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GRADED PLATE CARTRIDGEFOR PUREX A-TYPE EXTRACTION COLUMN

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GRADED PLATE CARTRIDGE FOR PUREX A-TYPE EXTRACTION COLUMNSINTRODUCTION

The Purex Plant A-type solvent extraction columns are dual-purpose pulsed columns, consisting of an extraction section (lower half) and a scrub section (upper half). In the extraction section, uranium, plutonium, and a small fraction of the fission products are extracted into the organic phase. In the scrub section, a portion of the extracted fission products are scrubbed back into the aqueous phase, leaving the uranium and plutonium relatively decontaminated.

The mass transfer requirements and aqueous to organic flow ratios of the two sections of the columns are quite different and result in different dispersions and flooding characteristics.

It was realized that with the radically different shape of the flooding curves (volume velocity vs. amplitude times frequency) it would not be possible to have optimum operating conditions in both the extraction and scrub sections over a wide range of capacities. The columns were, therefore, designed to give optimum performance in both sections at the most practical capacity region under consideration at the time. This was achieved by making the scrub section diameter larger than that of the extraction section, giving the scrub section a lower pulse amplitude and a lower superficial throughput for a given processing rate. However, the use of conservative estimates of the scrub section capacity from the available data resulted in the optimum performance of both sections occurring at a capacity well above those anticipated. This means that the extraction section controlled the flooding at lower capacities. With the extraction section controlling the flooding, the maximum operable pulse frequency is below the optimum operating frequency of the scrub section.

Results from the Hot Semiworks Purex studies indicated that the attainment of optimum frequency operation in the scrub section may be more important to adequate decontamination than optimum frequency operation in the extraction section is to low waste losses. Therefore, a development program directed toward an A-type extraction section cartridge which would permit operation at pulse frequencies closer to optimum for scrub section performance than was possible with the cartridge originally specified for the plant was undertaken.

SUMMARY

A graded cartridge, which is a nonuniform cartridge having plates in the region of the column where flooding originates with a larger hole size, greater free area and/or greater plate spacing, was developed and specified for the Purex Plant A-type columns. The cartridge eliminated cyclic local flooding and

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increased the complete flooding frequency by 10 and 25 cycles per minute at volume velocities of 530 and 1060 gal./hr. - sq.ft. (sum of both phases), respectively, and exhibited satisfactory extraction efficiency over a wide range of frequencies and throughputs.

DISCUSSION

The Purex Plant HA, IA, and 2D Columns are identical in purpose and have been designated A-type columns. They are dual-purpose pulse columns, and as designed the diameter ratio of scrub to extraction section was 4 to 3. Both sections were to contain standard cartridges, i.e., stainless steel sieve plates with 1/8-inch-diameter holes, 23 percent free area, spaced 2 inches apart. The extraction sections were specified to contain four 13 percent free area louver-type redistributor plates, and the scrub sections were to have three 18 percent free area louver plates.

As shown in Figure 1, the two sections have radically different flooding characteristics. The two curves for the extraction section indicate cyclic local flooding and complete flooding. While the column is operable at conditions lying between these two curves, such operation is not desirable. The local cyclic flooding curve is generally considered as representing the upper limit of operating conditions.

Insufficient data are available to permit establishing the scrub section flooding curve with the same degree of confidence as the extraction column curve. However, the curve drawn in Figure 1 represents the most probable flooding condition.

At volume velocities below 1590 gal./hr. - sq.ft., the extraction section controls the flooding conditions. In the range of 530 to 1325 gal./hr. - sq.ft., the maximum operable pulse frequency as limited by the extraction section varies approximately from 50 to 80 percent of the scrub section flooding frequency. This range is below the 80 to 90 percent of the flooding frequency generally recognized as optimum for pulse column operation.

Recent Purex studies at the Hot Semiworks have shown that satisfactory plutonium and uranium waste losses are readily attained in the A-type columns, but decontamination of the product stream of fission products varied to as much as ten-fold below process specifications requirements. While indications are that this problem may be partially or even primarily chemical in nature, operation of the A-type scrub sections at pulse frequencies nearer their optimum frequencies would be expected to improve decontamination. Therefore, a development program was started to obtain an A-type extraction cartridge whose flooding characteristics would more nearly match those of the scrub section.

The dispersion in an A-type extraction column varies sharply throughout the height of the column. Near the top of the column there is a loose dispersion of large drops; lower, there is a tight dispersion of small drops; and below

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this, there is a loose dispersion of larger drops. The reason for this is believed to be related to the mass transfer; that is, a large rate of mass transfer tends to stabilize the formation of large drops. The large uranium transfer near the top of the column, the relatively large nitric acid transfer in the lower half of the column, and the low net transfer of either component in the intermediate regions would by this theory account for the disparity in dispersion throughout the column.

Flooding in the extraction section usually originates in the region of tight dispersion. A way of increasing the flooding frequency would be to "open" the cartridge in the region of the tight dispersion, i.e., increase the sieve plate hole size, free area and/or spacing. Such a cartridge has been designated as a graded cartridge.

EXPERIMENTAL RESULTS

Seven graded cartridges were investigated in a 3-inch-diameter glass pulsed column and three in a 23.5-inch-diameter column. The degrees of grading ranged from a plate-free section to various combinations of plate spacing and the inclusion of 33 percent free area sieve plates. The ten graded cartridges are described in detail in Table I. The experimental flooding frequency data are presented in Table II and the experimental H.T.U. data are presented in Table III.

Three-Inch-Column Results. All seven of the graded cartridges for the 3-inch-diameter column increased the complete flooding frequencies by 30 to 40 cycles per minute at 1069 gal./hr. - sq.ft. and 5 to 20 cycles per minute at 530 gal./hr. - sq.ft. The cyclic local flooding frequency was increased proportionally except in Cartridges F and G where it was eliminated entirely.

Observations of the uranium color line positions indicated that satisfactory waste losses could probably be obtained with each of the cartridges at pulse frequencies near the cyclic local flooding frequency. Cartridges F and G exhibited color line positions consistent with low waste losses over a range of frequencies from 60 to 100 cycles per minute.

Extraction efficiency runs confirmed the estimates based on uranium color line positions. Cartridges F and G had waste losses lower than 0.001 percent and H.T.U. values less than one foot over a range of throughputs from 265 to 1060 gal./hr. - sq.ft. and pulse frequencies of 60 to 100 cycles per minute.

Large Scale Column Results. Three graded cartridges were investigated in a 23.5-inch-diameter extraction section. The first of these, Cartridge H, was "opened" by increasing the plate spacing to 4 and 6 inches in the center of the column. At a volume velocity of 530 gal./hr. - sq.ft., this cartridge had a cyclic local flooding frequency of 105 cycles per minute and a complete flooding frequency in excess of 110 cycles per minute, the maximum frequency of the pulse generator.

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Cartridge I was scaled up from Cartridge F, the most promising cartridge tested in the 3-inch-diameter column. It exhibited no cyclic flooding, but showed no increase in complete flooding frequency at a volume velocity of 530 gal./hr. - sq.ft. The lack of increase was attributed to the lower two 13 percent free area louver plates in the cartridge. These were replaced with 23 percent free area louver plates to make Cartridge J. This cartridge showed flooding frequency characteristics similar to 3-inch-diameter Cartridge F, which had been scaled up; i.e., no cyclic local flooding and a complete flooding frequency of 105 cycles per minute at 530 gal./hr. - sq.ft. and a flooding frequency above the scrub section flooding frequency of 80 cycles per minute at 1060 gal./hr. - sq.ft.

Efficiency runs made in the 23.5-inch-diameter column indicated that all three cartridges tested gave good extraction efficiency. Cartridge J, the cartridge having the most promising flooding frequency characteristics, had high extraction efficiencies at volume velocities from 265 to 1060 gal./hr. - sq.ft. and frequencies of from 40 to 100 cycles per minute. Typical uranium waste losses were less than 0.001 percent and H.T.U. values were approximately one foot.

Of the cartridges studied, F, G, and J permitted the greatest increase in stable operating region and exhibited satisfactory waste losses over a wide range of operating conditions.

The flooding curves for Cartridges F and J, together with the scrub section flooding curve, are presented in Figure 2. The match between the scrub and extraction section flooding frequencies has been improved. The maximum stable operating frequency has been increased from 50 to 75 percent of scrub section flooding frequency at 530 gal./hr. - sq.ft. and from 65 to 100 percent of the scrub section flooding frequency at 1060 gal./hr. - sq.ft.

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TABLE I

EXTRACTION SECTION "GRADED - PLATE" CARTRIDGES

(Cartridges are described from top to bottom)

<u>In. of Cartridge Height</u>	<u>Hole Diameter, In.</u>	<u>% Free Area</u>	<u>Plate Spacing, In.</u>	<u>In. of Cartridge Height</u>	<u>Hole Diameter, In.</u>	<u>% Free Area</u>	<u>Plate Spacing, In.</u>
<u>Cartridge A</u>				<u>Cartridge G</u>			
36	0.125	23	2	4	0.125	23	1
48	void			8	0.125	23	2
24	0.125	23	2	8	0.188	33	2
<u>Cartridge B</u>				36	0.188	33	4
12	0.125	23	2	12	0.125	23	4
12	0.125	23	4	38	0.125	23	2
60	void			<u>Cartridge H</u>			
24	0.125	23	4	10	0.125	23	2
<u>Cartridge C</u>				12	(13% F.A. louver plate)		
12	0.125	23	2	12	0.125	23	6
72	0.188	33	6	12	(13% F.A. louver plate)		
12	0.125	23	4	28	0.125	23	4
12	0.125	23	2	12	(13% F.A. louver plate)		
<u>Cartridge D</u>				28	0.125	23	4
12	0.125	23	2	12	(13% F.A. louver plate)		
72(1)	0.125	23	6	36	0.125	23	2
12	0.125	23	4	<u>Cartridge I</u>			
12	0.125	23	2	12	0.125	23	2
<u>Cartridge E</u>				12	(13% F.A. louver plate)		
24	0.125	23	2	4	0.125	23	2
48	0.188	33	4	8	0.188	33	2
24	0.125	23	4	12	(13% F.A. louver plate)		
12	0.125	23	2	16	0.188	33	4
<u>Cartridge F</u>				12	0.125	23	4
18	0.125	23	2	12	(13% F.A. louver plate)		
8	0.188	33	2	28	0.125	23	4
20	0.188	33	4	12	(13% F.A. louver plate)		
32	0.125	23	4	36	0.125	23	2
28	0.125	23	2	<u>Cartridge J</u>			
				Same as "I" except bottom two louver plates are 23% free area.			

(1) Nozzle plates. 0.03-in. nozzle depth.

TABLE II

HA EXTRACTION COLUMN FLOODING STUDIES USING GRADED PLATE CARTRIDGES

Flowsheet: Purex HW #3, Pu and F.P.'s omitted.

Modified as indicated in remarks.

Equipment: As noted.

Continuous Phase: Aqueous

Run No.	Equipment	Cartridge Design ^(a)	Diluent	Volume Velocity, Gal./((Hr.)(Sq.Ft.)), Sum of Phases	Frequency Flooding Thresholds		
					Cyclic	Local	Complete
3-169-AH	(b)	A	Soltrol-170	1100	95 ± 5		105 ± 5
3-170-AH	(b)	A	Soltrol-170	530	105 ± 5		115 ± 5
3-173a-AH	(b)	B	Soltrol-170	530	>120		>120
3-175-AH	(b)	C	Soltrol-170	530	>120		>120
3-176-AH	(b)	C	Soltrol-170	1000	105 ± 5		115 ± 5
3-180-AH	(b)	D	Soltrol-170	530	None		115 ± 5
3-181-AH	(b)	D	Soltrol-170	800	None		115 ± 5
3-182a-AH	(b)	D	Soltrol-170	1100	90 ± 10		>100
3-183-AH	(b)	E	Soltrol-170	1100	95 ± 5		105 ± 5
3-184-AH	(b)	E	Soltrol-170	530	105 ± 5		115 ± 5
3-188-AH	(b)	F	Soltrol-170	1060	None		105 ± 5
3-189-AH	(b)	F	Soltrol-170	530	None		115 ± 5
3-198-AH	(b)	G	Soltrol-170	1000	None		105 ± 5
3-200-AH	(b)	G	Soltrol-170	530	None		115 ± 5
PX-1	(c)	H	Shell E-2342	530	106 ± 3		>109
PBM-4b	(c)	H	Shell E-2342	530	>90		
PX-2	(c)	I	Shell E-2342	530	None		95 ± 5
PX-4	(c)	I	Shell E-2342	1060	None		>90 ^(d)
PX-7	(c)	J	Shell E-2342	530	None		105 ± 5
PX-10	(c)	J	Shell E-2342	1060	None		>80 ^(d)

Notes:

- (a) See Table I for a detailed description of the cartridges.
- (b) 3.00-inch precision bore glass column, 9-ft. high. Pulse amplitude of 1.0 inch.
- (c) Dual-purpose column. 23.5-in.-diameter extraction section with 13.5-ft. cartridge, 32-in.-diameter scrub section with 11 to 12 ft. of standard cartridge. Pulse amplitude of 1.1 in. in the extraction section
- (d) Scrub section flooded at 85 ± 5 cycles per minute.

TABLE III

HA EXTRACTION COLUMN EFFICIENCY STUDIES USING GRADED PLATE CARTRIDGES

Flowsheet: Purex HW #3. Pu and F.P.'s omitted
Modified as indicated in remarks.

Equipment: As noted.

Continuous Phase: Aqueous

Run No.	Equipment	Cartridge Design (a)	Diluent	Volume Velocity, Gal/(Hr.)(Sq.Ft.), Sum of Phases	Frequency, Cyc./Min.	% U Loss	H.T.U., Ft.
3-173b-AH	(b)	B	Soltrol-170	530	110	0.030	1.08
3-182b-AH	(b)	D	Soltrol-170	1100	60	0.046	1.14
3-185-AH	(b)	E	Soltrol-170	1060	60	0.032	1.08
3-186-AH	(b)	E	Soltrol-170	1060	80	0.0049	0.86
3-187-AH	(b)	E	Soltrol-170	530	100	0.0021	0.78
3-190-AH	(b)	F	Soltrol-170	1060	60	0.0095	0.94
3-191-AH	(b)	F	Soltrol-170	1060	80	0.0084	0.77
3-192-AH	(b)	F	Soltrol-170	530	100	0.0034	0.74
3-193-AH	(b)	F	Soltrol-170	260	100	0.0036	0.79
3-201a-AH	(c)	G	Soltrol-170	1060	60	0.0079	0.93(e)
3-202a-AH	(c)	G	Shell E-2342	1060	60	0.0094	0.95(e)
3-206a-AH	(c)	G	Shell E-2342	1100	80	0.0001	0.63
3-206b-AH	(c)	G	Shell E-2342	1100	80	0.00008	0.61
3-207a-AH	(c)	G	Shell E-2342	530	80	≤ 0.0005	≤ 0.72
3-207b-AH	(c)	G	Shell E-2342	530	80	0.0003	0.70
3-208a-AH	(c)	G	Shell E-2342	1060	70	0.0011	0.70
3-208b-AH	(c)	G	Shell E-2342	1060	70	0.0011	0.70
PBM-4a	(d)	H	Shell E-2342	530	70	0.0002	1.03(f)
PBM-4b	(d)	H	Shell E-2342	530	90	0.0001	1.00(f)
PBM-7	(d)	I	Shell E-2342	1000	60	0.00002	1.02(f)
PX-1	(d)	I	Shell E-2342	530	90	0.0002	1.03
PX-2	(d)	I	Shell E-2342	530	60	0.0001	1.00
PX-8	(d)	J	Shell E-2342	530	90	0.0003	1.05
PX-9	(d)	J	Shell E-2342	530	60	0.0008	1.13
PX-11	(d)	J	Shell E-2342	1060	70	0.0001	0.99
PX-12	(d)	J	Shell E-2342	1000	50	0.0076	1.40
PX-13	(d)	J	Shell E-2342	260	100	≤ 0.00008	≤ 0.96
PX-14	(d)	J	Shell E-2342	800	80	≤ 0.00005	≤ 0.92
PX-15	(d)	J	Shell E-2342	530	40	0.014	1.51

Notes:

- (a) See Table I for a detailed description of the cartridges.
 (b) 3.00-in. precision bore glass column, 9-ft. high. Pulse amplitude of 1.0 in.
 (c) Dual-purpose column. 9-ft. length of 3.00-in. precision bore glass column as extraction section. 4-in. standard bore scrub section containing 8 ft. of standard cartridge. Pulse amplitude of 1.0 in. in the extraction section.
 (d) Dual-purpose column. 23.5-in.-diameter extraction section with 13.5 ft. of cartridge. 32-in.-diameter scrub section with 11 to 12 ft. of standard cartridge. Pulse amplitude of 1.1 in. in the extraction section.
 (e) Feed was spiked with coating removal waste, 25 ppm Si, and 0.003 g. solids/l.
 (f) Feed concentration was 1.35 M U, 1.0 M HNO₃, and 1.0 M NaNO₃.

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Scrub Section Flooding.

Extraction Section Standard Cartridge Instability Threshold.

Extraction Section Standard Cartridge Flooding.

Extraction Section Graded Plate Cartridge Flooding

Pulse Amplitude is 1.07 Inches.

FIGURE 1

Flooding Characteristics of Purex A-Type Columns

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