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LIGHT WATER REACTOR FUEL REPROCESSING PROGRAM

QUARTERLY PROGRESS REPORT FOR THE PERIOD ENDING MARCH 31, 1977

Prepared under
Contract EY-76-C-03-0167
Project Agreement No. 62
for the San Francisco Operations Office
U.S. Energy Research and Development Administration

DATE PUBLISHED: APRIL 1977

MASTER

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FOREWORD

This quarterly report contains the results of solvent extraction contactor improvement studies undertaken by General Atomic Company in support of the ERDA-sponsored Light Water Reactor Fuel Recycle Program. The basic objective of the work is to improve the performance of pulse columns through the use of alternate pulsing conditions and/or alternate column internals.

CONTENTS

FOREWORD	iii
1. SUMMARY.	1-1
2. INTRODUCTION	2-1
3. EQUIPMENT DESCRIPTION.	3-1
4. RESULTS AND DISCUSSION	4-1
4.1. Extraction Section	4-1
4.2. Scrub Column	4-2
4.3. Uranium Strip Column	4-2
4.4. Solvent Wash Column.	4-3
References	4-3
APPENDIX: DISTRIBUTION LIST	A-1

FIGURES

3-1. Top two levels of solvent extraction pilot plant at General Atomic Company	3-4
3-2. Overall view of solvent extraction pilot plant	3-5
3-3. Top level of solvent extraction pilot plant.	3-6
3-4. Bottom level of solvent extraction pilot plant	3-7
4-1. Nominal LWR Purex test flowsheet	4-12
4-2. Extraction column flooding curve	4-13
4-3. Uranium strip column flooding curve.	4-14
4-4. Solvent wash column flooding curve	4-15

TABLES

4-1. Flowsheet conditions and analytical values for baseline tests.	4-4
4-2. Column capacity data	4-10

1. SUMMARY

Standard nozzle plate cartridges were operated under a cross section of light water reactor (LWR) Purex flowsheet conditions to provide baseline data on pulse column throughput capacities. End stream samples were taken for future reference, but efficiency calculations were not made.

The main effort was concentrated on the extraction section performance. However, information was also obtained on uranium strip and solvent wash columns, since these are required in a service capacity to allow reuse of both the solvent and the uranium nitrate solution.

The major observations from these baseline tests are as follows:

1. The extraction section promises to have high capacity using nozzle plates with either 1/8- or 3/16-in.-diameter holes. No change in capacity was noted as a function of pulse amplitude over the range of 1 to 2 in.
2. The scrub section floods more readily than the extraction section at 1AP solvent loadings above 95 g U/l. Scrub section flooding can be more easily prevented or cleared using a dual pulse.
3. The uranium strip column has a more limited throughput capacity than the extraction section, as expected.
4. The solvent wash column operation continues to appear promising. No problem, such as those noted in the literature, was observed with very low capacity. The capacity and recoverability from flooding were improved by doubling the pulse amplitude from 1 to 2 in.

2. INTRODUCTION

The development of pulse columns and their internals was the subject of much effort at USAEC laboratories and contractors in the 1950s. Since that time, however, almost all development has been aimed specifically at resolution of the Hanford Purex Plant problems, and little effort has been expended outside the constraints established by existing facilities.

In the present work, baseline data were taken on standard or the current best pulse column cartridges (column internals) to allow future comparison of these cartridges with the alternative designs to be tested later.

In the baseline tests reported herein, the capacity of nozzle plate cartridges in a full-length extraction section was studied. Baseline tests on nozzle plate cartridges in short extraction column sections to study both capacity and efficiency using profile samplers will be the subject of the next quarterly report.

3. EQUIPMENT DESCRIPTION

The solvent extraction pilot plant facility at General Atomic Company is comprised of the following major equipment items:

1. Five 2- and 3-in.-diameter glass columns with overall heights up to 28 ft.
2. One eight-stage Robatel centrifugal contactor.
3. Thirty-three stainless steel tanks ranging in capacity from 10 to 900 l.
4. Feed and transfer pumps, intercolumn airlifts, flow rotameters, and metering pumps.
5. Interface sensing and control instrumentation.
6. Pulse generators.
7. A thermosyphon evaporator fabricated from stainless steel with a boil-off capacity of about 1 l/min.
8. Heat exchangers.

The solvent extraction columns are assembled from sections of commercially available glass pipe. (The columns and associated equipment are shown in Figs. 3-1 through 3-4.*) Stainless steel inserts between pipe sections provide means for connecting inlet streams, for sampling, and for supporting the column internals. Glass pipe crosses are used at the column ends as disengaging sections. Stainless steel plates on the cross

*Figures and tables appear at the end of each section.

ends provide means for connecting inlet, outlet, and vent lines as well as interface sensor instruments and pulse lines. The column internals are made from commercially available sieve and nozzle plates. These plates are supported by a central rod with tubing sections used as plate spacers.

All the tanks are of stainless steel construction. Each tank, as well as the evaporator, is equipped with nitrogen-purged dip tubes to provide manometer or recorder liquid level readouts. Selected tanks and the evaporator are also equipped to provide specific gravity readouts. A network of stainless steel tubing is used for intertank transfers and stream flows to the columns.

Solution transfers between tanks and from the tanks to the columns are made with centrifugal or positive displacement pumps. Flows to the columns are controlled at the desired rates with visual rotameters and manual control valves. Shell and tube heat exchangers, which were commercially obtained, are used to control the temperatures of the streams to the columns.

Flows between columns are maintained by pumps or airlift systems when overflow hydraulics do not allow cascade flow to succeeding columns.

The pulse generators consist of Teflon bellows activated by variable-throw cranks driven by variable-speed motors. One-inch pipe and flexible hose connect the bellows to the column bottoms.

The product concentrator is a thermosyphon unit constructed entirely of stainless steel. The single tube bundle utilizes low-pressure steam. A packed section in the vertical vapor space between the feed point and the thermosyphon loop is used for stripping of organic materials from the feed by the rising vapor.

The solvent used in the pilot plant is technical grade tributyl phosphate (TBP) in normal paraffin hydrocarbon (NPH) diluent. The TBP is ordinarily 30% by volume in the organic phase. The NPH is a straight-chained hydrocarbon purchased to Atlantic Richfield Hanford Company and Allied Chemical-ICP specifications. NPH is 98% normal-C₁₀H₂₂ to normal-C₁₃H₂₈ with less than 0.2% aromatics by weight. The density is approximately 0.75 g/ml. The NPH was purchased from Southampton Oil Company, Sisbee, Texas.

Fig. 3-1. Top two levels of solvent extraction pilot plant at General Atomic Company. (The columns are in the plexiglass cabinet in the center of the photograph.)

Fig. 3-2. Overall view of solvent extraction pilot plant

Fig. 3-3. Top level of solvent extraction pilot plant

Fig. 3-4. Bottom level of solvent extraction pilot plant

4. RESULTS AND DISCUSSION

The nominal LWR Purex flowsheet used in the tests described herein is shown in Fig. 4-1. The actual flowsheet conditions of each run and analytical data are reported in Table 4-1. The column capacity data are reported in Table 4-2 and are shown graphically in Figs. 4-2 through 4-4.

4.1. EXTRACTION SECTION

The flooding curve for the extraction section (Fig. 4-2) shows little effect of plate hole size or pulse amplitude variations.

The slope of the flooding curve and the high frequency required to flood the column at a throughput rate of 2000 gal/hr/ft^2 indicate a high capacity will be possible at lower frequencies, as expected. The shape of the flooding curve at the lower frequencies and higher flow rates will be determined as part of the efficiency tests with the 6-ft extraction section length. One flooding point from the runs using the 6-ft length is included in Fig. 4-2 to show there is little difference in column flooding between the 18- and 6-ft column lengths.

All aqueous waste losses were at or near the analytical detection limit for uranium. Consequently, no meaningful extraction section efficiency calculations can be made from these data which could indicate changes in stage height as a function of operating conditions. Efficiency comparison data will be taken from the short 6-ft column tests, where the aqueous effluent uranium will be more a function of operating conditions and where two profile samplers will be used to take intermediate data within the column length.

4.2. SCRUB COLUMN

In all runs, the scrub section had 3/16-in.-diameter-hole nozzle plates with 23% free area and 4-in. plate spacing. Only a short scrub section of 4 ft could be operated with the 18-ft extraction section because of the overall height limitation of the pilot plant. However, some observations were made. Generally, where the uranium loading of the overflow (1AP) solvent was greater than 95 g/l, the scrub section could flood more readily than the extraction section. The flooding in the scrub section began differently than in the extraction section (i.e., aqueous phase bubbles would gather around a plate and continuous phase inversion occurred without gradual buildup of aqueous phase plates as occurs prior to extraction section flooding). The scrub section flooding could be easily cleared only through the use of a dual pulse where one amplitude was 2 in. and the other was 1 in.

Scrub section behavior will not be studied during the tests of alternative plates in the short extraction section. However, it is realized that any development of a high-efficiency, high-capacity extraction section must be coupled to a compatible scrub section. At present it is believed that the scrub section must be joined as a compound column stacked on top of the extraction section, since separated columns tend to limit capacity due to flow changes caused by interface controller interactions (Ref. 4-1). Scrub section improvement studies to find a suitable matching cartridge will be made using the plates tested in the extraction section if a high-capacity, high-efficiency extraction cartridge is developed as a result of these studies.

4.3. URANIUM STRIP COLUMN

The uranium strip column flooding curve (Fig. 4-3) is, as expected, lower than the flooding curve for extraction. The end stream data were taken and will be compared with data taken under dual pulse conditions used during the stripping operations in the extraction column efficiency

tests. Uranium stripping efficiency comparisons will be reported when more information is available.

4.4. SOLVENT WASH COLUMN

The solvent wash column operated very well with high capacity as shown in Fig. 4-4. No evidence of inoperability such as that cited in Refs. 4-2 and 4-3 was found. It is significant that the column has an apparent higher capacity with a 2-in. pulse amplitude than with a 1-in. amplitude. No significant effect on solvent wash column flooding was noted between 1/8- and 3/16-in.-diameter holes or aqueous to solvent flow ratios. In future tests, a 1/2-in. amplitude will be tested to see if the column becomes inoperable. The Hanford Purex plant had a fixed amplitude of 0.53-in. and has been cited as having very poor operability with the same size nozzle plate cartridges tested here (Ref. 4-2). In future runs the solvent wash column capacity will be determined at high flow rate and lower frequency with the 2-in. amplitude. It has been determined in these current tests that the column recovers better using the 3/16-in.-diameter-hole nozzle plates and the 2-in. amplitude. Prior work and the 1-in. amplitude tests reported here showed that it was almost impossible to recover a flooded column using low amplitudes with the 1/8-in.-diameter-hole nozzle plates. Dual pulse tests indicated little advantage to that approach for solvent washing.

REFERENCES

- 4-1. Reddick, G. W., "Solvent Extraction in HTGR Reprocessing Interim Development Report," ERDA Report GA-A13835, General Atomic Company, February 1976, p. A-2.
- 4-2. Richardson, G. L., and A. M. Platt, "The Design and Operation of Industrial Scale Pulse Columns for Purex Service," Prog. Nucl. Energy, Ser. IV, 4, 279-307 (1961).
- 4-3. Richardson, G. L., "Demonstration of Purex Process Condensate Recycle and Other Purex Pulse Column Studies," USAEC Report BNWL-CC-2260, Pacific Northwest Laboratory, September 26, 1969.

TABLE 4-1
FLOWSHEET CONDITIONS AND ANALYTICAL VALUES FOR BASELINE TESTS

Run L-1

Stream	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)	Relative Flow	Other Analyses
1AF-1	1	314.3	2.96	260	100	--
1AS	2	--	3.28	150	58	--
1CX	3	--	0.01	1200	461	--
1OS	4	--	--	115	44	0.3M Na ₂ CO ₃
1AX	5	--	--	760	292	--
1SP	6	--	0.1	250	96	--
1AP-1 (a)	7	90.9	0.3	--	--	--
1AP-2 (b)	7	94.3	0.27	--	--	--
1AW-1	8	0.8 x 10 ⁻³	2.7	--	--	--
1AW-2	8	1.1 x 10 ⁻³	2.7	--	--	--
1CU-1	9	70.9	0.16	--	--	--
1CU-2	9	64.5	0.17	--	--	--
1CW-1	10	3.3 x 10 ⁻³	0.008	--	--	--
1CW-2	10	2.4 x 10 ⁻³	0.008	--	--	--
100-1	11	<0.4 x 10 ⁻³	--	--	--	--
100-2	11	<0.4 x 10 ⁻³	--	--	--	28.2% TBP
10W-1	12	--	--	--	--	5.5 g NO ₃ ⁻ /l
10W-2	12	--	--	--	--	9.45 g NO ₃ ⁻ /l

(a) Sample series 1 taken at following conditions: 1A column, 1-in. amplitude and 68-cpm frequency; 1C column, 1-in. amplitude and 65-cpm frequency; 10 column, 1-in. amplitude and 80-cpm frequency.

(b) Sample series 2 taken at following conditions: 1A column, 1-in. amplitude and 90-cpm frequency; other columns same as series 1.

TABLE 4-1 (Continued)

Run L-2

Stream (a)	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)	Relative Flow	Other Analyses
1AF	1	281.6	2.89	485	100	--
1AS	2	--	3.2	215	44	--
1CX	3	--	0.01	2073	427	--
1OS	4	--	--	165	34	0.3M Na ₂ CO ₃
1AX	5	0.8 x 10 ⁻³	--	1410	290	--
1SP	6	0.8 x 10 ⁻³	0.1	510	105	--
1AP	7	80.2	0.27	--	--	--
1AW	8	<0.4 x 10 ⁻³	2.5	--	--	--
1CU	9	67.6	0.19	--	--	--
1CW	10	0.1	0.014	--	--	--
10O	11	<0.4 x 10 ⁻³	--	--	--	--
10W	12	--	--	--	--	0.05M NO ₃ ⁻

(a) Samples taken at following conditions: 1A column, 1-in. amplitude and 88-cpm frequency; 1C column, 1-in. amplitude and 49-cpm frequency; 10 column, 1-in. amplitude and 64-cpm frequency.

TABLE 4-1 (Continued)

Run L-3

Stream ^(a)	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)	Relative Flow	Other Analyses
1AF	1	296.9	2.4	315 (545) ^(b)	100 (100)	--
1AS	2	--	3.2	150 (225)	47 (41)	--
1CX	3	--	0.012	1310 (2680)	415 (491)	--
1OS	4	--	--	115 (140)	37 (26)	0.28M Na ₂ CO ₃
1AX	5	<0.4 x 10 ⁻³	--	705 (1350)	223 (247)	--
1SP	6	--	0.07	235 (540)	75 (99)	28% TBP
1AP-1	7	94.2	0.21	--	--	--
1AP (-2)	7	84.3	0.23	--	--	--
1AW-1	8	0.5 x 10 ⁻³	2.40	--	--	--
1AW (-2)	8	0.5 x 10 ⁻³	2.58	--	--	--
1OW-1	12	--	--	--	--	0.085M NO ₃ ⁻
1OW-2	12	--	--	--	--	0.058M NO ₃ ⁻

(a) Pulser conditions for each set of samples:

Set 1, 1A column - 85% of flooding

Set 2, 1A column - 68% of flooding

(b) Flows in parentheses correspond to last half of run, and (-2) samples were taken at those flow conditions.

TABLE 4-1 (Continued)

Run L-4

Stream (a)	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)	Relative Flow	Other Analyses
1AF	1	307.2	3.2	315 (335) (a)	100 (100)	--
1AS	2	--	3.1	175 (165)	55 (50)	--
1CX	3	--	0.02	1350 (1320)	430 (395)	--
1OS	4	--	--	142 (45)	45 (15)	0.26M Na ₂ CO ₃
1AX	5	0.9 x 10 ⁻³	--	740 (740)	235 (220)	30.1% TBP
1SP	6	--	--	235 (250)	75 (75)	--
1AP-1	7	99.5	0.18	--	--	--
1AP (-2)	7	105.6	0.16	--	--	--
1AW-1	8	<0.4 x 10 ⁻³	2.85	--	--	--
1AW (-2)	8	0.5 x 10 ⁻³	2.85	--	--	--
1CU-1	9	69.2	0.2	--	--	--
1CU (-2)	9	79.1	0.2	--	--	--
1CW-1	10	0.9 x 10 ⁻³	0.024	--	--	--
1CW (-2)	10	5.3 x 10 ⁻³	0.024	--	--	--
1OW-1	12	0.01	--	--	--	2 g NO ₃ ⁻ /l
1OW (-2)	12	0.03	--	--	--	3.4 g NO ₃ ⁻ /l

(a) Pulser conditions for each set of samples:

Set 1, 1A column	1-in. amplitude, 96-cpm frequency
1C column, dual pulse	1-in. amplitude, 56-cpm frequency
	1-in. amplitude, 39-cpm frequency
10 column	1.94-in. amplitude, 50-cpm frequency
Set 2, 1A column, dual pulse	1-in. amplitude, 60-cpm frequency
	1.94-in. amplitude, 30-cpm frequency
1C column, dual pulse	1-in. amplitude, 54-cpm frequency
	1-in. amplitude, 40-cpm frequency

(b) Flows in parentheses correspond to second half of run, and (-2) samples were taken at those flow conditions.

TABLE 4-1 (Continued)

Run L-5

Stream (a)	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)	Relative Flow	Other Analyses
1AF-1	1	299	2.78	480	100	--
1AS	2	--	3.03	225	47	--
1CX	3	--	0.01	2760	575	--
1OS	4	--	--	150	31	0.25M Na ₂ CO ₃
1AX	5	--	--	1345	280	29.4% TBP
1SP	6	--	0.1	525	110	29.4% TBP
1AP-1	7	79.3	0.11	--	--	--
1AF (-2)	1	299	2.78	545	114	--
1AP (-2)	7	91.8	0.08	--	--	--
1AW-1	8	$<0.4 \times 10^{-3}$	2.17	--	--	--
1CU-1	9	54.0	0.11	--	--	--
1CW-1	10	0.17	0.01	--	--	--
1OW-1	12	2.65	--	--	--	5.6 g NO ₃ ⁻ /l
100-1	11	5×10^{-3}	--	--	--	--

(a) Pulser conditions for both sample sets:

1A column 1-in. amplitude, 60-cpm frequency
 1C column, 1-in. amplitude, 36-cpm frequency
 dual pulse 1-in. amplitude, 23-cpm frequency
 10 column 1.94-in. amplitude, 46-cpm frequency

TABLE 4-1 (Continued)

Run L-6

Stream (a)	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)	Relative Flow	Other Analyses
1AF	1	298	2.9	315	100	--
1AS	2	--	2.95	150	48	--
1CX	3	--	0.01	1230	390	--
1OS	4	--	--	35	11	0.23M Na ₂ CO ₃
1AX	5	--	--	960	304	29% TBP
1SP	6	--	--	Added to 1AX flow		--
1AP	7	95.9	0.14	--	--	--
1AW	8	0.6 x 10 ⁻³	2.9	--	--	--
1CU	9	72.8	0.14	--	--	--
1CW	10	3 x 10 ⁻³	0.009	--	--	--
10O	11	0.5 x 10 ⁻³	--	--	--	--
10W	12	0.04	--	--	--	0.05M NO ₃ ⁻

(a) Pulser conditions at sampling:

1A column 1.94-in. amplitude, 50-cpm frequency

1C column, 1-in. amplitude, 53-cpm frequency

dual pulse 1-in. amplitude, 36-cpm frequency

10 column 1.94-in. amplitude, 56-cpm frequency

Note: Diluent for TBP for all tests is normal paraffin hydrocarbon (NPH) from Southampton Oil Company, Silsbee, Texas.

TABLE 4-2
COLUMN CAPACITY DATA

Extraction Section

Run No.	Volume Velocity, Sum of Phases (gal/hr/ft ²)	Flooding Frequency (cpm)	Pulse Amplitude (in.)	Cartridge ^(a)
L-1	1040	112	1	1/8-in. nozzles; 23% free area; 2-in. spacing
L-2	1825	107	1	1/8-in. nozzles; 23% free area; 2-in. spacing
L-3	1020	86	1.94	1/8-in. nozzles; 23% free area; 2-in. spacing
L-4	1060	118	1	3/16-in. nozzles; 23% free area; 2-in. spacing
L-5	1825	110	1	3/16-in. nozzles; 23% free area; 2-in. spacing
L-6	1045	63	1.94	3/16-in. nozzles; 23% free area; 2-in. spacing
	1030	66	1.94	3/16-in. nozzles; 23% free area; 2-in. spacing
	2030	54	1.94	3/16-in. nozzles; 23% free area; 2-in. spacing
	2050	52	1.94	3/16-in. nozzles; 23% free area; 2-in. spacing
L-7 ^(b)	1120	123	1	3/16-in. nozzles; 23% free area; 2-in. spacing

(a) Cartridge height was 18 ft in runs L-1 through L-6 and 6 ft in run L-7.

(b) Capacity data included for runs to be reported later to give more complete flooding data at this reporting.

TABLE 4-2 (Continued)

Solvent Wash

Run No.	Volume Velocity, Sum of Phases (gal/hr/ft ²)	Flooding Frequency (cpm)	Pulse Amplitude (in.)	Cartridge ^(a)
L-1	820 (Aq./Org. = 0.11)	100	1	1/8-in. nozzles; 23% free area; 2-in. spacing
L-2	1430 (Aq./Org. = 0.09)	90	1	1/8-in. nozzles; 23% free area; 2-in. spacing
L-3	1480 (Aq./Org. = 0.08)	63	1-15/16 (1.94)	1/8-in. nozzles; 23% free area; 2-in. spacing
L-4	815 (Aq./Org. = 0.14)	69	1.94	1/8-in. nozzles; 23% free area; 2-in. spacing
	740 (Aq./Org. = 0.04)	>69	1.94	1/8-in. nozzles; 23% free area; 2-in. spacing
L-5	1090 (Aq./Org. = 0.09)	63	1.94	3/16-in. nozzles; 23% free area; 3-in. spacing
L-6	No 10 column flooding data taken			
L-7 ^(b)	925 (Aq./Org. = 0.22)	65	1.94	3/16-in. nozzles; 23% free area; 3-in. spacing
L-8 ^(b)	770 (Aq./Org. = 0.11)	106	1	3/16-in. nozzles; 23% free area; 3-in. spacing
	1550 (Aq./Org. = 0.11)	95	1	3/16-in. nozzles; 23% free area; 3-in. spacing

(a) Cartridge height was 18 ft in all runs.

(b) Capacity data included for runs to be reported later to give more complete flooding data at this reporting.

STREAM NO.	STREAM DESIGNATION	PHASE	U (g/l)	HNO ₃ (M)	RELATIVE FLOW
1	1AF	AQ.	290	2.5	100
2	1AS	AQ.	—	3.0	48
3	1CX	AQ.	—	0.01	400
4	10S	AQ.	0.27M Na ₂ CO ₃		32
5	1AX	ORG.	30% TBP-NPH		240
6	1SP	ORG.	30% TBP-NPH		82
7	1AP	ORG.	90	0.2	322
8	1AW	AQ.	0.001	2.3	148
9	1CU	AQ.	72.5	0.16	400
10	1CW	ORG.	0.001	0.01	322
11	100	ORG.	30% TBP-NPH		322
12	10W	AQ.	{ 0.15M Na ₂ CO ₃ 0.1M NaHCO ₃ 0.1M NaNO ₃		32

Fig. 4-1. Nominal LWR Purex test flowsheet

Fig. 4-2. Extraction column flooding curve

Fig. 4-3. Uranium strip column flooding curve

Fig. 4-4. Solvent wash column flooding curve

APPENDIX
DISTRIBUTION LIST

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