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PERFORMANCE OF A CENTRIFUGAL MIXER-SETTLER AS A PHASE SEPARATOR

C. B. GOODLETT

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Savannah River Laboratory

Aiken, South Carolina

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PERFORMANCE OF A CENTRIFUGAL MIXER-SETTLER AS A PHASE SEPARATOR

by

C. B. Goodlett

Approved by

D. S. Webster, Research Manager
Separations Engineering Division

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E. I. DU PONT DE NEMOURS & COMPANY
SAVANNAH RIVER LABORATORY
AIKEN, S. C. 29801

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ABSTRACT

A single centrifugal mixer-settler used as a separator provides endstreams containing less than 0.5% entrainment when fed a thoroughly mixed emulsion at feed rates up to 25-30 gpm. The capacity of the unit exceeds 40 gpm if the phases are partially settled prior to feeding the separator.

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INTRODUCTION

The "rerun station" in either of the two separations areas of the Savannah River Plant (SRP) can extract specific products from high activity waste concentrate from various solvent extraction processes. For the greatest operating convenience, the processing rate should equal the waste generation rate. However, this equivalence is seldom realized because of the formation of emulsions that cannot be separated by the conventional gravity decanter. The emulsions are stabilized by surface active agents such as dibutyl phosphate, a decomposition product of the tributyl phosphate (TBP) in the solvent. In the laboratory, these emulsions separate more slowly at exposures as low as ~15 watt-hr/liter of solvent, and stable emulsions form when solvent exposure reaches 300 watt-hr/liter.

Most emulsions encountered in the plant can be separated in a centrifugal field. A tested centrifuge design[1] was available, and an 18-stage bank of centrifugal mixer-settlers has performed well since October 1966 in the extraction-scrub service of the Purex process.[2] Therefore, to provide higher capacity and simpler operation of the rerun stations for present and projected processes, a single 10-inch-diameter centrifugal mixer-settler was installed in the H-Area separations plant, for initial use as a separator in batch-extraction processes. This report describes the performance of the unit prior to installation.

SUMMARY

A single-stage centrifugal mixer-settler used as a high capacity separator was able to separate 25-30 gpm of a thoroughly mixed emulsion into phases containing less than 0.5% entrainment. When the feed consisted of partially settled phases, the capacity of the unit exceeded 40 gpm.

During initial tests the hydraulic capacity of the unit was limited by entrained air in the aqueous exit stream; venting and increasing the size of the exit chamber eliminated this problem.

The operating characteristics of a feed jet (OF-5) were measured for several different feed materials.

The unit is presently operating satisfactorily on process solutions in the plant.

DISCUSSION

PLANT INSTALLATION

In the plant, the centrifugal mixer-settler (used as a phase separator) is installed parallel to the gravity decanter (Fig. 1), and shares the same process piping. A pneumatic diversion weir sends the aqueous stream from the separator to either the product or waste tank (to eliminate cross-contamination that would result from using a common tank). The gravity decanter serves now only as an emergency backup. The content of the separator (4-gallons) drains to the feed tank when the operation is stopped, whereas the 100-gallon content of the decanter must be removed by jetting. A variable rate jet feeds the unit.

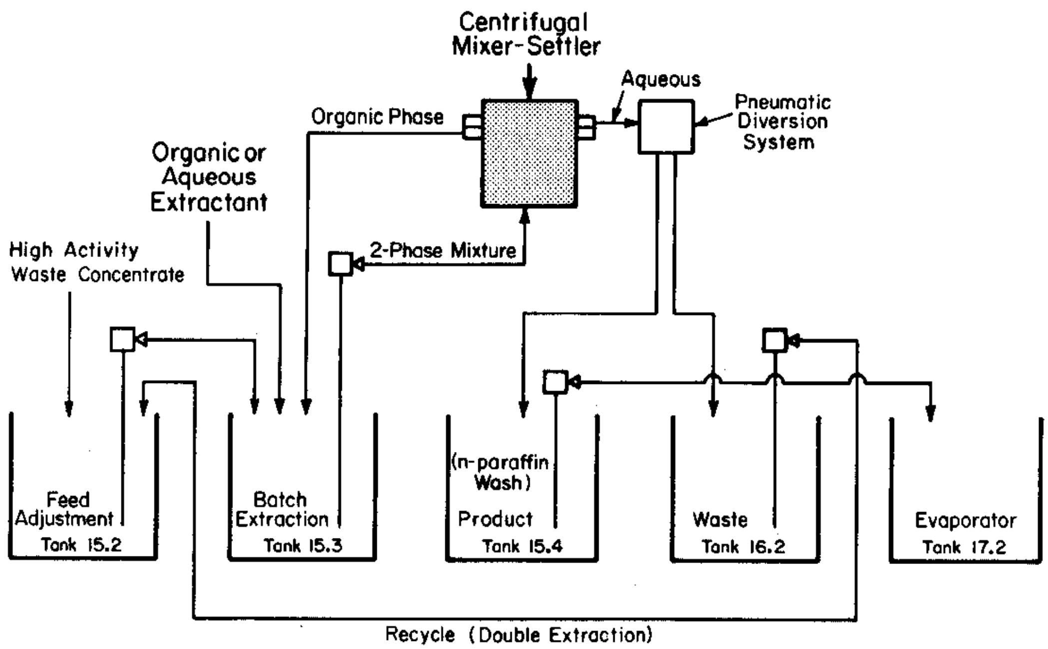


FIG. 1 SINGLE-STAGE CENTRIFUGAL MIXER-SETTLER IN RERUN STATION

The separator (Fig. 2) is identical with each of the 18 mixer-settler stages that have been operating smoothly as the Purex 1A bank in F Area since October 1966, except that only one feed line is provided; a second feed line can be added if the equipment is to be used as a mixer-settler rather than as a simple decanter. Information pertaining to development of the centrifugal mixer-settler is presented in References 3 through 7.

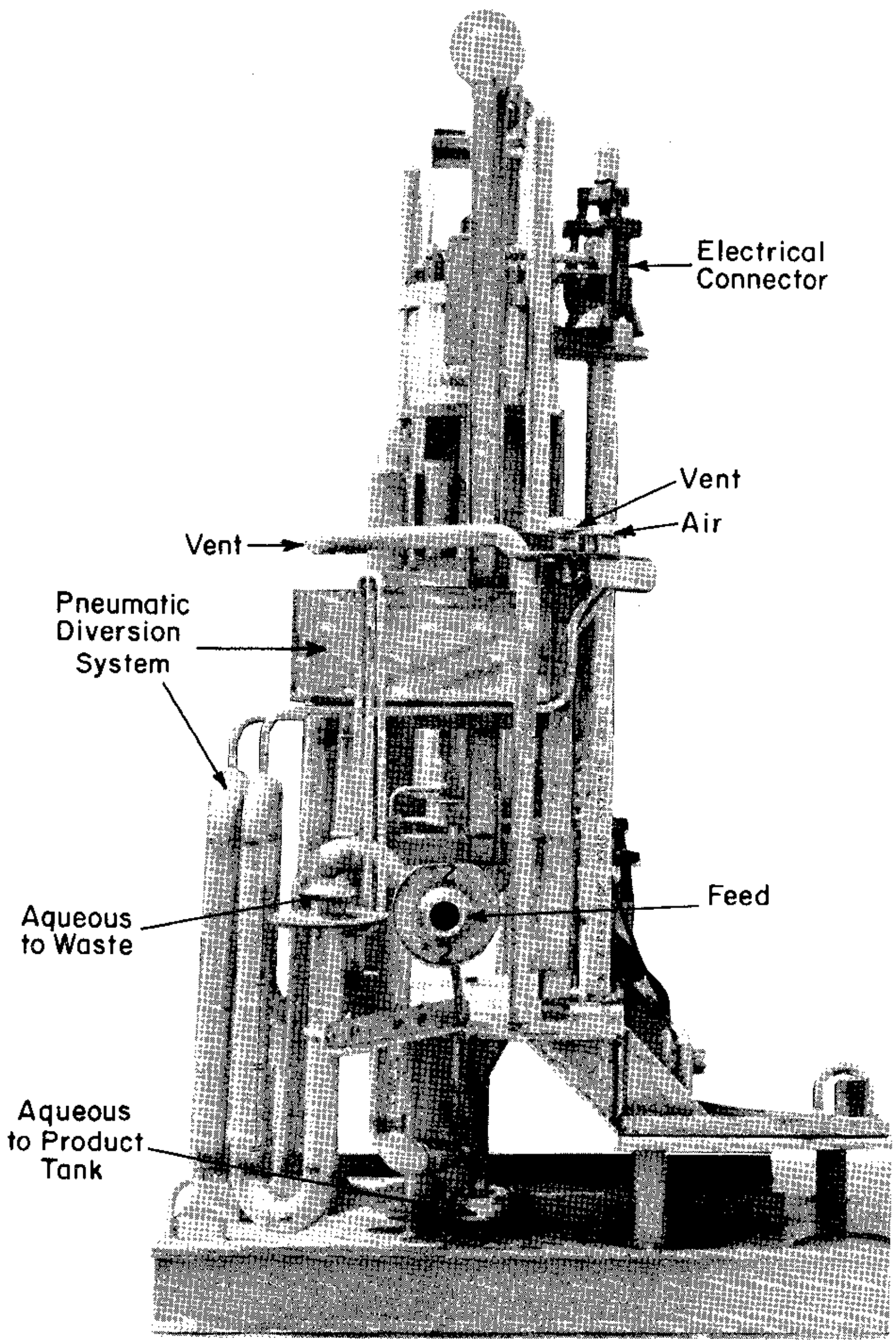
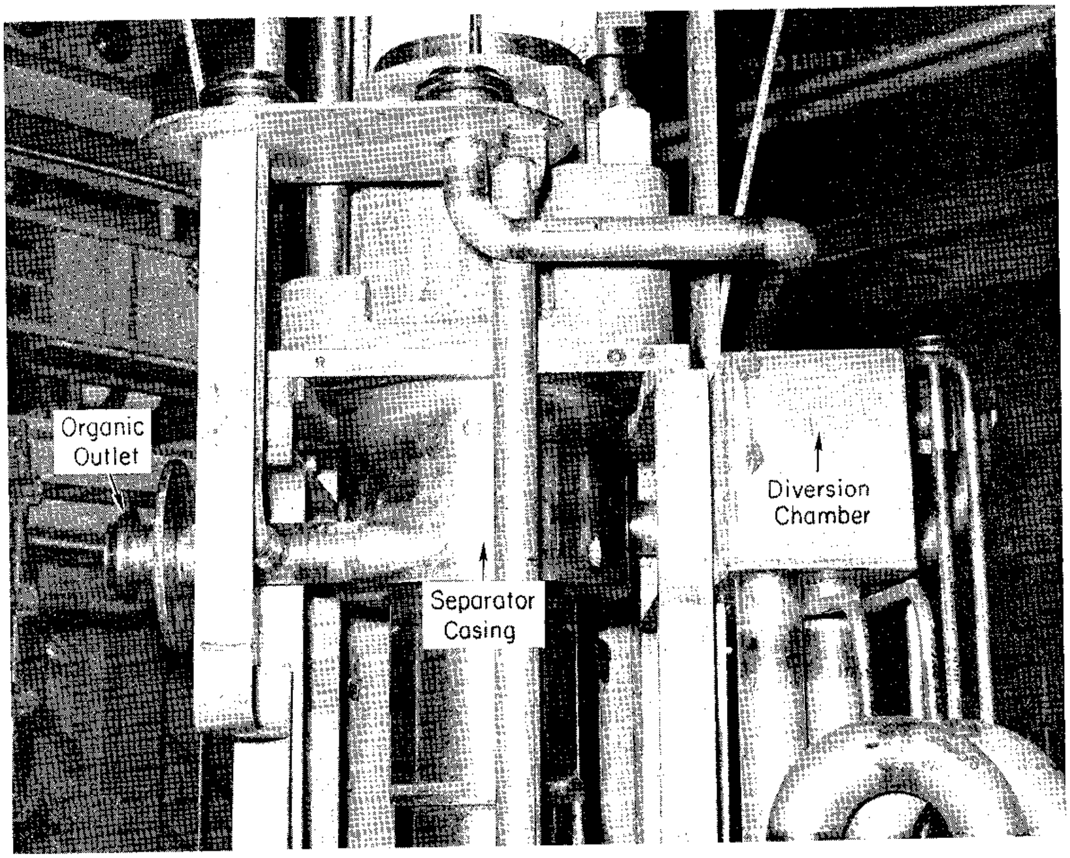
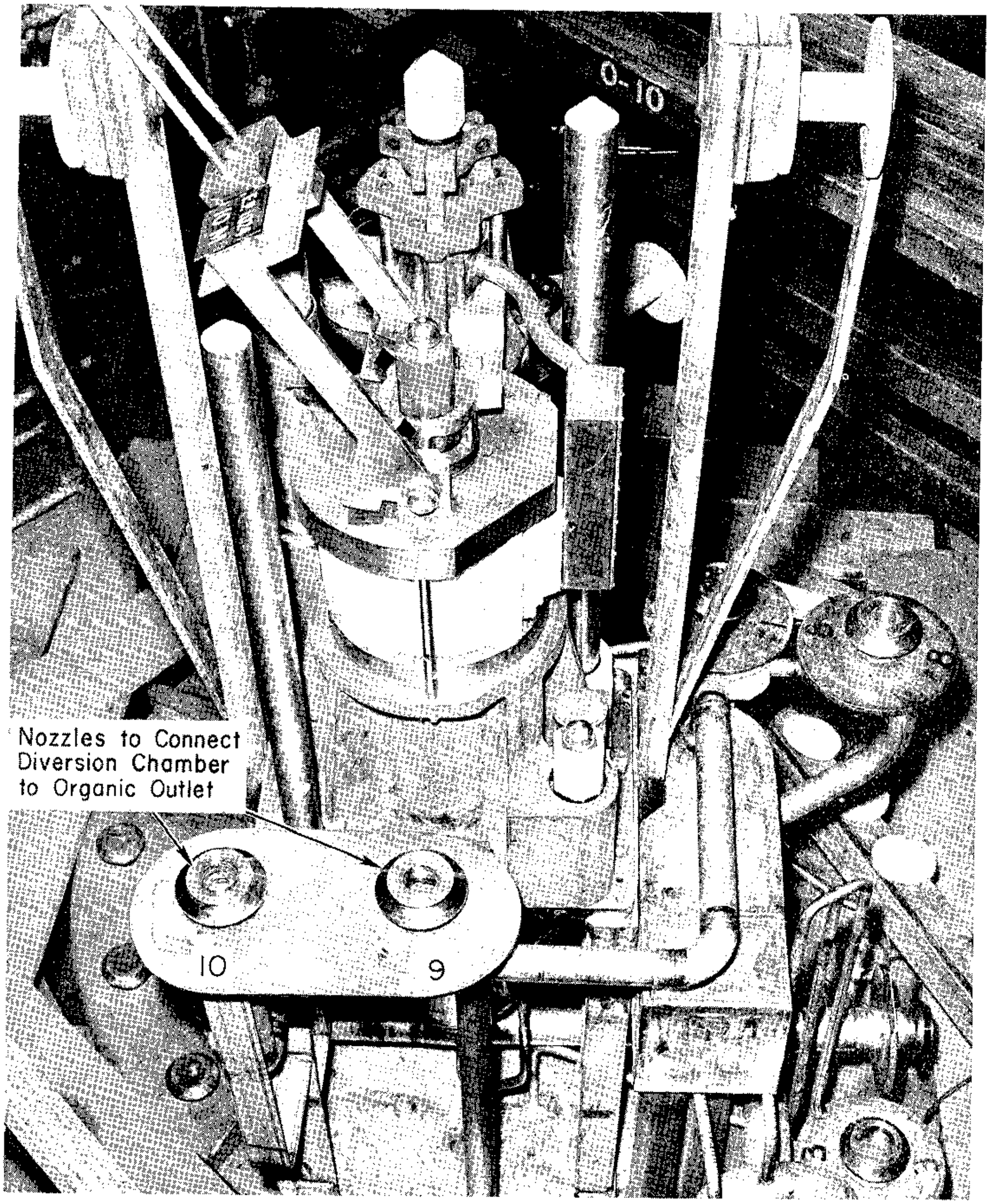


FIG. 2 SINGLE-STAGE CENTRIFUGAL SEPARATOR (Overall View)
Additional Views on Pages 8 and 9



(Detailed View)



(Detailed View)

OPERATION OF SEPARATOR

The aqueous and/or organic solutions enter at the bottom of the separator (Fig. 3), where they are mixed by a paddle that pumps the dispersion into the centrifuge bowl for continuous separation.

The organic light phase moves inward toward the axis of the centrifuge, flows over a circular weir at the top, and is thrown outward through radial ducts into the organic collection chamber.

The aqueous heavy phase moves outward toward the wall of the centrifuge, flows under a baffle at the top, and is then forced inward and over a circular weir. The aqueous phase then flows under a second baffle and over a second weir before it is thrown out into the aqueous collection chamber. The second baffle and weir form a seal for air pressure applied to the surface of the liquid flowing over the first weir.

An increase in air pressure forces the mixed dispersion inward toward the organic-phase weir; a decrease in air pressure allows the dispersion to move outward toward the aqueous-phase exit.

At low flows, the band of dispersion is narrow and can be moved inward or outward without reaching an exit; as the flow is increased, the band widens. The hydraulic capacity of the separator is the limiting rate of processing at which the band of dispersion fills the bowl and any change in air pressure causes entrainment in one effluent phase or the other. The maximum hydraulic capacity of the separator is governed by the aqueous-to-organic ratio and by the maximum allowable endstream entrainment (Fig. 4).

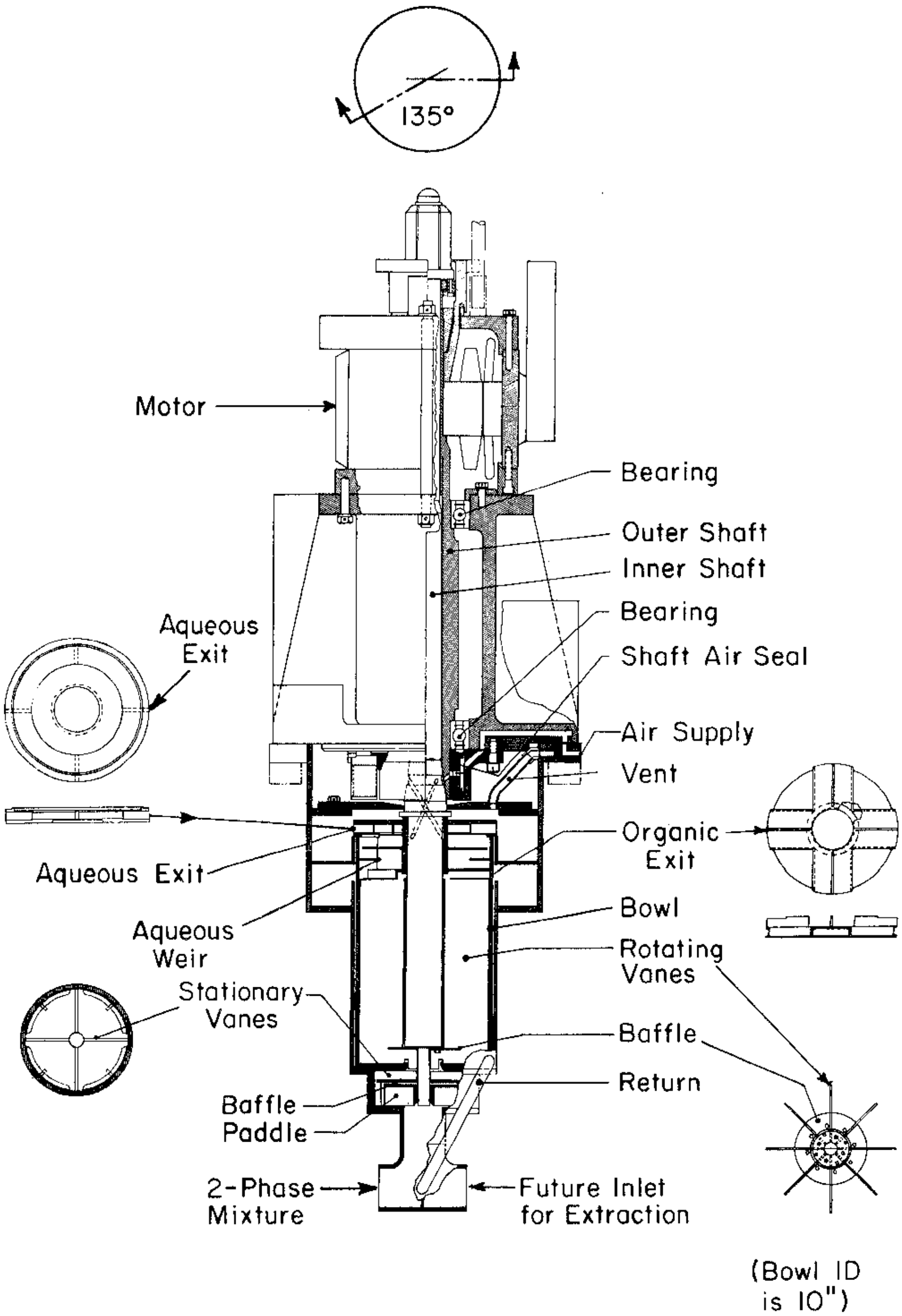
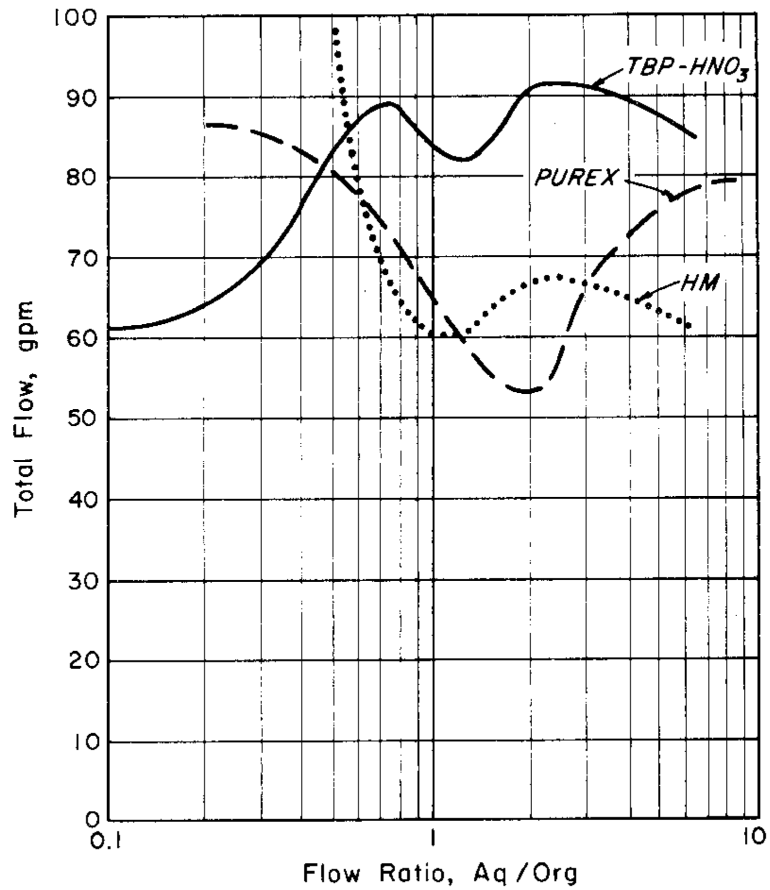


FIG. 3 CROSS SECTION OF CENTRIFUGAL SEPARATOR



Solutions: <u>TBP-HNO$_3$</u>

Aq - 3M HNO$_3$, 1.112 sp gr
Org - 2M HNO$_3$, 1.051 sp gr, 100% TBP

<u>HM</u>

Aq - 0.5M HNO$_3$, 1.20 sp gr 1.2M Al(NO$_3$)$_3$
Org - 0.780 sp gr, 2.5% TBP in "Ultrasene"*

*Refined kerosene. Trademark of Atlantic Refining Co., Phila., Pa.

<u>Purex</u>

Aq - 0.5M HNO$_3$, 1.021 sp gr
Org - 0.846 sp gr, 30.5% TBP in "Ultrasene"

Basis: 0.5% entrainment in each endstream

FIG. 4 HYDRAULIC CAPACITY OF CENTRIFUGAL MIXER-SETTLER
(from Reference 2)

EXPERIMENTAL PROCEDURE AND RESULTS

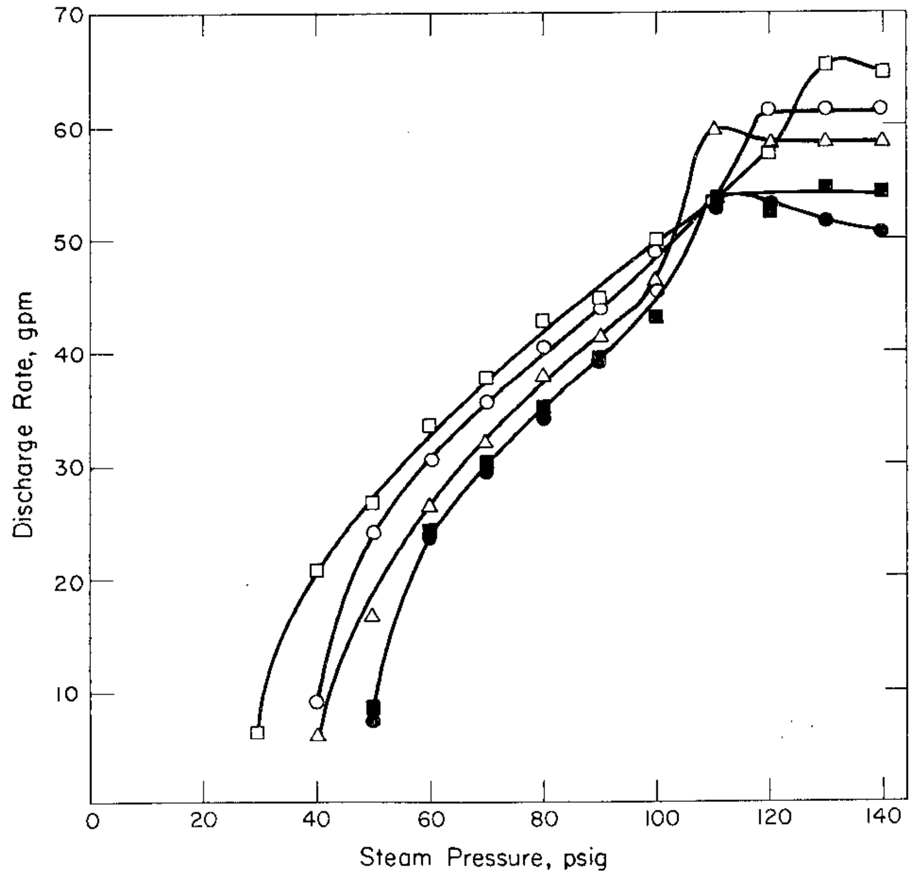
Because the centrifugal separator was to be used first in the ^{244}Cm separations process, the unit was tested at the semiworks with simulated ^{244}Cm process solutions using the feed jet and the aqueous outlet jumpers from the plant. The organic outlet jumper was simulated with provisions made to deliver the solvent to the collection tank through either a vented dip leg or an open line.

Feed Jet

Previous data indicated that the OF-5 jet would be suitable for feeding the centrifugal separator. This jet was therefore tested in a facility that simulated the plant installation, with lift up to 13 ft and physical discharge head of ~2 ft. Water, 30 vol % TBP in "Ultrasene", nitric acid solution, and concentrated uranyl nitrate solution were jetted at physical lifts of 5, 7, 9, 11, and 12 ft. Controlled jetting of each solution was demonstrated throughout the range of ~15 to 50 gpm, at a suction lift as high as 13 ft; the data are shown in Fig. 5 through 8.

Air Entrainment

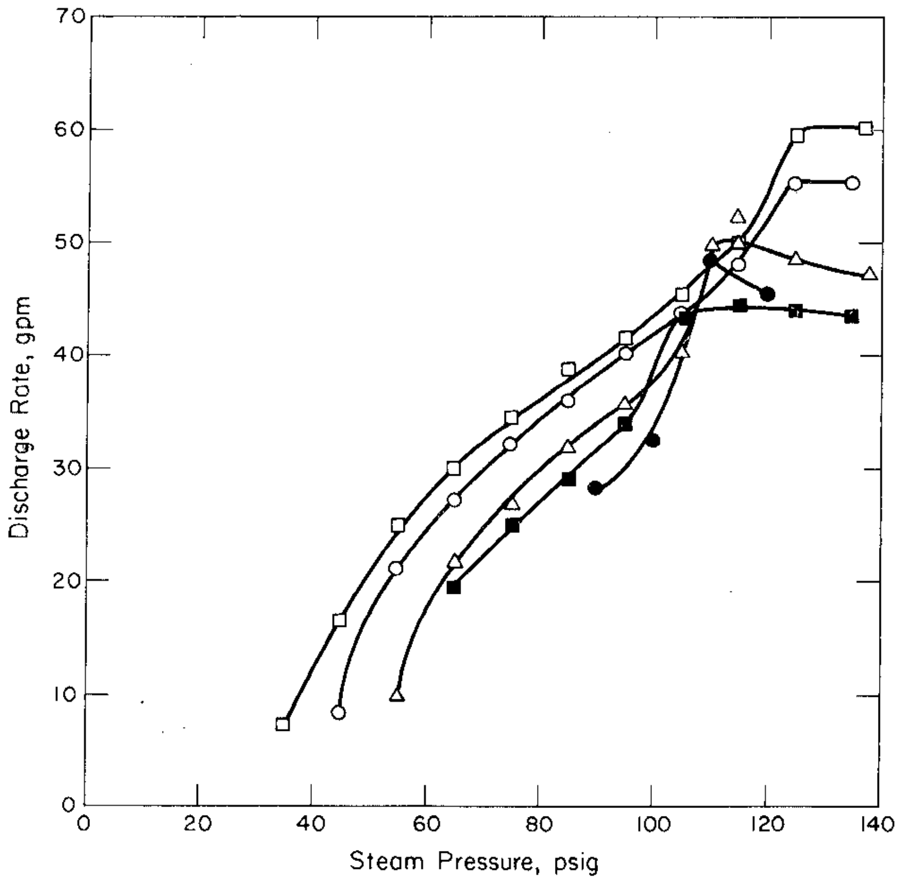
During initial tests with water, the hydraulic capacity of the unit was limited to ~22 gpm by entrained air that caused excessive pressure drop in the aqueous exit piping of the diversion system (Fig. 9). Entrainment of air in the 18-stage bank of centrifugal mixer-settlers causes no problems because there are no restrictions in the discharge line similar to those in the diversion system. Venting the diversion chamber and increasing its size from 0.08 to 1.5 ft^3 de-entrained the air and eliminated the problem. A baffle in the modified diversion chamber reduces liquid entrainment in the vented air (Fig. 10). For the ^{244}Cm process, the chamber is vented to the organic outlet pipe, but provisions permit venting to other vessels if necessary.



Solution: Water @ 35°C
Discharge Head: ~2 ft solution

Symbol	Physical Lift, ft	Cutoff Press., psi	Maximum Temperature Rise, °C
□	5 1/6	25	20 @ 30 psi; 12 @ 40 to 140 psi
○	7 1/6	34	20 @ 40 psi; 10 @ 50 to 140 psi
△	9 1/6	40	30 @ 40 psi; 10 @ 50 to 140 psi
●	11	45	24 @ 50 psi; 11 @ 60 to 140 psi
■	12	50	28 @ 50 psi: 12 @ 60 to 140 psi

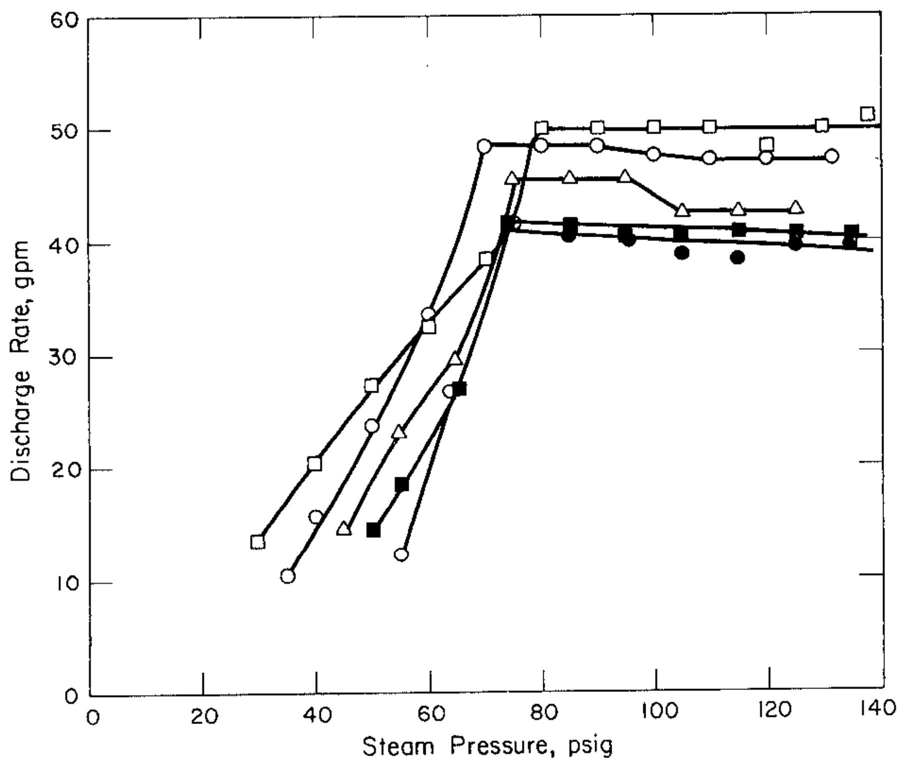
FIG. 5 PERFORMANCE OF OF-5 JET WITH WATER



Solution: 1.24 sp gr HNO_3 @ 35°C
Discharge Head: ~2 ft solution

Symbol	Physical Lift, ft	Cutoff Press., psi	Maximum Temperature Rise, °C
□	5	30	26 @ 35 psi; 18 @ 45 to 137 psi
○	7	35	29 @ 45 psi; 13 @ 55 to 135 psi
△	9	48	30 @ 55 psi; 14 @ 65 to 138 psi
■	11 1/6	60	16.5 @ 65 to 145 psi
●	12 1/6	80	14 @ 90 to 120 psi

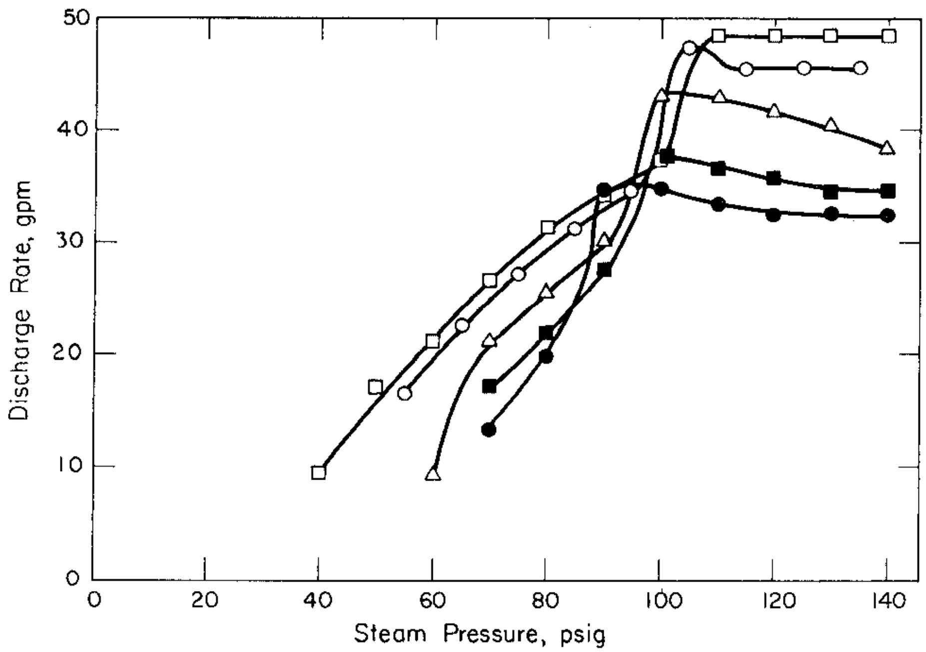
FIG. 6 PERFORMANCE OF OF-5 JET WITH HNO_3



Solution: 30 Volume % TBP in "Ultrasene"
Discharge Head: ~2 ft solution

Symbol	Physical Lift, ft	Cutoff Press., psi	Maximum Temperature Rise, °C
□	5 1/6	25	25 @ 30 to 138 psi
○	7 1/6	32	34 @ 35 psi; 26 @ 40 to 132 psi
△	9 1/6	42	31 @ 45 psi; 27 @ 55 to 125 psi
■	11 1/6	48	35 @ 50 psi; 29 @ 55 to 135 psi
●	12	53	28 @ 55 psi; 23 @ 65 to 137 psi

FIG. 7 PERFORMANCE OF OF-5 JET WITH ORGANIC
(Jet will pick up with 12′ 11″ lift)



Solution: 1.51 sp gr (330 g/l uranium) uranyl nitrate @ 35°C
Discharge Head: ~2 ft solution

Symbol	Physical Lift, ft	Cutoff Press., psi	Maximum Temperature Rise, °C
□	5 1/6	37	24 @ 45 psi; 14 @ 50 to 140 psi
○	7	48	17 @ 55 to 135 psi
△	9	55	22 @ 60 psi; 16 @ 70 to 140 psi
■	11	62	19 @ 70 to 140 psi
●	12	65	24 @ 70 psi; 19 @ 80 to 140 psi

FIG. 8 PERFORMANCE OF OF-5 JET WITH URANYL NITRATE

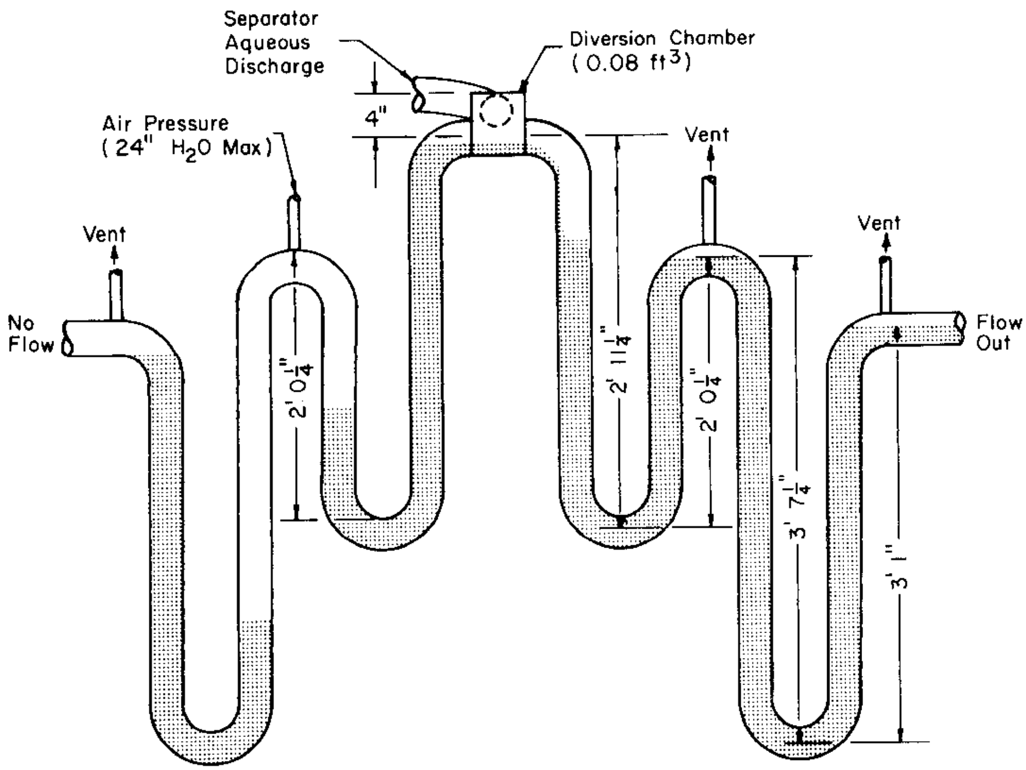


FIG. 9 CENTRIFUGAL SEPARATOR DIVERSION SYSTEM

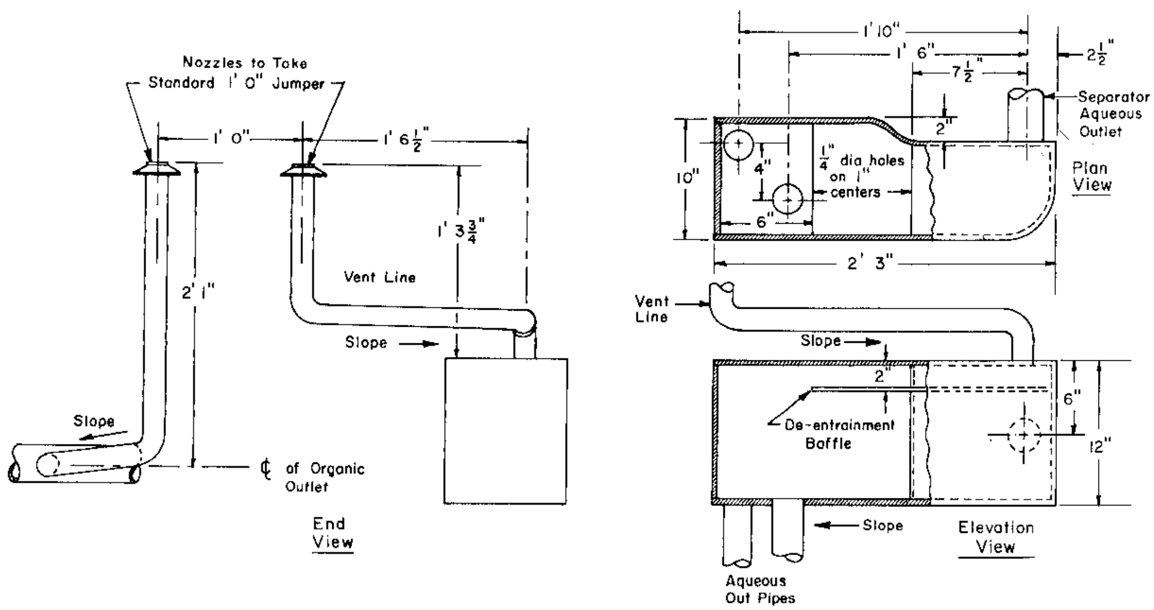


FIG. 10 MODIFIED DIVERSION CHAMBER

Capacity Tests

Capacity tests were made with simulated ^{244}Cm process conditions for batch extraction, stripping, and solvent washing. In each test, the two liquid phases were mixed for 30 minutes in the feed tank with agitation similar to the maximum in the plant. The agitation was then stopped, and the mixed-phase feed was jetted immediately to the centrifugal separator. The following mixtures were fed.

	Aqueous Phase	Organic Phase	Phase Volume Ratio, Aq/Org
Extraction	1.36 sp gr $Al(NO_3)_3$ 0.5M HNO_3	50% TBP in "Ultrasene"	1
Stripping	0.2M HNO_3	"	1/2
Washing	2.5% Na_2CO_3 0.8% NaOH	"	1/4

The mixed solutions in the feed tank were maintained between the expected maximum and minimum plant temperatures (45 and 25°C). With 120-psig steam to the feed jet (maximum rate), the processing flows were 40 to 50 gpm for the four solutions; the feed temperature had only a small effect on feed flow.

Feed tests with a pump showed that the capacity of the centrifuge exit line to the waste tank is about 60 gpm (higher flows fill a gravity decanter that discharges into the same piping). The capacity of the exit line to the product tank is more than 65 gpm.

Delivery of the separated organic liquid to the collection tank was equally satisfactory through either the open line or the sealed dip leg with a 1/4-inch-diameter vent hole above the solution level.

The extraction, stripping, and solvent washing solutions were centrifuged as thoroughly mixed (>4.2 hp/1000 gal) emulsions to determine the liquid entrainment in each endstream at different weir air pressures. To maintain <0.5% liquid entrainment in both endstreams, as shown in Fig. 11, the feed

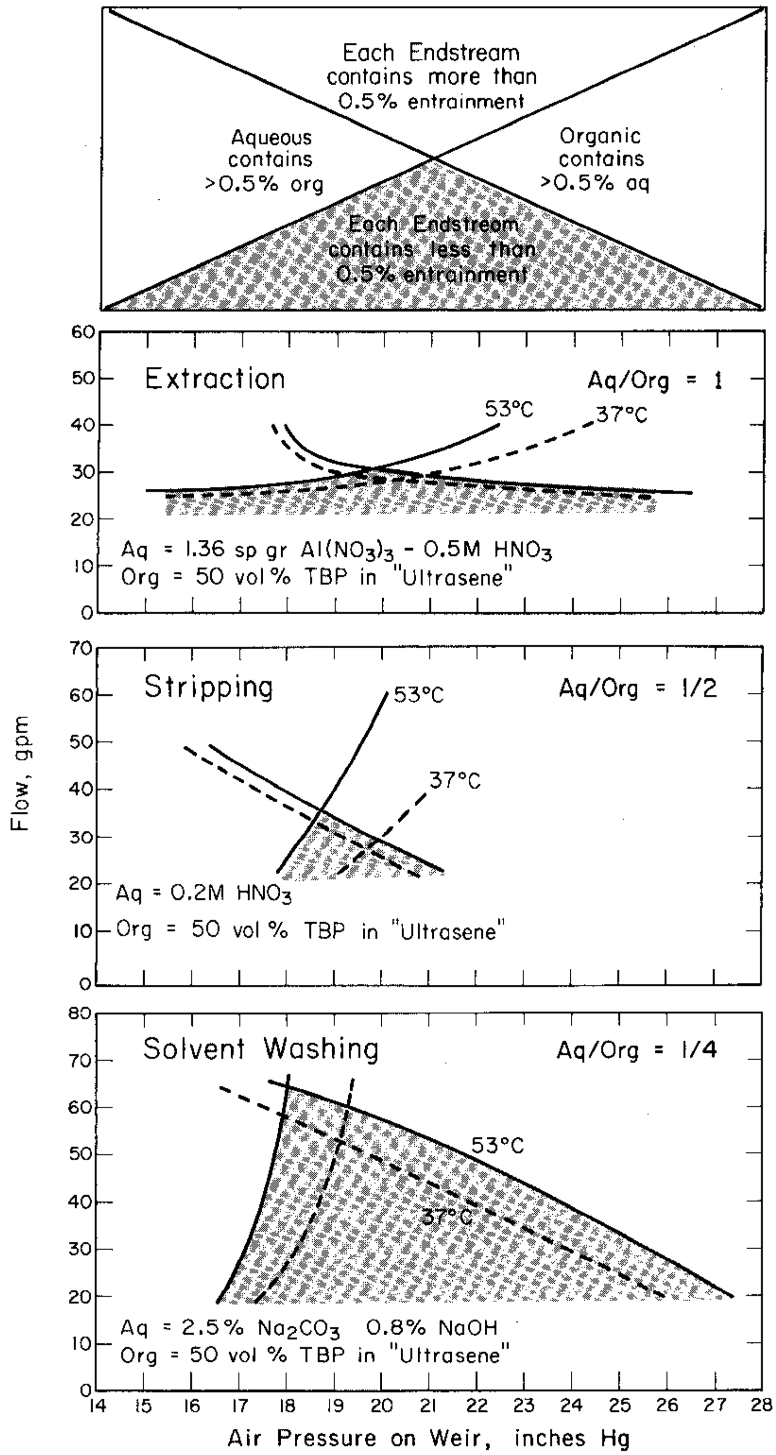


FIG. 11 ENDSTREAM ENTRAINMENT WHEN PROCESSING THOROUGHLY MIXED EMULSIONS

rate should be <25 gpm at 25°C, and <30 gpm at 45°C; the data are plotted for 37 and 53°C which include heating by the jet. A clear aqueous stream is obtained at higher rates of processing by increasing the air pressure on the centrifuge weir and so forcing the entrainment to the organic exit stream, from which it returns to the feed tank. In this way, the overall rates of processing can be somewhat higher with both the stripping and washing mixtures. Graphs showing entrainment as a function of air pressure for the solutions tested are shown in Fig. 12 through 14.

Power Requirements

The power requirements for the separator were affected by both throughput and type of solution, as shown in Fig. 15. The sharp increase in power with increase in flow observed with the 50% TBP was caused by liquid backup into the annulus between the bowl and casing; the backup was caused in turn by the resistance to flow of the two-phase mixture (air and organic) through the long organic exit line.

The unit is driven by a nominal 5 hp motor with Class F insulation instead of the normal Class A. With the better insulation, which permits operating at 155°C instead of 105°C, the motor should operate at loads of 7 to 8 hp and still have a standard life of 35,000 hours.

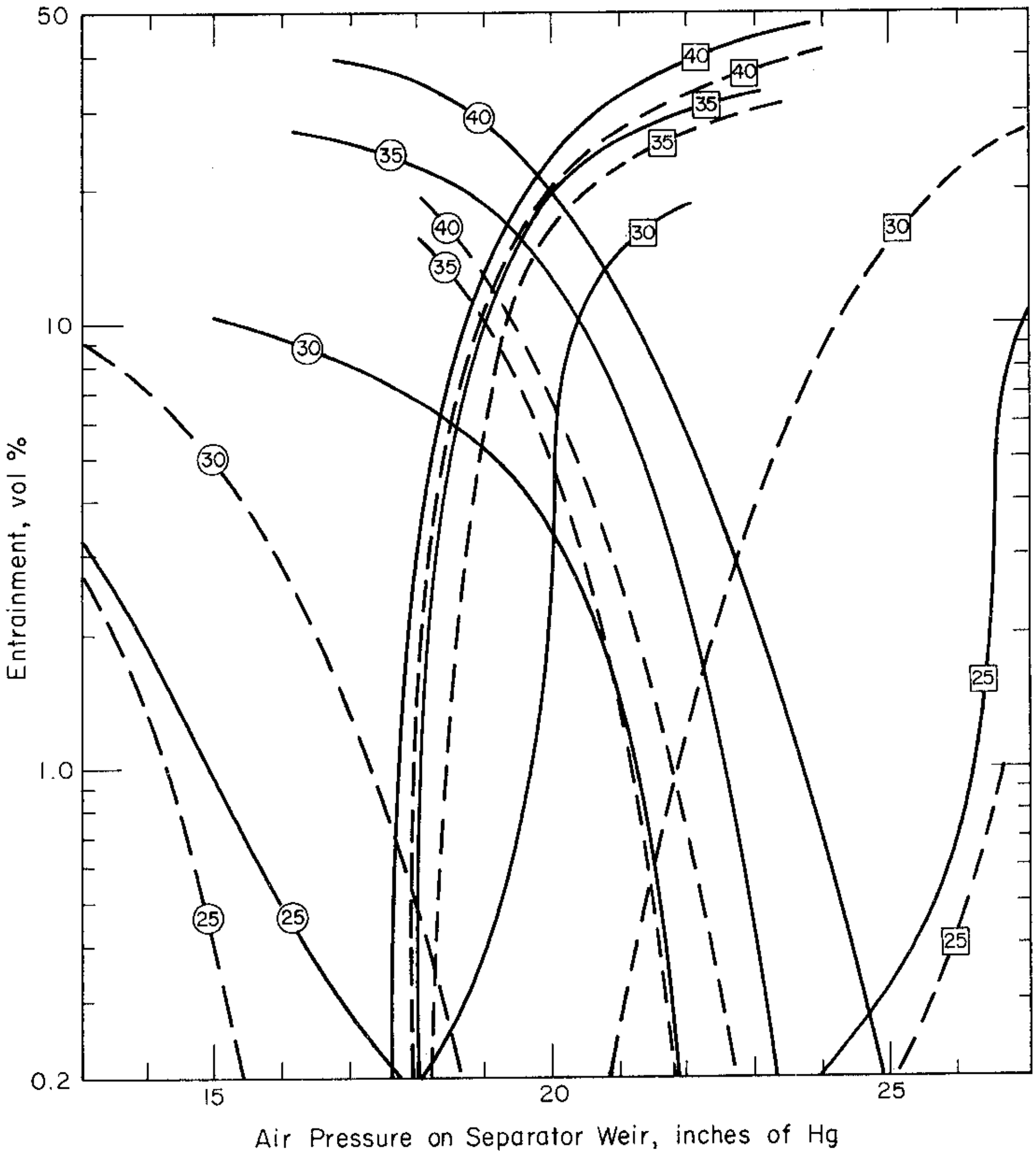
Motor current was also measured at 495 volts during the tests. The currents, corrected to the 440 volts in the plant, are shown in Fig. 16.

Air and Vent Requirements

The separator has a "Koppers"* floating bushing air seal to transfer air from the stationary encasement to the rotating shaft without significant pressure drop. This seal operates with a continuous air leakage. The separator had an air leakage rate of 1.0 cfm at 20 inches of mercury, which is acceptable.

The pumping action of the separator maintains a vacuum of 1 to 6 inches of water on its vent during operation; the vacuum varies, generally increasing with increase of feed rate to the unit. The normal vacuum in the vessel vent system of the plant is ~7/8-inch of water. Under normal operating conditions the separator pulls air from the building vent system, but this has presented no problems.

*Trademark of Koppers Company, Inc., Pittsburgh, Pa.

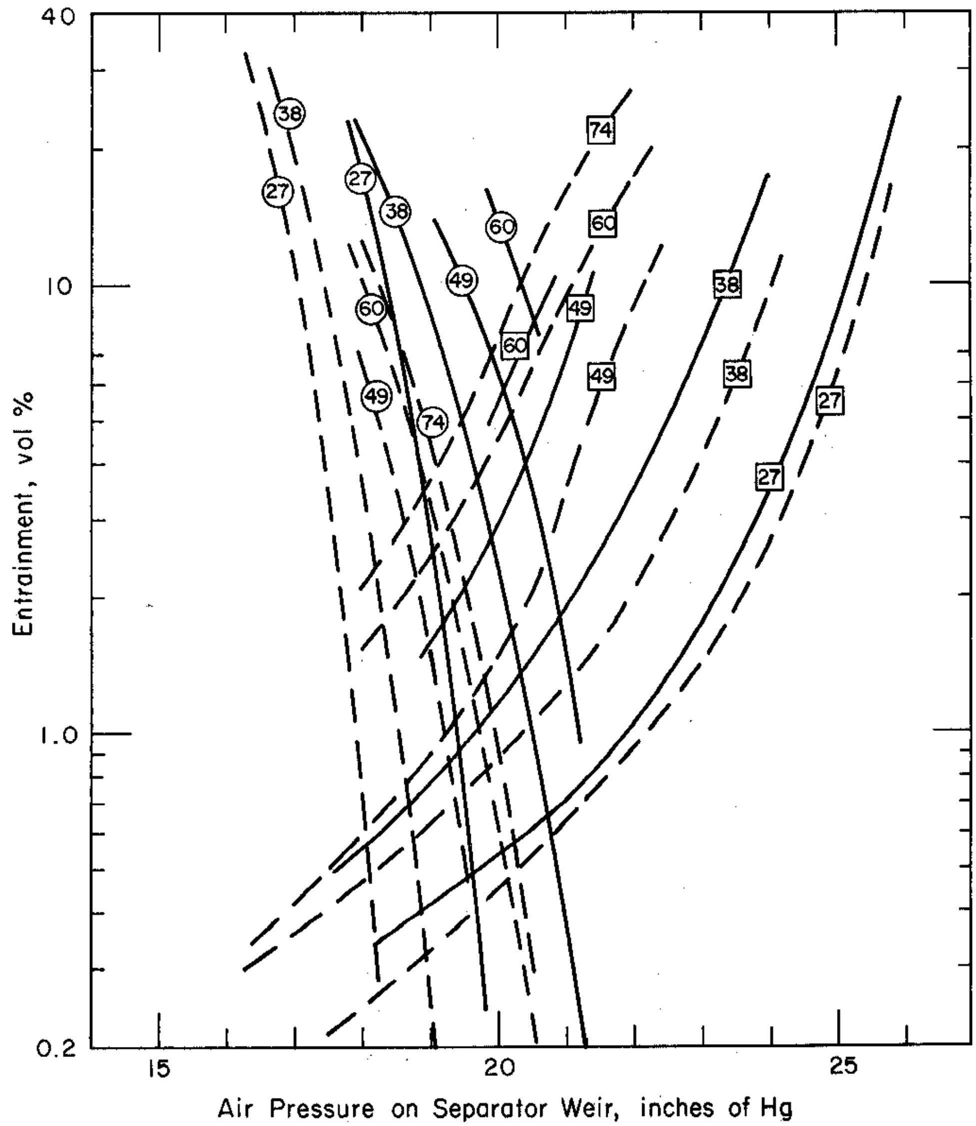


Feed: Equal volumes of 1.36 sp gr $Al(NO_3)_3$-0.5M HNO_3 and 50 vol % TBP in "Ultrasene", completely mixed.

Temperature: 37°C ————
53°C - - - - -

Flow, gpm: Shown within symbols □ aqueous entrained in organic
○ organic entrained in aqueous

FIG. 12 ENTRAINMENT AS A FUNCTION OF CENTRIFUGAL SEPARATOR WEIR PRESSURE WITH $Al(NO_3)_3$ -TBP MIXTURE

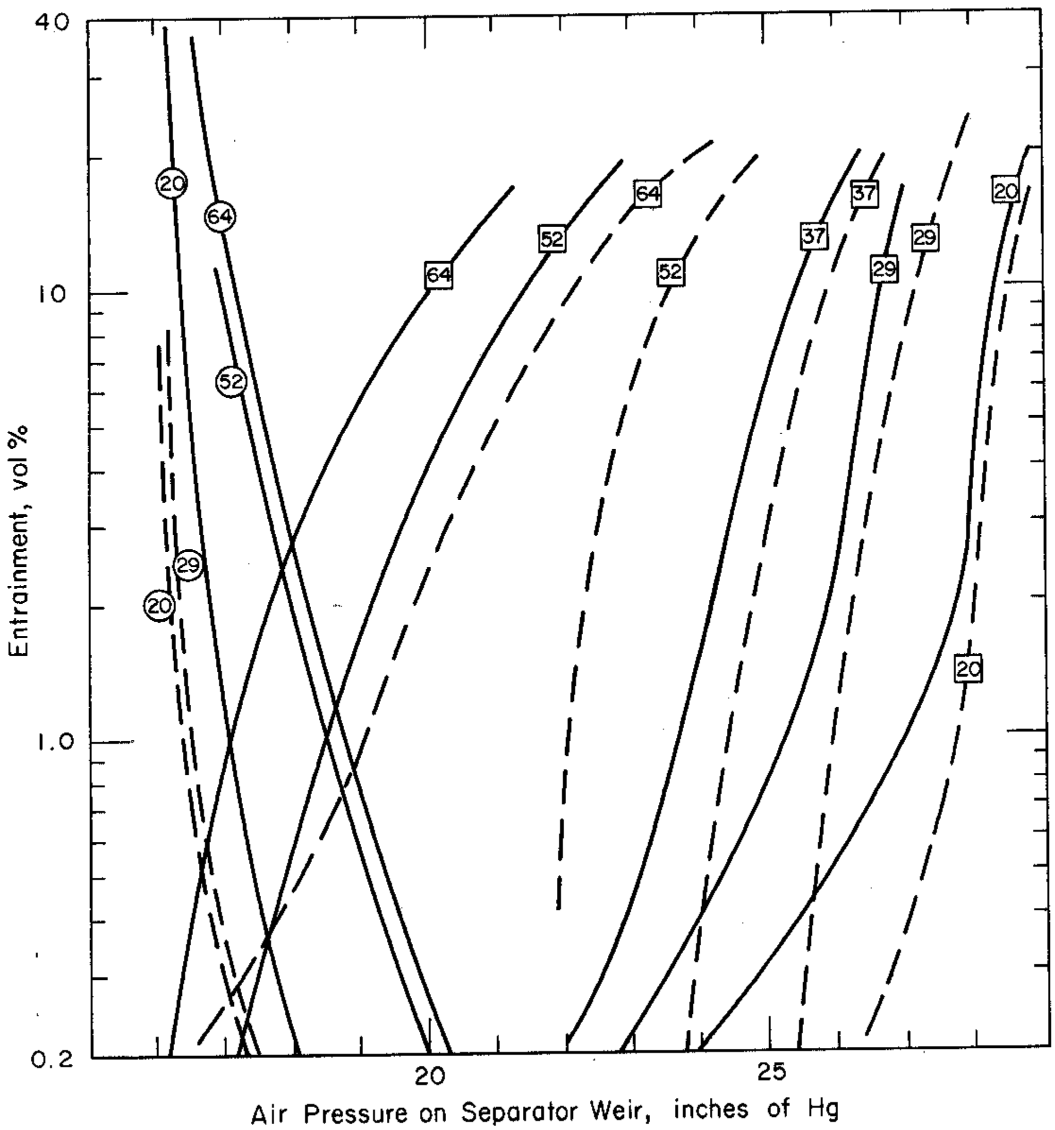


Solutions: 1/2 volume 0.2M HNO_3 and 1 volume 50 vol % TBP in "Ultrasene", completely mixed.

Temperature: 37°C ———— 53°C - - - - -

Flow, gpm: Shown within symbols □aqueous entrained in organic ○organic entrained in aqueous

FIG. 13 ENTRAINMENT AS A FUNCTION OF CENTRIFUGAL SEPARATOR WEIR PRESSURE WITH 0.2M HNO_3 - TBP MIXTURE



Solutions:	1/4 volume 2.5% Na_2CO_3-0.8% NaOH and 1 volume 50 vol % TBP in "Ultrasene", completely mixed.	
Temperature:	37°C ———	
	53°C - - - - -	
Flow, gpm:	Shown within symbols	□ aqueous entrained in organic
		○ organic entrained in aqueous

FIG. 14 ENTRAINMENT AS A FUNCTION OF CENTRIFUGAL SEPARATOR WEIR PRESSURE WITH (2.5% Na_2Co_3 - 0.8% NaOH) - TBP MIXTURE

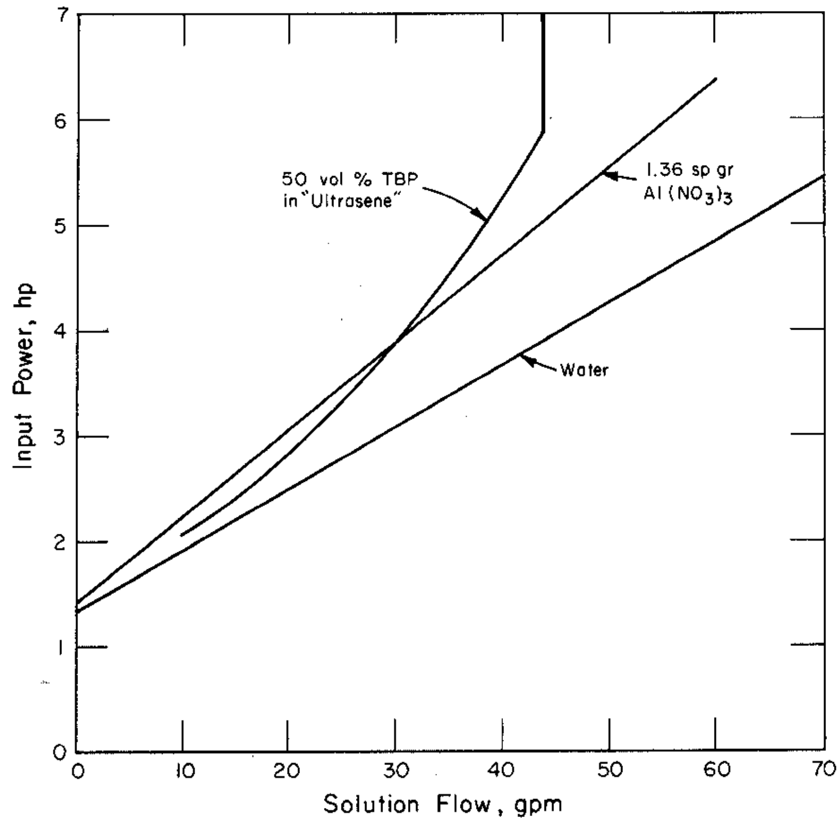
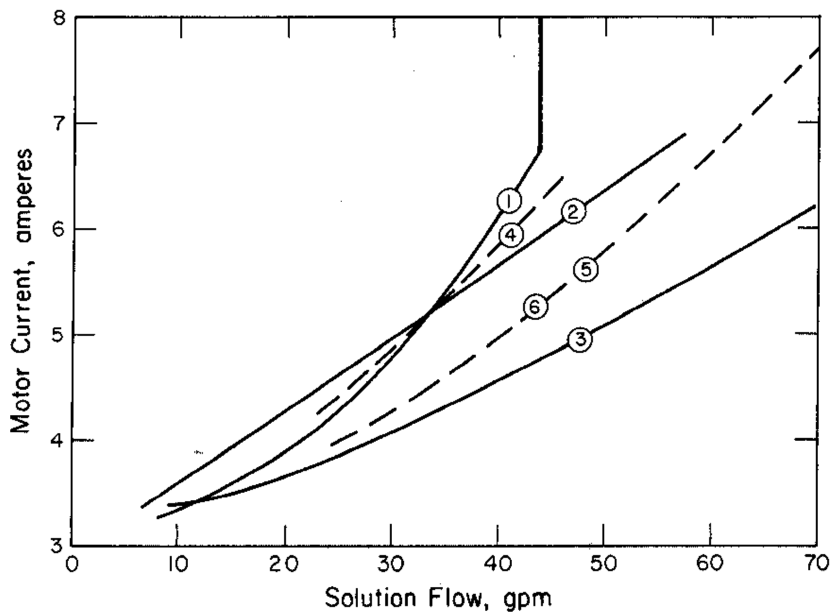


FIG. 15 POWER REQUIREMENTS AS A FUNCTION OF FLOW RATE FOR CENTRIFUGAL SEPARATOR (Motor Voltage: 440 volts)

Tests were made to determine whether the centrifugal separator would be pressurized either during normal air "blowout" of the feed jet steam line (normal canyon operation), or in the event that the feed jet should deliver live steam to the separator. A 13-scfm air flow (after the jetting step) did not pressurize the unit, as long as the organic outlet line was open. However, 170 scfm of steam (originally at 110 psig) did pressurize the unit. Neither test caused apparent damage to the unit.

PLANT OPERATION

The separator was installed in the H-Area rerun station and operated with synthetic solutions prior to processing plant material. Operation during the ^{244}Cm campaign was excellent; the material was processed in about half of the time allowed, the organic was not damaged sufficiently by radiation to require changing, the emulsion that formed between organic and caustic wash was separated, and product loss was negligible.



Curve	Solution	Temp, °C
1	50 vol % TBP in "Ultrasene"	36
2	1.36 sp gr $Al(NO_3)_3$	36
3	Water	36
4	Equal volumes 50% TBP in "Ultrasene" and 1.36 sp gr $Al(NO_3)_3$-0.5M HNO_3	36,53
5	1 volume 50% TBP in "Ultrasene" and 1/2 volume 0.2M HNO_3	36,53
6	1 volume 50% TBP in "Ultrasene" and 1/4 volume 25% Na_2CO_3-0.8% NaOH	36,53

FIG. 16 MOTOR CURRENT VERSUS FLOW RATE FOR CENTRIFUGAL SEPARATOR

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