



Equipment, Methods, and Techniques

AEC Research and Development Report

HYDRAULIC CHARACTERISTICS OF A  
CONTINUOUS SOLVENT WASHER  
WITH CRITICALLY SAFE DIMENSIONS

by

H. J. Clark and A. Tournas

Separations Engineering Division

March 1960

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EQUIPMENT, METHODS, AND TECHNIQUES  
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HYDRAULIC CHARACTERISTICS OF A CONTINUOUS SOLVENT  
WASHER WITH CRITICALLY SAFE DIMENSIONS

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#### ABSTRACT

The capacity of a continuous solvent washer with critically safe dimensions was found to be 0.5 gallon per minute when either 6% or 30% tributyl phosphate in kerosene was washed with a 1 molar solution of sodium carbonate.

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# HYDRAULIC CHARACTERISTICS OF A CONTINUOUS SOLVENT WASHER WITH CRITICALLY SAFE DIMENSIONS

## INTRODUCTION

Economical processing of spent reactor fuel elements by solvent extraction requires that the solvent be recycled. The tributyl phosphate - kerosene solvent used in Purex plants contains both fission products and contaminants, the latter resulting from degradation of solvent during the extraction operation. One method of removing these contaminants consists of washing the solvent with a solution of sodium carbonate<sup>(1,2)</sup>. The low concentration of contaminants and their irreversible extraction into aqueous carbonate permit the washing of several hundred volumes of solvent with each volume of wash solution. The advantage of this ratio can be realized when the solvent is continuously fed to and withdrawn from a tank containing the wash solution<sup>(3,4)</sup>. Extrainment of the wash solution in the solvent must be low to avoid recontamination of the solvent and to permit continuous operation without loss of the wash solution.

The present work was undertaken to establish the capacity of a solvent washer suitable for use in a processing plant for enriched nuclear fuels. On the basis of nuclear data from the Oak Ridge National Laboratory<sup>(5)</sup>, the washer has "always safe" dimensions for partially reflected systems. Washers of a similar design were described at the 1958 Geneva Conference<sup>(6)</sup> and in an earlier report from the Oak Ridge National Laboratory<sup>(7)</sup>.

## SUMMARY AND CONCLUSIONS

The capacity of a critically safe washer, 7 inches in diameter by 9 feet, 8 inches high, was determined for washing a solvent consisting of tributyl phosphate dissolved in "Ultrasene", a refined kerosene. The unit was operated satisfactorily at a solvent feed rate of 0.5 gallon per minute with a wash solution of 1 molar sodium carbonate. The tip speed of the paddle was 4.8 feet per second and the temperature was maintained at 90-100°F. The capacity for 6% TBP-"Ultrasene" was only slightly greater than for 30% TBP-"Ultrasene".

Increased temperature permitted operation at a higher paddle tip speed but at no increase in capacity. The feed rate could be increased to 0.9 gallon per minute by decreasing the paddle tip speed to 3.8 feet per second, but only at a great sacrifice in the pumping capacity of the paddle and hence a postulated drop in washing efficiency. It was found necessary to maintain the aqueous liquid level above the shrouded paddle for satisfactory operation.

## DISCUSSION

### EQUIPMENT

The washer, shown in Figure 1, was a stainless steel tank 7 inches in diameter and 9 feet, 8 inches tall, which had a concentric core. The core, Figure 2, was a 2.75-inch paddle, fitted with a top plate and concentric within a vaned stator, or shroud. A 3-inch draft tube extended from the shroud to near the bottom and to the top of the tank. The solvent feed line discharged into the lower end of the draft tube. The 1-1/2-inch paddle shaft extended through the upper end of the draft tube to a variable speed motor. The paddle was rotated so that the solution discharged parallel to the vanes at the shroud. A 1-1/2-inch-diameter flow control orifice was located in the draft tube 2 inches below the paddle. Sample taps were installed at the shroud discharge, at levels 1, 2, and 3 feet above the shroud, at levels 1, 2, 3, and 4 feet below the shroud, at the lower end of the draft tube, at the flow control orifice, and on the solvent discharge line. The volume of the washer to the solvent outlet pipe was 16-1/2 gallons, and the volume to the top of the shroud was 9.9 gallons. A heating coil was wound around the lower end of the tank. Accessory equipment included feed and catch tanks with pumping and sampling facilities.

In normal operation the aqueous wash charge was recirculated with solvent up the draft tube and down the outer annulus. The incoming solvent, after discharge from the shroud, partially recycled with the wash and partially overflowed after coalescing in the upper 4 feet of the unit.

### EXPERIMENTAL PROCEDURE

Sufficient wash solution was added to the washer to give the desired operating liquid level, which was 9.9 gallons for the 62-inch level necessary to cover the shroud and 11.7 gallons for a 74-inch level. Flow was started by pumping solvent from the feed tank. At the first sign of overflow from the washer the solvent feed was stopped and the agitator was started. After 15 minutes of operation under these conditions, the solvent flow was restarted at a specified rate. All runs were continued for 4 hours to ensure that steady-state operation had been attained. Samples were taken from the washed solvent at the overflow and from the various points within the washer. The solution was heated, when necessary, by passing steam through the heating coil. At the end of each run, the wash solution was drained and its volume was measured to determine the amount lost.

## RESULTS

Satisfactory operation of the washer was obtained at a solvent feed rate of 0.5 gallon per minute when either 30% or 6% TBP in kerosene was used (Appendix, Runs 1 and 5). A 1 molar solution of sodium carbonate was used as the wash.

The wash volume was sufficient to just cover the shroud and the temperature was maintained at 90-100°F. The paddle tip speed was 4.8 feet per second. Operation at the higher liquid interface level of 74 inches with a 30% TBP feed resulted in a loss of aqueous until the level had been reduced to about 62 inches, at which level operation again became satisfactory (Run 3). Operation at the liquid level of 74 inches with a 6% TBP feed was satisfactory (Run 6).

Operation at feed rates higher than 0.5 gallon per minute resulted in loss of the wash solution (Runs 2 and 10). The higher feed rates reduced the residence time of the solvent in the settling zone above the shroud and allowed entrainment of the wash solution. This loss of wash solution usually occurred for only the first hour, after which the increase in residence time decreased the entrainment.

Runs made at reduced temperature also resulted in excessive entrainment with loss of wash solution. The lower temperature reduced the rate of coalescence, and hence increased the residence time required in the settling zone; conversely, it would be expected that higher temperatures would result in a higher feed rate. A run made at 120°F showed that a solvent feed rate of 0.95 gallon per minute was too high (Run 11). A rate somewhere between 0.5 and 0.9 gallon per minute can probably be obtained at the higher temperature.

Reduction in the tip speed of the agitator permitted a higher solvent feed rate (Run 8). The normal tip speed of 4.8 feet per second gave a pumping rate of approximately 7 gallons per minute through the draft tube. Decreasing the tip speed to 3.8 feet per second allowed an increase of feed rate to 0.9 gallon per minute, but only at the expense of reducing the pumping to 1.3 gallons per minute. The loss of pumping would reduce the washing efficiency to an unacceptable level. Operation of the washer at a higher tip speed, 6.6 feet per second, gave a slight increase in pumping and hence a slight increase in washing efficiency but required a high temperature to minimize extrainment (Run 7).

The pumping or recirculation rate through the washer shroud was shown to be dependent upon the volume of aqueous in the unit. A high recirculation rate was necessary to ensure recycle of solvent. A low aqueous interface limited the pumping of aqueous up the draft tube. A manometer effect existed whereby the lifting of aqueous in the draft tube lowered the level outside this tube. The resultant pressure differential reduced the pumping capacity of the system. For this reason the aqueous level must be maintained above the shroud.

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## BIBLIOGRAPHY

1. Flanary, J. R., H. J. Groh, L. E. Line, and V. J. Reilly. Purex Process Laboratory Development Progress Report - August 1952 Through January 1953. Oak Ridge National Laboratory Oak Ridge, Tenn. AEC Research and Development Report ORNL-1548, 50 pp. (July 1953) (declassified March 1957)
2. Holmes, J. H. and A. S. Jennings. Purex Solvent Recovery; A Summary of KAPL Purex Solvent Wash Data. Knolls Atomic Power Laboratory, Schenectady, N. Y. AEC Research and Development Report KAPL-820, 29 pp. (1952) (Secret).
3. Kishbaugh, A. A. Continuous Solvent Washing - Hydraulic Characteristics of Large Units. E. I. du Pont de Nemours and Co., Aiken, S. C. AEC Research and Development Report DP-333, 17 pp. (1959)
4. Occhipinti, E. S. Performance of a Continuous Washer for Purex Solvent. E. I. du Pont de Nemours and Co., Aiken, S. C. AEC Research and Development Report DP-305, 17 pp. (1958).
5. Applied Nuclear Physics Division Annual Progress Report for Period Ending September 1, 1957. Oak Ridge National Laboratory, Oak Ridge, Tenn. AEC Research and Development Report ORNL-2389, 283 pp. (1957)
6. Colven, T. J. "Critically Safe Equipment for Aqueous Separations Processes". Proc. U. N. Intern. Conf. Peaceful Uses Atomic Energy, 2nd, Geneva, 17, 555-63 (1958). P/518
7. Flanary, J. R., et al. Chemical Development of the 25-TBP Process. Oak Ridge National Laboratory, Oak Ridge, Tenn. AEC Research and Development Report ORNL-1993 (Rev.) 72 pp. (April 1957) (declassified with deletions September 1957).

**APPENDIX  
SUMMARY OF ENTRAINMENT DATA**

Run Conditions	Run Number										
	1	2	3	4	5	6	7	8	9	10	11
Duration, hr	4	4	4	4	4	4	4	4	4	4	4
Interface, inch	62	62	74	62	62	74	62	62	62	62	62
Na <sub>2</sub> CO <sub>3</sub> , M	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.2	1.0	1.0	1.0
TBP, %	30	30	30	30	6	6	6	6	6	6	6
Temp., °F	91	96	100	83	99	97	125	106	81	97	120
Feed rate, gpm	0.48	0.56	0.47	0.48	0.47	0.51	0.47	0.90	0.45	0.67	0.95
Paddle tip speed, fps	4.8	4.8	4.8	4.8	4.8	4.8	6.6	3.8	4.8	4.8	4.8
<b>Results, % aqueous at points in washer</b>											
Effluent	0.03	2	6(a)	2	0.01	0.01	0.02	0.2	0.02	8	4(a)
3 feet above shroud	48	44	46	45	0.01	47	0.02	17	40	32	26
2 feet above shroud	60	54	79	53	47	78	-	-	-	30	-
1 foot above shroud	74	56	80	57	80	74	-	-	-	25	-
Shroud exit	70	53	73	52	74	73	72	32	73	33	50
1 foot below shroud	70	52	72	55	75	73	-	-	-	25	-
2 feet below shroud	70	51	75	55	72	70	-	-	-	25	-
3 feet below shroud	69	51	73	50	72	69	68	99	-	25	98
4 feet below shroud	70	52	74	57	73	70	72	99	63	25	98
Draft tube	86	56	79	56	79	78	76	99	77	29	98
Na <sub>2</sub> CO <sub>3</sub> lost, gal.	0	2	1	2	0	0	0	0	0.5(e)	5	1
A/O ratio at shroud	2.3	1.1	2.7	1.1	2.8	2.8	2.6	0.5	2.8	0.5	1
Pumping rate, gpm	7	7(b)	6	6	7	7	9	1.3	8	(d)	1.5

- (a) Average - loss actually high for 1/2-1 hr then dropped to 0.01%  
 (b) Calculated from beginning of run figures before loss upset unit  
 (c) Loss occurred only during first few minutes of operation then dropped to the 0.02% shown in effluent  
 (d) High loss obscures all data making pump rate calculation impossible

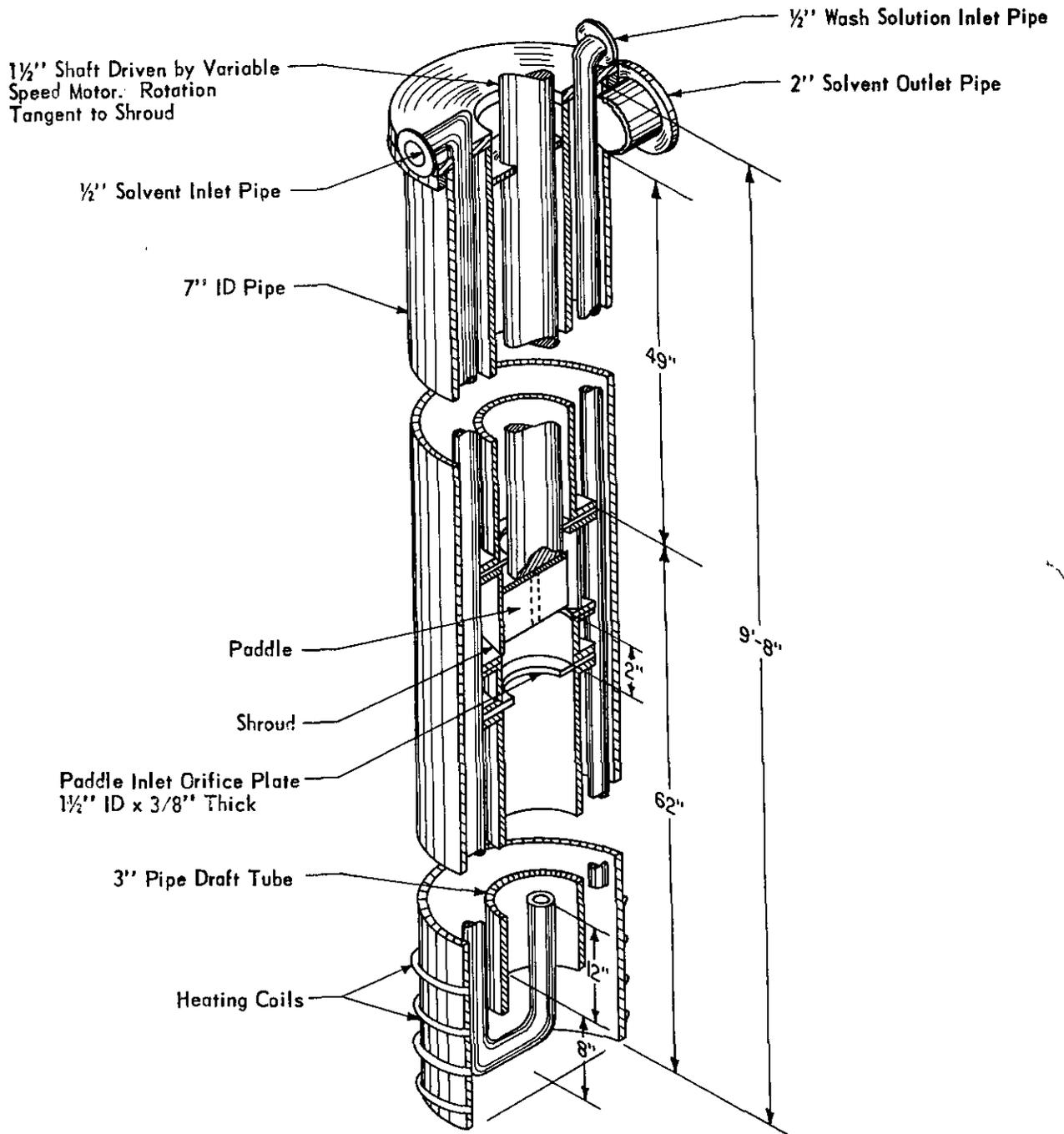


FIG. 1 CONTINUOUS SOLVENT WASHER

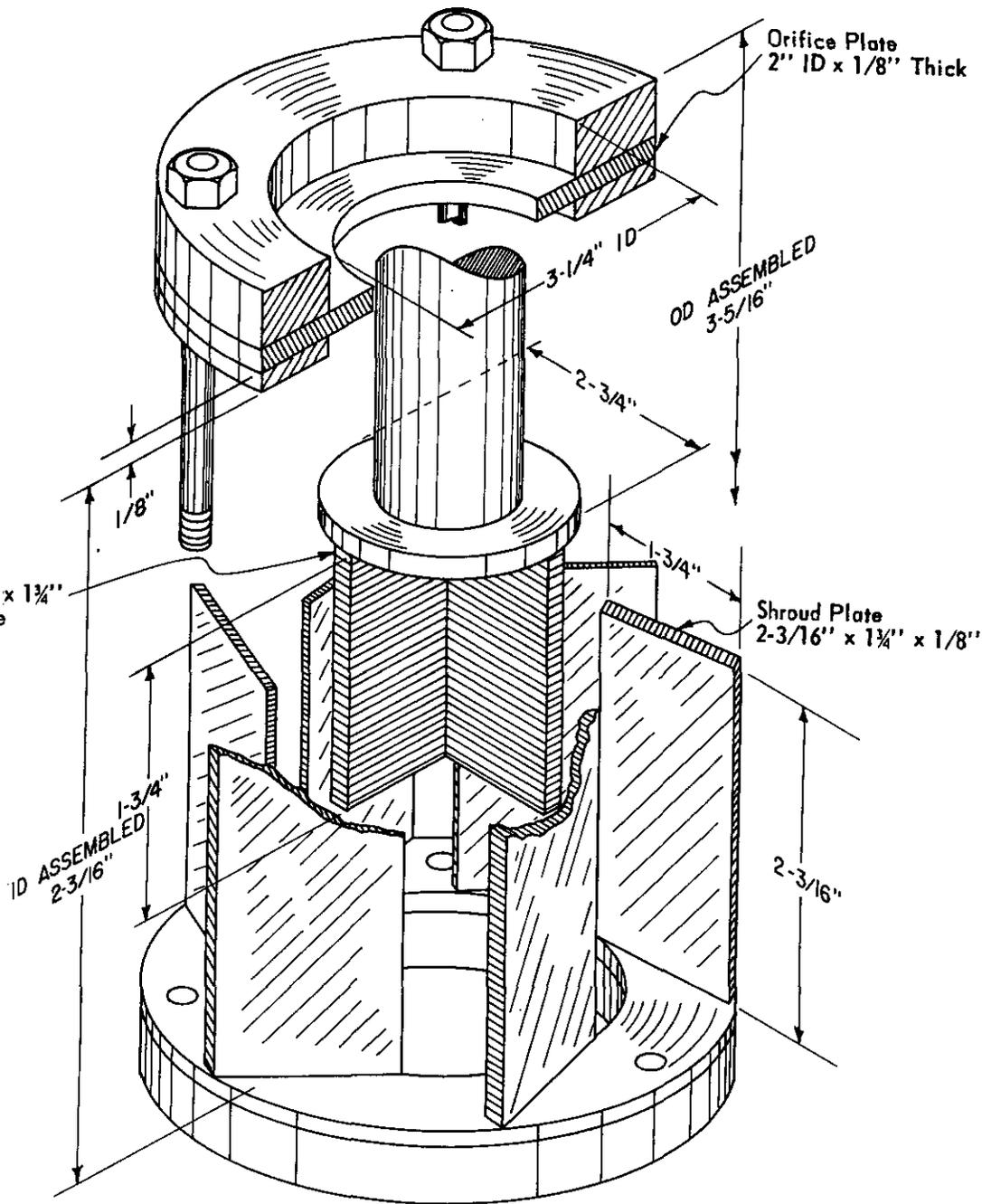


FIG. 2 DETAIL OF SHROUD FOR CONTINUOUS SOLVENT WASHER