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CONSOLIDATED FUEL RECYCLE PROGRAM

ANNULAR PULSE COLUMN DEVELOPMENT STUDIES INTERIM DEVELOPMENT REPORT

by
G. E. BENEDICT

Prepared under
Contract DE-AT03-76SF71053
for the San Francisco Operations Office
Department of Energy

DATE PUBLISHED: JULY 1979

GENERAL ATOMIC COMPANY

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GENERAL ATOMIC COMPANY

ABSTRACT

The capacity of critically safe cylindrical pulse columns limits the size of nuclear fuel solvent extraction plants because of the limited cross-sectional area of plutonium, U-235, or U-233 processing columns. Thus, there is a need to increase the cross-sectional area of these columns. This can be accomplished through the use of a column having an annular cross section. The preliminary testing of a pilot-plant-scale annular column has been completed and is reported herein.

The column is made from 152.4-mm (6-in.) glass pipe sections with an 89-mm (3.5-in.) o.d. internal tube, giving an annular width of 32 mm (1.25 in.). Louver plates are used to swirl the column contents to prevent channeling of the phases.

The data from this testing indicate that this approach can successfully provide larger-cross-section critically safe pulse columns. While the capacity is only 70% of that of a cylindrical column of similar cross section, the efficiency is almost identical to that of a cylindrical column. No evidence was seen of any nonuniform pulsing action from one side of the column to the other.

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1. SUMMARY

Preliminary testing of the 152.4-mm (6-in.) i.d. annular column for the General Atomic solvent extraction pilot plant was completed in the 22 extraction runs reported herein. The column internals consisted of standard 23% free area nozzle plates in combination with either 14% or 23% free area louver plates, which were used to provide swirling action around the annulus, thus preventing channeling of the two phases.

The main observations from the tests with the annular pulse column in the extraction mode are:

- With the internal plate configurations tested, the annular column had a flooding frequency about 70% to 80% as high as that of a 50.8-mm (2-in.) cylindrical column with standard nozzle plate internals. However, at 80% of flooding, the annular column efficiencies as noted from height of theoretical stage (HETS) calculations were almost identical to those of a cylindrical column.
- Significant changes in plate spacing did not produce significant changes in annular column capacity or efficiency.
- The louver plates of 14% or 23% free area did a more than adequate job of swirling the dispersed phase completely around the annulus from a single feed addition point.
- Column flooding always originated in the 23% free area nozzle plates rather than in the louver plates.

- No evidence was seen of any nonuniform pulsing action from one side of the column to the other.
- The top three plates in the annular column scrub section must be nozzle plates rather than louver plates to minimize entrainment in the solvent overflow (1AP) stream.

2. INTRODUCTION

Cylindrical pulse columns must be limited to a specific diameter as a criticality control measure when used to process fissile materials. This restriction on diameter limits the available cross section for these columns and will limit plant size unless multiple processing lines are installed on final fissile stream columns. Thus, there is a need to increase the cross-sectional area of columns for processing fissile materials. This can be accomplished through the use of a column having an annular cross section. The preliminary testing of a pilot-plant-scale annular pulse column has been completed and is reported herein.

3. REQUIREMENTS

The requirements for an annular pulse column necessary to allow its use in nuclear applications include the following:

1. Efficiencies comparable to those of cylindrical columns to allow a practical height limitation of 12.2 m (40 ft) to apply.
2. Capacities high enough to enable the annular column to significantly increase throughput over that achievable with critically safe cylindrical columns.
3. Operating characteristics similar to those of cylindrical columns with regard to entrainment, recovery from upset conditions, and control.

4. EQUIPMENT DESCRIPTION

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

1. Five glass cylindrical pulse columns of 50.8- and 76.2-mm (2- and 3-in.) diameter with overall heights up to 8.5 m (28 ft).
2. One glass annular pulse column of 152.4-mm (6-in.) diameter with a stainless steel center post of 89-mm (3.5-in.) diameter having an annulus of 31.75 mm (1.25 in.). The overall active height of the annular column is 2.75 m (9 ft).
3. One eight-stage Robatel centrifugal contactor.
4. Thirty-three stainless steel tanks ranging in capacity from 10 to 900 liters.
5. Feed and transfer pumps, intercolumn airlifts, flowmeters, and metering pumps.
6. Interface sensing and control instrumentation.
7. Pulse generators.
8. A thermosyphon evaporator fabricated from stainless steel with a boil-off capacity of about 1 liter per minute.
9. Heat exchangers.

The solvent extraction columns are assembled from sections of commercially available glass pipe. The columns and associated equipment are

shown in Figs. 4-1 and 4-2.* The annular column drawing and assembled picture are shown in Figs. 4-3 and 4-4. Typical annular column plate materials are shown in Fig. 4-5.

All of the tanks are 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 fluorocarbon bellows activated by variable throw cranks driven by variable speed motors. Pipe and flexible hose connect the bellows to the column bottoms.

The product concentrator is a thermosyphon unit constructed of all-stainless-steel material. 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 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

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

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.

Previous studies using this pilot plant are documented in Refs. 4-1 through 4-9.

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Fig. 4-1. Overall view of the solvent extraction pilot plant

Fig. 4-2. Centrifugal contactor unit

Fig. 4-4. Section of annular pulse column assembled with 23% free area louver plates

23% FREE AREA NOZZLE PLATE

14% FREE AREA LOUVER PLATE

Fig. 4-5. Annular pulse column plates

5. RESULTS AND DISCUSSION

The nominal flowsheet used in the annular column tests is shown in Fig. 5-1. The uranium strip column and solvent wash column flowsheets are included. The actual flowsheet conditions and stream analysis results are shown in Tables 5-1 and 5-2. The column capacity and efficiency data and the column internal configurations are given in Tables 5-3 and 5-4. A comparison of selected data for the annular column with data for a cylindrical column is shown in Table 5-5. This comparison indicates that the annular column capacity with 14% free area louver plates and 23% free area nozzle plates is only about 70% of the capacity in a cylindrical column using the same nozzle plates. The efficiencies of the two columns are almost the same at about 80% of flooding.

Tests on 23% free area louver plates in the same configuration with the 23% free area nozzle plates showed that the flooding points changed only slightly from those with 14% free area louver plates when using a 25.4-mm (1-in.) amplitude. Using a 50.8-mm (2-in.) amplitude increased the flooding points about 15% with the 23% free area louver plates.

The column efficiency was reduced only slightly (about 7%) with the 23% free area louver plates compared with the 14% free area louver plates.

Only small changes in efficiency and capacity have been noted when plate spacing was changed in the annular column.

These results suggest that the capacity of the annular column is being limited by side-wall effects in the 31.75-mm (1.25-in.) annulus. It is known that cylindrical columns of 31.75-mm (1.25-in.) diameter have a lower capacity per unit of cross section than 50.8-mm (2-in.) cylindrical columns.

The 14% and 23% free area louver plates both did a more than adequate job of swirling the dispersed phase completely around the column annulus from a single feed addition point.

No evidence was seen of any nonuniform pulsing action from one side of the column to the other.

Severe entrainment of scrub solution into the solvent overflow was noted when the top plate was a louver plate. It is recommended that at least three nozzle plates be used at the top of the scrub section, above the top louver plate, to coalesce the finely divided aqueous droplets.

TABLE 5-1
 FLOWSHEET CONDITIONS AND ANALYTICAL VALUES FOR ANNULAR COLUMN
 WITH 23% FREE AREA STANDARD NOZZLE PLATES
 AND 14% FREE AREA LOUVER PLATES

Run L-71

Stream	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)
1AF	1	284	2.67	1240
1AS	2	--	3.17	595
1AX	3	$<4 \times 10^{-4}$	29.28 vol % TBP	4380
1AW	4	0.0056		--
1AP	5	$86.8 \times 10.05^{(a)}$	0.234	--
1AO	12	0.2	0.77	--

Run L-73

1AF	1	293	2.46	1230
1AS	2	--	3.2	585
1AX	3	0.0006	--	4325
1AW	4	0.0004	--	--
1AP	5	$84.7 \times 10.5^{(a)}$	0.2	--
1AO	12	0.03	0.78	--

Run L-76

1AF	1	293	2.74	1620
1AS	2	--	3.2	710
1AX	3	$<4 \times 10^{-4}$	30.1 vol % TBP	5000
1AW	4	0.0007		--
1AP	5	$91.4 \times 1.05^{(a)}$	0.18	--
1AO	12	0.08	0.72	--

TABLE 5-1 (continued)

Run L-77

Stream	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)
1AF-1	1	294	2.95	1650
1AF-2	1	294	2.95	1650
1AX-1	2	0.0006	29.8 vol % TBP	5075
1AX-2	2	0.0006	29.8 vol % TBP	5180
1AS-1	3	--	3.2	715
1AS-2	3	--	3.2	720
1AW-1	4	0.001	2.7	--
1AW-2	4	0.0065	2.8	--
1AP-1	5	93.9 x 1.05 ^(a)	0.16	--
1AP-2	5	94.9 x 1.05 ^(a)	0.16	--
1AO-1	12	0.096	0.78	--
1AO-2	12	1.47	0.73	--

Run L-78

1AF	1	301	2.9	1570
1AX	2	$<4 \times 10^{-4}$	29.9 vol % TBP	5020
1AS	3	--	3.3	710
1AW	4	0.002	2.8	--
1AP	5	97.2 x 1.05 ^(a)	0.17	--
1AO	12	0.46	0.83	--

Run L-79

1AF	1	295	2.5	1650
1AX	2	4×10^{-4}	30.1 vol % TBP	4880
1AS	3	--	3.2	680
1AW	4	0.003	2.8	--
1AP	5	103 x 1.05 ^(a)	0.16	--
1AO	12	0.36	0.75	--

TABLE 5-1 (continued)

Run L-80

Stream	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)
1AF	1	303	2.9	1630
1AS	2	--	3.2	4920
1AX	3	$<4 \times 10^{-4}$	30.0 vol % TBP	690
1AW	4	0.002	1.75	Low HNO ₃ due to high entrainment of 1AS in 1AP
1AP	5	$93.9 \times 1.05^{(a)}$	0.17	--
1AO	12	0.034	0.54	--

Run L-81

1AF	1	295	2.9	1710
1AS	2	--	3.0	710
1AX	3	$<4 \times 10^{-4}$	29.7 vol % TBP	5050
1AW	4	0.001	2.7	--
1AP	5	$105 \times 1.05^{(a)}$	0.16	--
1AO	12	0.89	0.86	--

Run L-82

1AF	1	274	2.7	1730
1AS	2	--	3.0	670
1AX	3	$<4 \times 10^{-4}$	29.6 vol % TBP	5245
1AW	4	0.001	2.7	--
1AP	5	$87.7 \times 1.05^{(a)}$	0.16	--
1AO	12	0.7	0.72	--

(a) 1.05 factor on 1AP sample approximates reflux of uranium in scrub section.

TABLE 5-2
 FLOWSHEET CONDITIONS AND ANALYTICAL VALUES FOR ANNULAR COLUMN
 WITH 23% FREE AREA STANDARD NOZZLE PLATES
 AND 23% FREE AREA LOUVER PLATES

Run L-87

Stream	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)
1AF	1	295	2.8	1600
1AS	2	--	3.0	720
1AX	3	--	29.67 vol % TBP	5045
1AW	4	0.0026	2.6	--
1AP	5	92 x 1.05 ^(a)	0.18	--
1AO	12	1.0	--	--

Run L-88

1AF	1	296	2.7	1535
1AS	2	--	3.2	710
1AX	3	--	29.97 vol % TBP	5000
1AW	4	0.0037	2.7	--
1AP	5	92 x 1.05 ^(a)	0.18	--
1AO	12	1.1	--	--

Run L-89

1AF	1	285	3.2	1640
1AS	2	--	3.2	740
1AX	3	--	30.0 vol % TBP	5050
1AW	4	0.0027	3.1	--
1AP	5	92.8 x 1.05 ^(a)	0.18	--
1AO	12	0.7	--	--

TABLE 5-2 (continued)

Run L-90

Stream	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)
1AF	1	296	3.3	1575
1AS	2	--	3.3	695
1AX	3	--	29.8 vol % TBP	5025
1AW	4	0.0029	3.1	--
1AP	5	90.8 x 1.05 ^(a)	0.19	--
1AO	12	1.48	--	--

Run L-91

1AF	1	309	2.5	1640
1AS	2	--	3.2	730
1AX	3	--	29.95 vol % TBP	5040
1AW	4	0.005	2.9	--
1AP	5	105 x 1.05 ^(a)	0.18	--
1AO	12	0.63	--	--

Run L-92

1AF	1	322	2.8	1640
1AS	2	--	3.1	730
1AX	3	--	29.7 vol % TBP	4980
1AW	4	0.0036	2.9	--
1AP	5	98 x 1.05 ^(a)	0.16	--
1AO	12	0.32	0.59	--

TABLE 5-2 (continued)

Run L-93

Stream	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)
1AF	1	286	3.1	2300
1AS	2	--	3.0	990
1AX	3	--	--	7120
1AW	4	0.004	2.9	--
1AP	5	91.8 x 1.05 ^(a)	0.3	--
1AO	12	0.45	--	--

Run L-94

1AF	1	294	3.3	2270
1AS	2	--	3.2	1020
1AX	3	--	--	5045
1AW	4	0.0046	3.1	--
1AP	5	88.4 x 1.05 ^(a)	0.4	--
1AO	12	0.70	--	--

Run L-95

1AF	1	301	2.5	2240
1AS	2	--	3.5	980
1AX	3	--	30 vol % TBP	6580
1AW	4	0.13	2.7	--
1AP	5	99.7 x 1.05 ^(a)	0.2	--
1AO	12	2.7 - - -	--	--

TABLE 5-2 (continued)

Run L-106

Stream	Stream No.	U (g/l)	HNO ₃ (M)	Flow (ml/min)
1AF	1	306	2.7	1610
1AS	2	--	3.02	700
1AX	3	--	30 vol % TBP	4950
1AW	4	0.003	2.6	--
1AP	5	97.2 x 1.05 ^(a)	0.14	--
1AO	12	0.99	0.8	--

Run L-107

1AF	1	283	1.73	2070
1AS	2	--	2.77	1020
1AX	3	--	30 vol % TBP	6830
1AW	4	0.01	1.85	--
1AP	5	93.8 x 1.05 ^(a)	0.2	--
1AO	12	2.1	0.42	--

Run L-108

1AF	1	306	3.0	1680
1AS	2	--	3.2	730
1AX	3	--	29.7 vol % TBP	5165
1AW	4	0.01	2.7	--
1AP	5	104 x 1.05 ^(a)	0.13	--
1AO	12	5.5	0.8	--

(a) 1.05 factor on 1AP sample approximates reflux of uranium in scrub section.

TABLE 5-3
HETS DATA FOR ANNULAR COLUMN WITH 23% FREE AREA STANDARD NOZZLE PLATES AND 14% FREE AREA LOUVER PLATES

Run No.	HETS [m (ft)]			Superficial Velocity		Volume Velocity (gal/hr/ft ²)	Column Configuration(a)	Sample Percent of Flooding	Pulse Amplitude [mm (in.)]	Sample Pulse Frequency (cpm)	Flooding Frequency x Amplitude Ratio, Annular/Cylindrical
	Top 0.91 m (3 ft)	Bottom 0.91 m (3 ft)	Total Column	$\overline{V}_a$	$\overline{V}_o$						
				(mm/s)	(mm/s)						
L-71	0.46 (1.5)	0.75 (2.45)	0.61 (2.0)	2.54	6.0	760	A	80	50.8 (2)	41	0.81
L-73	0.37 (1.2)	0.55 (1.8)	0.49 (1.6)	2.5	6.0	750	A	79	25.4 (1)	67.5	0.67
L-76	0.4 (1.3)	0.52 (1.7)	0.47 (1.55)	3.25	6.95	900	A	79	25.4 (1)	62	0.64
L-77-1	0.4 (1.3)	0.53 (1.75)	0.49 (1.6)	3.28	7.03	910	A	79	25.4 (1)	62	0.64
L-77-2	0.49 (1.6)	0.49 (1.6)	0.61 (2.0)	3.28	7.11	920	A	61	25.4 (1)	48	0.64
L-78	0.46 (1.5)	0.49 (1.6)	0.49 (1.6)	3.17	6.9	890	A	80	25.4 (1)	55	0.56
L-79	0.41 (1.35)	0.52 (1.7)	0.49 (1.6)	3.22	6.72	880	A	81	50.8 (2)	37	0.61
L-80	0.32 (1.05)	0.91 (3.0)	0.50 (1.65)	3.2	6.9	890	B	80	50.8 (2)	45	0.90
L-81	0.46 (1.5)	0.46 (1.5)	0.46 (1.5)	3.3	6.9	905	C	80	50.8 (2)	39	0.79
L-82	0.46 (1.5)	0.46 (1.5)	0.49 (1.6)	3.3	7.15	925	C	81	25.4 (1)	60	0.62

(a) Column configuration for 2.74-m (9-ft) total length (from bottom to top), 14% free area louver plates, and 23% free area nozzle plates:

- A - 2 louver plates, 5 nozzle plates, 1 louver plate, 5 nozzle plates, 1 louver plate, 5 nozzle plates, 2 louver plates, feed point, 6 nozzle plates, 1 louver plate, 5 nozzle plates, 1 louver plate, 3 nozzle plates, 2 louver plates. All plate spacing was 50.8 mm (2 in.) except for the top two louver plates, which had 25.4-mm (1-in.) spacing.
- B - 5 nozzle plates on 50.8-mm (2-in.) spacing, 101.6-mm (4-in.) space, 1 louver plate, 101.6-mm (4-in.) space, 6 nozzle plates on 50.8-mm (2-in.) spacing, 101.6-mm (4-in.) space, 1 louver plate, 101.6-mm (4-in.) space, 6 nozzle plates on 50.8-mm (2-in.) spacing, 101.6-mm (4-in.) space, 1 louver plate, feed point, 152.4-mm (6-in.) space, 1 louver plate, 154.2-mm (6-in.) space, 1 louver plate, 152.4-mm (6-in.) space, 1 louver plate, 152.4-mm (6-in.) space, 1 louver plate, 203.2-mm (8-in.) space, 1 louver plate.
- C - Same as B from bottom of column to feed point, then 3 nozzle plates on 50.8-mm (2-in.) spacing, 101.6-mm (4-in.) space, 1 louver plate, 101.6-mm (4-in.) space, 3 nozzle plates on 50.8-mm (2-in.) spacing, 101.6-mm (4-in.) space, 1 louver plate, 101.6-mm (4-in.) space, 3 nozzle plates on 50.8-mm (2-in.) spacing.

TABLE 5-5
COMPARISON OF SELECTED CAPACITY AND EFFICIENCY DATA FOR CYLINDRICAL PULSE COLUMN [50.8-MM (2-IN. I.D.)]
AND ANNULAR PULSE COLUMN [152.4-MM (6-IN.) I.D.], 31.8-M (1.25-IN.) ANNULUS

Run No.	Superficial Velocity		Volume Velocity (gal/hr/ft ²)	Pulse Amplitude [mm (in.)]	1AP (U g/l)	HETS [m (ft)]			Percent of Flooding	Flooding Frequency x Amplitude [m/min (in./min)]
	$\overline{V}_a$ (mm/s)	$\overline{V}_o$ (mm/s)				Top 0.91 m (3 ft)	Bottom 0.91 m (3 ft)	Total Column		
Cylindrical Column										
L-7-5	4.2	8.45	1120	50.8 (2)	104	0.46 (1.5)	0.49 (1.6)	0.49 (1.6)	75	3.05 (120)
L-8-1	3.96	7.68	1030	25.4 (1)	108	0.50 (1.65)	0.49 (1.6)	0.55 (1.8)	75	3.05 (120)
L-49-1	3.7	8.2	1050	50.8 (2)	99.7	0.38 (1.25)	0.53 (1.75)	0.47 (1.55)	81	3.0 (118.5)
L-72-1	3.9	8.1	1065	25.4 (1)	102	0.4 (1.3)	0.49 (1.6)	0.47 (1.55)	80	3.0 (118.5)
Average	3.94	8.11	1065		103.4	0.43 (1.42)	0.5 (1.64)	0.49 (1.62)	77.8	3.03 (119.3)
Annular Column, 14% Louver Plates										
L-76	3.25	6.95	900	25.4 (1)	95	0.4 (1.3)	0.52 (1.7)	0.47 (1.55)	79	1.99 (78.5)
L-82	3.3	7.15	925	25.4 (1)	92	0.46 (1.5)	0.46 (1.5)	0.49 (1.6)	81	1.88 (74)
L-79	3.22	6.72	880	50.8 (2)	108	0.41 (1.35)	0.52 (1.7)	0.49 (1.6)	81	2.3 (91)
L-81	3.3	6.9	905	50.8 (2)	110	0.46 (1.5)	0.46 (1.5)	0.46 (1.5)	80	2.48 (97.5)
Average	3.27	6.93	900		101.3	0.43 (1.41)	0.49 (1.60)	0.475(1.56)	80	2.17 (85.3)
Annular Column, 23% Louver Plates										
L-87	3.2	7.0	900	25.4 (1)	97	0.47 (1.55)	0.47 (1.55)	0.55 (1.8)	80	2.16 (85)
L-90	3.1	7.0	890	50.8 (2)	95	0.49 (1.6)	0.47 (1.55)	0.53 (1.75)	79	2.82 (111)
L-91	3.25	6.95	905	50.8 (2)	110	0.46 (1.5)	0.53 (1.75)	0.50 (1.65)	80	2.79 (110)
L-92	3.25	6.9	895	25.4 (1)	103	0.46 (1.5)	0.55 (1.8)	0.52 (1.7)	80	2.03 (80)
L-106	3.2	6.9	890	50.8 (2)	102	0.47 (1.55)	0.47 (1.55)	0.50 (1.65)	79	2.72 (107)
L-108	3.3	7.1	925	25.4 (1)	109	0.50 (1.65)	0.52 (1.7)	0.49 (1.6)	75	2.0 (78.4)
Average	3.22	6.98	900		103	0.475(1.56)	0.50 (1.65)	0.515(1.69)	79	2.42 (95.23)

Fig. 5-1. Flowsheet for annular column tests

6. FUTURE WORK

Future annular pulse column studies should include:

1. Extraction testing with more dilute feeds to verify satisfactory operation with a variety of aqueous-to-organic flow ratios.
2. Stripping tests to verify satisfactory results with aqueous continuous stripping operation.
3. Design of a full-scale unit based on the above and current studies complete with criticality analysis to determine optimum annulus width.
4. Fabrication, installation, and testing of the full-scale extraction, scrub, and stripping annular columns to provide a verified design for use in solvent extraction purification of fissile materials in large-cross-section critically safe equipment.