bellows return (intake half of the bellows cycle) was supplied by a spring pressing downward against a collar near the lower end of the bellows push-rod. The maximum pressure on the pulsing bellows occurs at the beginning of each intake, and compression stroke and this pressure is proportional to the square of the frequency (5). It is likely that insufficient force was available to give immediate bellows expansion at high frequencies and that therefore, the bellows did not faithfully follow the cam, with resulting reduction of pulse displacement. No measurements were made of the expansion force available but observation of the operating bellows at frequencies greater than 150 cycles/min. indicated that full expansion was not reached between the compression halves of the cycles. More recent

6
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TBP Studies (4) were carried out using a single or twin piston mechanically linked to the drive cam and no similar failure to obtain full pulse amplitude was observed.

Operating Experience: Total project experience with packed solvent extraction columns as contactors for Redox, "25", and other processes is approximately 12,000 hrs. The pulse studies reported here summarize approximately 260 hrs. of operation. Although satisfactory Redox plant pulse column specifications could probably have been developed from these studies it is felt that more data are required to provide a firm design basis.

Calculation of Data: The methods used for calculation of H. E. T. S. and H. T. U. values are explained and illustrated in Redox Technical Data Studies 10, 11, and 14 (HW-12371, HW-12968, and HW-13908). The equilibrium data used were reported in HW-14984, HW-12350, HW-15209, and 1C data to be reported in HW-18773. H. T. U. calculations were based on concentration driving forces in the phase from which the uranium transferred (6). Steady operating portions of the runs (designated H. E. T. S. portion) were evaluated for the determination of H. E. T. S. and H. T. U. values.

Comparison of Pulse and Packed Column Runs: Plotted H. T. U. vs. volume velocity curves are generally convex downward for either packed or pulsed 1A, 1B, and 1C Column operation. These curves exhibit a rapid increase in H. T. U. at very low flow rates or at high flow rates as flooding is approached, and show an intermediate (plateau) region in which the H. T. U. is low and varies little with changes in flow rate. For ready comparison of pulse column and packed column operation a short summary table (based on Tables I, II, and III and Figs. 1, 2, 3, and 4 of this document and on Figs. 2, 3, and 4 of HW-15663) follows:

PULSE COLUMN STUDIES

Type Run	H. T. U. Ft. (a)	Pulse Conditions		Gal. / (Hr.) (Sq. Ft.), Sum of Both Phases.	
		Ampl. In. (b)	Freq. Cycles/Min.	Range for H. T. U.	Flooding
1A	0.6	0.45	50	300-1300	1500
1B	0.5	0.45	125	400-3000	3800
1C	0.8	0.45	100	500-1600	2000
1C	0.7	0.40	50	500-1300	1400

PACKED COLUMN STUDIES (c)

Type Run	Runs in 1/2-in. by 1/2-in. R/R			Runs in 1-in. by 1-in R/R		
	H. T. U. Ft. (a)	Gal. / (Hr.) (Sq. Ft.), Sum of Both Phases		H. T. U. Ft. (a)	Gal. / (Hr.) (Sq. Ft.), Sum of Both Phases	
		Range for H. T. U.	Flooding		Range for (H. T. U.)	Flooding
1A	1.6	500-1100	1700	1.6	1200-2500	3700
1B	1.6	400-2000	2700	1.8	800-2600	4100
1C	2.0	400-1200	1800	2.5	500-2500	3000

Notes to Table:

- (a) "Overall water-film" basis for 1A and 1B runs, and "overall organic-film" basis for 1C runs. Maximum values for listed conditions.
- (b) Pulse movement in the column, inches.
- (c) Packed length, feet:

	<u>1A</u>	<u>1B</u>	<u>1C</u>
1/2-in. by 1/2-in. Raschig rings	20	19	20
1-in. by 1-in. Raschig rings	20	19	20

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Comparison of these Redox pulse column operations with packed column operations under similar flowsheet conditions are applicable only to the pulse column as defined by Fig. 5 and at the pulse conditions (amplitude and frequency) listed. A more comprehensive study of pulse column variables such as that reported in HW-19170(4) would permit a more complete comparison of the pulse column with the packed column as a Redox contactor. Within the limits as defined for the pulse column in the above table some comparisons with packed column operation may be made as follows:

The 1A Column:

Flooding capacity (at 0.45-in. amplitude and 50 cycles/min.) in the pulse column and in 1/2-in. by 1/2-in. Raschig rings is approximately the same while the use of 1-in. by 1-in. Raschig rings permits approximate doubling of the packed column maximum capacity.

The 0.6-ft. H. T. U. ("overall water-film" basis) in the pulse column, (at pulse conditions listed and within the operating range indicated) is approximately 40% of the 1.6-ft. H. T. U. obtained in either 1/2-in. by 1/2-in. or 1-in. by 1-in. Raschig rings within the respective listed operating ranges.

The useful operating range (plateau) for the pulse column extends from 20 to 90 per cent of the total flooding capacity, while in the packed column this range is from 35 to 65 per cent of the total flooding capacity. The tabulated flooding capacity of the pulse column may be increased from 1500 to more than 2500 gal. / (hr.)(sq. ft.), sum of both phases, by varying the pulse frequency from 50 to 100 cycles per minute at 0.45-in. amplitude- thus extending the useful pulse column operating range. (Fig. 4, 1A Column). It may therefore be possible to select pulse conditions which will give the best H. T. U. at a given throughput in the pulse column while in the packed column only one operating range is available and that range is predetermined by the size and type of packing and by the flowsheet.

The 1B Column:

Each listed total flooding capacity for the 1B Column is for a 1B Simple Scrub Section with no ferrous sulfamate present in the IBFX. Packed column

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studies in 1/2-in. by 1/2-in. Raschig rings gave a 25 per cent lower capacity (2000 gal. / (hr.)(sq. ft.), sum of both phases) with ferrous sulfamate present and the total flooding capacity of the 1B pulse column might be lowered by a similar amount if ferrous sulfamate were employed in 1BFX. The data (tabulated above) indicate that the maximum total flooding capacity of 3800 gal. / (hr.)(sq. ft.), in the pulsed 1B Simple Scrub Section at 0.45-inch pulse amplitude (Fig. 4, 1B Column) is nearly as great as for 1-in. by 1-in. Raschig rings (4100 gal. / (hr.)(sq. ft.)) and greater than for 1/2-in. by 1/2-in. Raschig rings (2700 gal. / (hr.)(sq. ft.)).

The 0.5-ft. uranium H. T. U. (overall water-film basis) in the pulse column at the pulse conditions listed and within the operating range indicated is approximately constant (Fig. 2) at less than 35 per cent of the 1.6-ft. and 1.8-ft. H. T. U. obtained in 1/2-in. by 1/2-in., and in 1-in. by 1-in. Raschig rings respectively.

The useful operating range (plateau) for the pulse column included from 10 to at least 80 per cent of flooding while in the packed column the useful operating range (plateau) is limited to 20 to 70 per cent of flooding.

The 1C Column:

The maximum total flooding capacity of the 1C pulse column is 2000 gal. / (hr.)(sq. ft.), sum of both phases, for 0.45-in. amplitude at 100 cycles per minute (Fig. 4, 1C Column). This value is approximately the same as the capacity of 1/2-in. by 1/2-in. Raschig rings (1800 gal. / (hr.)(sq. ft.)) and about 2/3 of the capacity of 1-in. by 1-in. Raschig rings (3000 gal. / (hr.)(sq. ft.)).

A uranium H. T. U. of 0.8 ft. or less ("overall-organic-film" basis) may be obtained in the 1C pulse column operating at 0.4 to 0.45-in. amplitude and 50 to 100 cycles per minute frequency in the range of 500 to 1600 gal. / (hr.)(sq. ft.), sum of both phases, and this value may be compared with the 2.0-ft. H. T. U. obtained in 1/2-in. by 1/2-in. Raschig rings over the narrower range of 400 to 1200 gal. / (hr.)(sq. ft.). The operating range of the packed column may be extended to 2500 gal. / (hr.)(sq. ft.) by the use of 1-in. by 1-in. Raschig rings but the H. T. U. is increased to 2.5 ft.

The useful operating range (plateau) for the 1C pulse column is from 25 to

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80 per cent of the maximum flooding capacity (at 0.45-in. pulse amplitude). In 1-inch by 1-inch Raschig rings a similar plateau is found at 20 to 85 per cent of flooding while in 1/2-inch by 1/2-inch rings the plateau is defined by narrower limits to within 20 to 65 per cent of flooding.

Future Work: The most attractive feature of the pulse column is the shorter length (compared with a packed column) needed to perform a given extraction operation. The TBP Waste Metal Recovery pulse columns (4) are designed to take advantage of the short pulse column length and thus to fit within space provided by present 221 Bldg. cells.

Pulse columns should be considered as contactors in any future solvent extraction process where column length is important because of architectural cost consideration or the requirement for a large number of transfer units in a single column must be met.

A major disadvantage of the pulse column is that the pulse generating mechanism may be subject to mechanical failure with resulting shutdown and loss of production. The expected shutdown frequency may be somewhat greater by the pulse generator failure rate for a pulse-column plant than for a packed-column plant having the same number of columns, but subject to less maintenance than a plant employing mixer-settler units.

In all solvent extraction process development work an economic evaluation should be prepared to include both initial plant and equipment cost, and operating costs for pulse and packed columns. It is possible that an advantageous combination of pulsed and packed columns may be employed in the same cascade battery or in the same plant where the less complex packed columns are employed wherever feasible and pulse columns are employed where large numbers of transfer units are required.

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W. S. Figg

J. G. Bradley

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Fig. 2 — EFFECT OF VOLUME VELOCITY ON H.E.T.S. AND H.T.U. IN A 5.05-IN. I.D. REDOX SIMPLE IB PULSE COLUMN SCRUB SECTION

Plate Cartridge

Effective Length	4.72 ft.
Plate Hole Size	0.04 in.
Number of Holes	ca. 3000 per plate
Free Area	22 %
Number of Plates	28
Thickness of Plates	0.025 in.
Spacing of Plates	2.0 in.

SCHEMATIC SIMPLE REDOX IB PULSE COLUMN

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Fig. 3 — EFFECT OF VOLUME VELOCITY ON H.E.T.S. AND H.T.U. IN A 5.05-IN. I.D. REDOX IC PULSE COLUMN

Plate Cartridge

Effective Length	4.72 ft.
Plate Hole Size	0.04 in.
Number of Holes	ca. 3000 per Plate
Free Area	22 %
Number of Plates	28
Thickness of Plates	0.025 in.
Spacing of Plates	2.0 in.

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Fig. 1 — EFFECT OF VOLUME VELOCITY ON H.E.T.S. AND H.T.U. IN A 5.05-IN. I.D. REDOX SIMPLE IA PULSE COLUMN

Plate Cartridge Data

Effective Length	4.72 ft.
Plate Hole Size	0.04 in.
Number of Holes	ca. 3000 per plate
Free Area	22 %
Number of Plates	28
Thickness of Plates	0.025 in.
Spacing of Plates	2.0 in.

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Fig. 4— EFFECT OF PULSE FREQUENCY ON TOTAL FLOODING CAPACITY
OF 5.05—IN. I.D. REDOX SIMPLE IA EXTRACTION, SIMPLE IB SCRUB,
AND IC PULSE COLUMNS

Note a: See text, Section IX, "Equipment and Operating Procedure"

Note b: No ferrous sulfamate present in these runs.

Definitions

Amplitude = Inches of Vertical Displacement Given an Interface in the Column When the Pulse Generator Bellows Move from Minimum to Maximum Compression

$$\text{Pulsed Volume Velocity} = \left(\frac{\text{Gal. per Sq. Ft.}}{\text{Cycle}} \right) \left(\frac{\text{Cycles}}{\text{Hr.}} \right)$$

$$= 74.8 (\text{Inches Amp. in a Column}) (\text{Frequency, Cyc. per Min.})$$

Amplitude: Approx. 0.45 in. for All Points Plotted

$$\text{For This Amplitude Pulsed Volume Velocity} = 33.7 (\text{Frequency, Cycles/Min.})$$

Plate Cartridge Data

Effective Length	4.72 Ft.
Number of Plates	28
Spacing of Plates	2.0 In.
Thickness of Plates	0.025 In.
Plate Hole Size	0.04 In.
Number of Holes	ca. 3000 per Plate
Free Area	22 %

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Figure 5

5.05-In. I.D. Stainless Steel Pulse Column with 9.62-In. I.D. Top Section
as Used in Runs 5"-24-U through 5"-33-U, 5"-17-BU through 5"-21-BU
and 5"-64-CU through 5"-72-CU

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TABLE I

TABLE I
A SIMPLE EXTRACTION COLUMN STUDIES: 5.05-IN. DIAMETER PLATE COLUMN

Run No.	Primary Diluent	Fractional Mass-Transfer				Fractional Plate Efficiency		Volume Velocity (c) gals./sq.ft./min.	Equivalent Short Tons of Metal/ hr. (Based on 100% Feed)	Feed Concentration % of Total in IAW	ANN Equiv. in IAW (Based on 100% Feed)	L/V Dilute Ratio	Pulse		Disengagement Time, Seconds	Uranium Material Bald, % Recovery	Approximate Operating Line, % Feed Phase		Run Dates	
		H.S.T.S., %	No. of Plates	H.T.C., %	No. of Transfer Units	Murphree Single Plate	Murphree Overall						Frequency Surges/Min.	Amplitude, inches						
5*-2-U	H.S.T.S.	0.65	7.25	0.35	13.60	0.26	0.22	814	0.88	0.015	0.008	411	51	0.40	13	105.4	16	9	10-17	10-17
5*-2-U	Flooding	Not to be determined							1500 ± 50	1.71	—	—	51	0.40	13	—	—	—	10-17	10-17
5*-25-U	H.S.T.S.	0.76	6.25	0.47	9.96	0.22	0.24	1319	1.50	0.18	1.00	411	52	0.40	13	96.6	10	8	10-17	10-18
5*-26-U	H.S.T.S.	0.78	6.36	0.52	9.09	0.22	0.32	600	0.69	0.60	3.11	407	52	0.42	13	103.3	12	5	10-18	10-19
5*-27-U	H.S.T.S.	0.81	5.80	0.55	8.55	0.21	0.32	321	0.37	0.69	3.70	407	52	0.42	13	105.4	29	11	10-19	10-20
5*-28-U	H.S.T.S.	Flooded							< 900	—	—	—	52	0.110	22	—	1	0	10-30	10-30
5*-29-U	H.S.T.S.	Flooded							< 900	—	—	—	52	0.225	22	—	6	—	10-30	10-31
5*-30-U	H.S.T.S.	0.61	7.70	0.37	12.91	0.23	0.37	528	1.03	0.037	0.18	412	52	0.30	22	97.6	4	6	10-31	10-31
5*-31-U	H.S.T.S.	0.61	7.70	0.36	13.11	0.23	0.37	927	1.03	0.036	0.17	410	52	0.45	22	96.3	9	6	10-31	10-31
5*-32-U	Flooding	Not to be determined							2200 ± 200 (j)	2.43	—	—	125	0.45	22	—	—	—	11-1	11-1
5*-32-U	H.S.T.S.	0.70	6.75	0.42	11.82	0.24	0.38	1126	1.30	0.089	0.44	414	125	0.45	22	97.6	3	2	11-1	11-1
5*-32-U	Flooding	Not to be determined							2000 ± 200 (j)	2.24	—	—	200	0.45	22	—	—	—	11-1	11-1
5*-32-U	H.S.T.S.	0.72	6.53	0.43	11.04	0.24	0.38	997	1.05	0.053	0.26	413	200	0.45	22	98.8	3	2	11-11	11-1
5*-32-U	Flooding	Not to be determined							2600 ± 200 (j)	2.87	—	—	90	0.45	22	—	—	—	11-1	11-1
5*-33-U	H.S.T.S.	Flooded							4100 (k)	—	—	—	100	0.45	—	—	—	—	—	—

FLOODING CAPACITY OF COLUMN

FLOODING CONDITIONS, MINIMUM

Run No.	Plate Efficiency		Disengagement Time, Seconds	Feed Concentration % of Total in IAW	Column I.D., In.		ANN Equiv. in IAW (Based on 100% Feed)	L/V Dilute Ratio	Frequency Surges/Min.	Amplitude, inches	Disengagement Time, Seconds	Uranium Material Bald, % Recovery	Approximate Operating Line, % Feed Phase		Run Dates	Notes
	Experimental	Calculated			Below Feed	Disengaging Section										
5*-2-U Through 5*-33-U	22 sieve plates, 0.025-in. thick stain- less steel, approx. 3000 holes, 0.04-in. diameter, triangular spacing, approx. 0.086-in. on side. Supported by 1/4-in. in. stainless steel rods arranged in a square on a 3/4-in. diameter circle. Plates separated by 2-in. long spacers of 1/4-in. IPS stainless steel pipe. Free area of the plates was ca. 25%.	4.72	3-hole spider fabricated from pipe fittings, located 1/2-in. below bottom plate. Distributor holes are 1/4-in. IPS pipe on approxi- mately 3 1/2-in. diameter circle.	1-hole bagnet-type fabricated from 1 1/2-in. stainless steel pipe, dis- charging aqueous feed into the aque- ous phase approximately 7-in. above the top plate.	5.05	9.62	5*-2-U Through 5*-33-U	HW-13320 (HW-1)	500	0	-13	3.40	25	13	10-17	This column functioned to transfer uranium from the aqueous phase to the organic phase and was operated to simulate the extraction section of a Redox II Column. The aqueous IAW feed contained 10% and 10% entered the top of the column as the continuous phase and flowed downwardly. The organic IAW feed entered the bottom of the column as the dispersed phase and flowed upwardly. After leaving the upper end of the plate cartridge, the organic was the dispersed phase which it reached the interface above the aqueous feed distributor. The level of this interface was maintained automatically by the aqueous feed rate controller, which was regulated by the pressure differential between two inert gas bubbling dip tubes, one above and the other below the interface. The IAW organic feed was controlled by a simple 1/2-in. diameter line orifice - diaphragm-operated valve system, and the IAW aqueous feed was controlled by an orifice-controlled diaphragm-operated valve system. The IAW organic effluent was not controlled directly and was removed as overflow from the top of the column. These streams were pumped up Northington stainless-steel pumps.

Description of Operation

This column functioned to transfer uranium from the aqueous phase to the organic phase and was operated to simulate the extraction section of a Redox II Column. The aqueous IAW feed contained 10% and 10% entered the top of the column as the continuous phase and flowed downwardly. The organic IAW feed entered the bottom of the column as the dispersed phase and flowed upwardly. After leaving the upper end of the plate cartridge, the organic was the dispersed phase which it reached the interface above the aqueous feed distributor. The level of this interface was maintained automatically by the aqueous feed rate controller, which was regulated by the pressure differential between two inert gas bubbling dip tubes, one above and the other below the interface. The IAW organic feed was controlled by a simple 1/2-in. diameter line orifice - diaphragm-operated valve system, and the IAW aqueous feed was controlled by an orifice-controlled diaphragm-operated valve system. The IAW organic effluent was not controlled directly and was removed as overflow from the top of the column. These streams were pumped up Northington stainless-steel pumps.

Description of Flooding and Capacity

Flooding determinations were made at constant frequency and amplitude, starting column operation at a low volume velocity and increasing rates in increments of 50 gals./hr. until a flood occurred. Each determinate was made over a period of eight five-minute runs after which the rates were halved and the column was again started. Flooding was indicated by excessive organic entrainment in the IAW stream. Flooding rate increase was also indicated by a pressure differential between two inert gas bubbling dip tubes, one above and the other below the interface. Flooding was then reduced by decreasing the feed rate, and the flooding point was again approached in increments of 10 gals./hr. until a steady state was reached. Flooding determinations were made at rates of 100, 200, 400, and 800 gals./hr. until the column again flooded.

Description of Pulse Generator

The pulse generator mechanism used with the 5.05-in. column for these runs employed six stainless steel bellows with a total of 87.0 square inches cross sectional area, which compressed and expanded in simple harmonic motion. This motion was generated by a diaphragm pump on which rode a roller with a lever connection to the bellows. The frequency of the pulses was variable by means of a "stepped" cone from 25 to 400 cycles per minute. The bellows were bellows in section was 1.125 inches, which gave a displacement range of 1 to 1.125 in. in the 5.05-in. i.d. section of the column when 3 bellows were operating (one bellows was valued off as a spare).

NOTES FOR TABLE I

- Calculated on an "overall water film" basis.
- Murphree efficiency of a single plate, E_m is defined by the equation $E_m = \frac{P - P_0}{P - P_1}$, where P is the ratio of the slope of the operating line, L/V , to the slope of the equilibrium line, m , and P_0 is the overall plate efficiency.
- Total volume velocity is the sum of both phases calculated for the superficial (unpacked) 0.139 sq.ft. cross sectional area of the column and based on inlet stream rates.
- Calculated on the basis that 1.000 gal./min. of IAW (approx. 1.00 UNH) or 0.1343 gal./min. of IAW (2.00 UNH) is equivalent to a plant capacity of 1.0 short tons of uranium metal processed per twenty-four hours.
- Based on concentration in g./l. of 11.80% Na_2CO_3 plus 0.735 times concentration in g./l. of $NaNO_3$. High ANN equivalent in IAW for runs 5*-2-U through 5*-32-U is due to greater than flow-sheet $NaNO_3$ present in IAW.
- "L" is weight per unit time of UNT-free IAW. "V" is weight per unit time of UNT-free IAW (same weight units as "L"). L/V is the slope of the operating line at the dilute end of the column.
- "Frequency" is the number of complete cycles made per minute by the pulsing pump and is the same as the number of surges in the column.
- "Amplitude" is the vertical movement from maximum to minimum given an interface in the 5.05-in. i.d. column by the pulsing pump, assuming no net flow through the column.
- Disengagement time is defined as the time required for one volume of aqueous feed (IAWS) and two volumes of organic feed (IAO) to separate with not over 1/8-in. of emulsion at the interface following 20 contacts made by inverting a 50 ml. stoppered graduate once per second for twenty seconds.
- Each flooding determination in this run was characterized by the presence of an emulsion in the bottom section of the column at volume velocities somewhat lower than the flooding capacity.
- Column was washed out with 5% $NaNO_3$ between 5*-32-U and 5*-33-U; the first attempt to run following this acid wash resulted in ca. 50% entrainment of hexone phase in the IAW at 900 gal./hr. (sq.ft.); after soaking the column for 12 hr. in IAW, the flooding capacity was measured as 2100 gal./hr. (sq.ft.). Difference between this run and 5*-32-U is possibly due to surface conditioning of the plates.

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TABLE II
 IB SIMPLIFIED SCRUB SECTION STUDIES: 5-INCH DIAMETER PULSED COLUMN

Run No.	Primary Purpose	Uranium Mass Transfer						Fractional Plate Efficiencies		Volume Velocity, gal./min.	Equivalent Short Tons U Metal/24 Hr. (a)	U in IBP		% Contamination of Pu With U in IBP (c)	AN Equiv. In IBP (d)	L/V (b)	L/V (1)	Pulse		Uranium Material Bal., % Recovery	Approximate Operating Time, hours		Run Dates (194)		
		H.E.T.S., Ft.	No. of Plates	H.E.T.S., Ft.	No. of Transfer Plates	Murphree Single Plate Eff.	Overall Plate Eff.	U in IBP (g./l.)	U in IBP (g./l.)			Frequency	Amplitude, in.					Total	Excess, Period						
5"-17a-BU	Flooding	Not to be Determined								1300 ± 200	4.35	---	---	---	---	---	---	49	0.45	---	---	---	11-14	11-14	
5"-17b-BU	Flooding	Not to be Determined								2500 ± 200	6.30	---	---	---	---	---	---	---	75	0.43	---	---	---	11-14	11-14
5"-17c-BU	Flooding	Not to be Determined								3400 ± 200	8.35	---	---	---	---	---	---	---	102	0.42	---	---	---	11-14	11-14
5"-17d-BU	Flooding	Not to be Determined								3800 ± 200	9.20	---	---	---	---	---	---	---	122	0.45	---	---	---	11-14	11-14
5"-17e-BU	Flooding	Not to be Determined								3000 ± 200	7.25	---	---	---	---	---	---	---	153	0.45	---	---	---	11-15	11-15
5"-18-BU	H.E.T.S.	1.48	3.18	0.47	10.11	0.11	0.31	390	1.17	1730	6800	27 x 10 ⁻⁵	1.08	490	0.413	0.049	125	0.40	103.6	19	7	11-15	11-16		
5"-19-BU	H.E.T.S.	1.58	2.81	0.51	9.34	0.10	0.28	1212	3.41	1700	13000	51 x 10 ⁻⁵	2.05	479	0.305	0.041	125	0.45	98.1	8	6	11-16	11-16		
5"-20-BU	H.E.T.S.	1.50	3.15	0.48	11.30	0.11	0.33	2076	5.32	1330	7300	29 x 10 ⁻⁵	1.16	487	0.256	0.031	125	0.42	110.2	6	5	11-16	11-16		
5"-21-BU	H.E.T.S.	1.64	2.87	0.49	9.57	0.10	0.30	2971	8.11	860	3000	32 x 10 ⁻⁵	1.27	486	0.305	0.038	124	0.40	95.3	4	2	11-16	11-17		

SALIENT DETAILS OF COLUMNS

Run No.	Plate Cartridge Description	Height, Ft.	Scrub Introduction	Feed Introduction	Column I.D., in.	
					Below Feed	Top Disengaging Section
5"-17a-BU Through 5"-21-BU	28 sieve plates, 0.025-in. thick stainless steel, approx. 3000 holes, 0.04-in. diameter, triangular spacing, approx. 0.060-in. on side. Supported by 4 - 5/16-in. stainless steel rods arranged in a square on a 3 1/2-in. diameter circle. Plates separated by 2-in. long spacers of 1/4-in. IPS stainless steel pipe. Free area of the plates was ca. 22%.	4.72	3-hole spider fabricated from pipe fittings, located 1 1/4-in. below bottom plate. Distributor holes are 1/4-in. IPS or on approximately 3 1/2-in. diameter circle.	3-hole bayonet type fabricated from 1 1/2-in. stainless steel pipe, discharging aqueous feed into the aqueous phase approximately 7-in. above the top plate.	5.05	9.62

IB FLOWSHEET CONDITIONS (DUAL PURPOSE COLUMN)

Run No.	Reference	Organic Feeds-Solvent		Aqueous Feed	
		Feed (L/V)	Solvent (L/V)	Feed (L/V)	Extraction (L/V)
Flowsheet	EW-13320 Redox H ₂ O ₂	UO ₂ 240 g./l. HNO ₃ 3.6 g./l.	Hexane HNO ₃ 3.2 g./l.	ANX HNO ₃ Fe(XL ₂ SO ₃) ₂	438 g./l. 3.2 g./l. 12.4 g./l.

IB MONITORING CONDITIONS (SIMPLE SCRUB SECTION)

Reference	Feed (IBP)	Scrub. (IBS)	Aqueous Feed (IBTX)	
			Feed	Extraction
5"-17-BU Through 5"-21-BU	Not used for simple scrub section runs.	Hexane HNO ₃ 3.2 g./l.	ANX HNO ₃ Fe(XL ₂ SO ₃) ₂	438 g./l. 3.2 g./l. 12.4 g./l.

*Ferrous sulfamate omitted from aqueous IBTX used in these runs.

NOTES FOR TABLE II

- (a) Calculated on an "overall water-film" basis.
- (b) "Murphree efficiency, E_m " of a single plate is defined by the equation $E_m = \frac{P - 1}{P}$, where P is the ratio of the slope of the operating line, L/V , to the slope of the equilibrium line, m , and E_o is the "overall plate efficiency".
- (c) Total volume velocity is the sum of both phases calculated for the superficial (unpacked) cross section of the column (0.139 sq.ft.), and based on inlet stream rates.
- (d) For IB dual purpose runs the uranium processed is calculated from IBP feed composition and rates. For IB simple scrub column operation, a IBTX rate of 0.1449 gal./min. is considered equivalent to operation at 1 short ton (in the IBP stream) per 24 hr.
- (e) For IB dual purpose runs, the % U in the IBP is based on the IBP and IBTX analyses and rates. This value is not significantly different from the % of the IBP uranium, since the loss to IAW at Flowsheet conditions is small (less than 1%). For simple column operation, Flowsheet values are assumed to hold throughout and IBP loss is calculated as $(\text{micro g. UO}_2/\text{l.}/620) \times 2.5 \times 10^{-5}\%$.
- (f) % contamination of U in IBP = $100 \left(\frac{\text{g. Pu/l.}}{\text{g. U/l.} + \text{g. U/l.}} \right)$. For dual purpose runs, calculation of contamination is based on the Flowsheet ratio of Pu/U in the IBP and on actual flow rates and measured U concentration in the IBP. For simple runs, the % contamination = $100 \left(\frac{0.47 \times \text{g. UO}_2/\text{l. in IBP}}{0.295 + (0.47 \times \text{g. UO}_2/\text{l. in IBP})} \right)$.
- (g) Based on concentration in g./l. of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ plus 0.735 times the concentration of NaNO_3 in g./l.
- (h) "L" is weight per unit time of UO₂-free aqueous phase, "V" is the weight per unit time of UO₂-free organic phase (same wt. units as "L"). L/V is the slope of the dilute end operating line in the IB scrub section.
- (i) "m" is the slope of the equilibrium line. "L/V" is the slope of the operating line divided by the slope of the equilibrium line, both for the IB scrub section.
- (j) "Frequency" is the number of complete cycles per minute, made by the pulsing pump, and corresponds to the frequency of pulse cycles in the column.
- (k) "Amplitude" is the vertical movement from maximum to minimum given the interface in the 5.05-in. i.d. section of the column by the pulsing pump, assuming no net flow of liquid through the column.

Description of Operation

This column functioned as transfer uranium from the aqueous phase to the organic phase and was operated to simulate the scrub section of a reactor. The aqueous IBTX feed (representing the aqueous phase at the feed point of a reactor) entered the top of the column as the continuous phase and flowed downward; the organic IBP feed entered the bottom of the column as the dispersed phase and flowed upward through the plate cartridge. After leaving the top of the plate cartridge organic was transferred phase until it reached the interface above the aqueous feed distributor. The level of this interface was maintained automatically on the aqueous feed rate controller, which was regulated by the pressure differential between two inert gas bubbling lines: one above and one below the interface. The IBP organic feed was controlled by a Smith & Rooting controlling rotameter - diaphragm-operated valve system, and the IBP aqueous effluent was controlled by an orifice-controller - diaphragm-operated valve system. The IBP organic effluent was not controlled directly and was removed as overflow from the top of the column. These streams were pumped by Worthington stainless-steel pumps. The pulsing pump was located on the organic feed line.

Description of Flooding Point Determinations

Flooding point determinations were made at constant frequency and amplitude, starting column operation at a low volume velocity and increasing rates in increments of 500 gal./hr. (sq.ft.). Each increase was made over a period of about five minutes, after which the rates were held constant for twenty minutes. If no flooding occurred, flooding was indicated by excessive organic entrainment in the IBP stream; another rate increase was made and a similar procedure followed until flooding occurred. Rates were then reduced 500 gal./hr. (sq.ft.), and the flooding point was again approached, this time in increments of 100 gal./hr. (sq.ft.), until the column again flooded.

Description of Pulse Generator

(See Table I.)

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 CHEMICAL DEVELOPMENT SECTION
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