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PROGRESS REPORT OF PILOT PLANT SECTION

RAW MATERIALS PROCESS TESTING

February 1955

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PROGRESS REPORT OF PILOT PLANT SECTION
RAW MATERIALS PROCESS TESTING

February, 1955

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AMINE EXTRACTION PROCESS TESTING

Effect of Leach Liquor Variables

The selectivity of Rohm and Haas amine 9D-178 (formerly EB-765-2) for uranium was studied in four runs using the four synthetic leach liquor compositions chosen previously for testing amines. Three of these compositions presented the most severe conditions for uranium extraction by Armeen 2-12 (ORNL-1914, page 25); the fourth was the standard synthetic Marysvale composition.

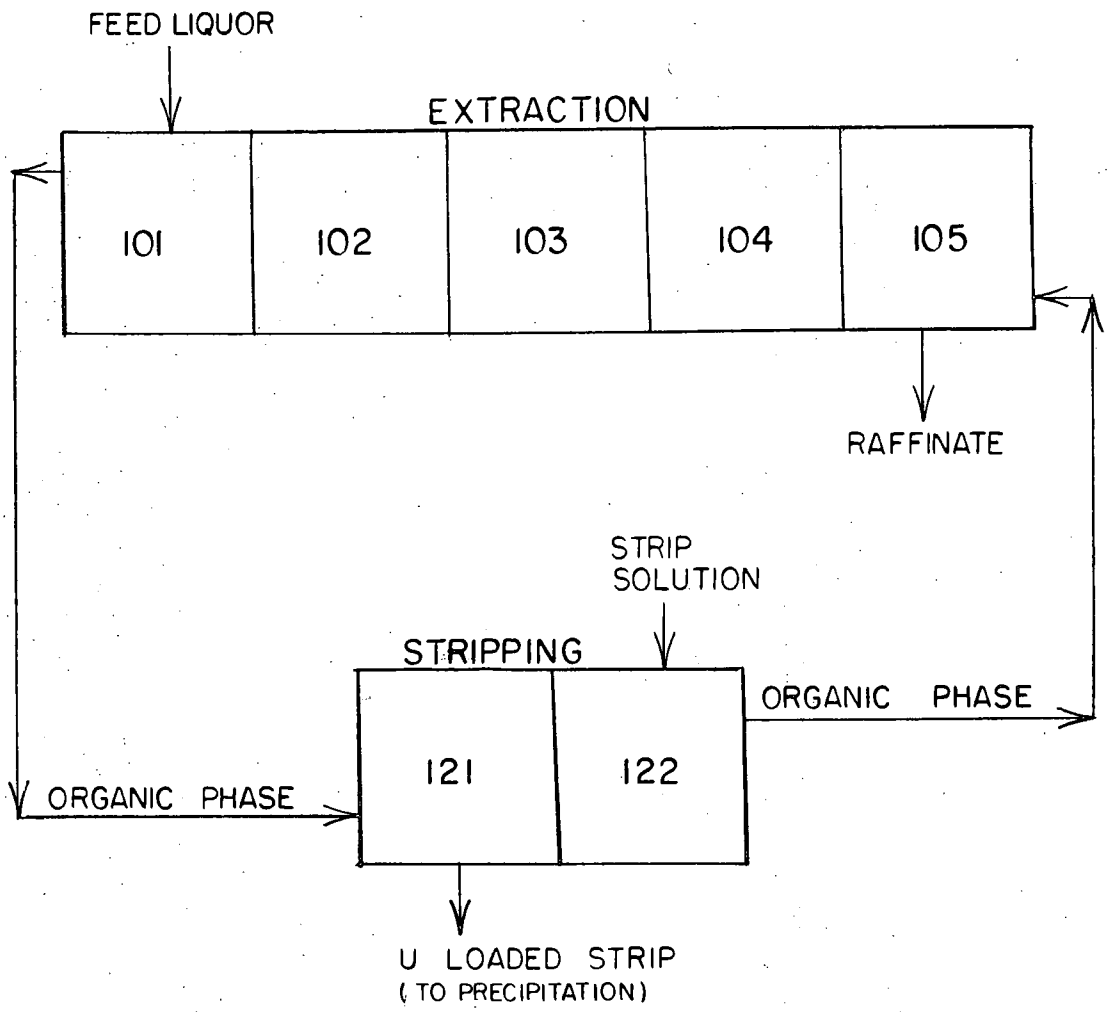
The runs were made in a mixer-settler test array that contained an extraction section and a stripping section both of which were operated with countercurrent phase flows as shown in Fig. 1. The equipment arrangement and the operating conditions shown in Table 1 are essentially the same as those employed in two tests made previously* (runs 14-15, ORNL-1914), except that the organic phase was pumped into mixer 105 by a micro-bellows pump (Corson-Cerveny) to provide a more accurate control of the flow rate.

The compositions of the feed streams are shown in Table 2. One of the head liquor compositions was typical for Marysvale type ore without vanadium or molybdenum (run 1), one contained a high concentration of iron without fluoride and phosphate (run 2), and two contained high concentrations of Fe^{+3}, V^{+5}, Mo, Ti, PO_4, F, and SO_4 differing only in that the pH was 1.0 in one (run 3) and 0.5 in the other (run 4). The organic phase was a 0.1 molar solution of 9D-178 in kerosene. The stripping agent was about 10 wt % solution of sodium carbonate in water. At the end of a run each mixer-settler unit was sampled to obtain stagewise concentration gradients. The operating data are reported in Table 3. An analysis of the uranium product and uranium material balance are shown for each run in Tables 4 and 5, respectively.

Extraction

The data in Table 3 show that uranium extraction was essentially complete in all runs. With head liquor compositions

*In runs 14 and 15 the solvent contained 5 vol % capryl alcohol to improve phase separation; subsequently, it was shown that, with 9D-178 in kerosene, phase separation was not appreciably affected by the addition of capryl alcohol; therefore, its use as a phase conditioner is unnecessary.



NOTE: ALL NUMBERS REFER TO MIXER-SETTLER UNITS.

FIG. 1 - FLOW DIAGRAM FOR RUNS 1 THROUGH 6.

TABLE 1. OPERATING CONDITIONS

Run number	1	2	3	4
Operating time, hours	4	4	4	4
Flow rates, ml/min				
Head liquor	50.2	49.5	47.6	48.4
Organic phase	20.1	20.1	20.0	20.1
Strip solution*	2.85	2.76	2.81	2.84
Extraction section				
Number of units	5	5	5	5
Size**	A	A	A	A
Agitator speed, unit 101, rpm	450	450	700	700
Agitator speed, units 102-105, rpm	450	450	450	450
Stripping section				
Number of units	2	2	2	2
Size**, unit 121	B	B	B	B
Size**, unit 122	A	A	A	A
Agitator speed, unit 121, rpm	1050	1050	1050	1050
Agitator speed, unit 122, rpm	450	450	350	350

*50% in excess of the theoretical Na_2CO_3 requirements which are: 3 moles per mole of uranium and 0.5 mole per mole of amine.

**Fig. 2, ORNL-1970.

TABLE 2. COMPOSITION OF THE FEED STREAMS

(All concentrations in grams per liter unless otherwise noted)

	Concentration			
	Run 1	Run 2	Run 3	Run 4
Head Liquor				
pH	1.0	1.0	1.0	0.5
Uranium	1.2	1.2	1.1	1.2
Iron (Fe^3)	5.4	16.	16.	16.
Vanadium (total)	0	0	3.0	3.0
Vanadium (V^5)	0	0	2.5	2.5
Titanium	0	0	0.5	0.5
Molybdenum	0	0	0.8	0.9
Aluminum	3.1	3.1	3.1	3.1
Sulfate	63.0	70.0	120.	120.
Phosphate	2.1	0	5.6	5.4
Fluoride	1.0	0	4.6	4.6
Organic Phase				
Amine 9D-178 molarity	0.1	0.1	0.1	0.1
Uranium	0.001	0.001	0.001	0.001
Iron	0.004	0.004	0.004	0.008
Strip Solution				
Na_2CO_3	97	97	97	97

TABLE 3. OPERATING DATA, EXTRACTION SECTION

Run No.	Ratio: Head Liquor/ Organic*	Mixer-Settler Number	Aqueous, g/l				Organic, g/l					Distribution org/aq
			pH	U	V	Mo	U	Fe	V	Mo	Ti	U
1	2.50	101	1.1	0.040	-	-	2.8	0.1	-	-	-	70
		102	1.1	0.001	-	-	0.13	0.5	-	-	-	130
		103	1.1	<0.001	-	-	0.005	0.5	-	-	-	-
		104	1.1	<0.001	-	-	0.003	0.5	-	-	-	-
		105	1.1	<0.001	-	-	0.003	0.6	-	-	-	-
2	2.46	101	1.3	0.18	-	-	3.0	0.4	-	-	-	17
		102	1.2	0.010	-	-	0.49	1.	-	-	-	49
		103	1.2	0.002	-	-	0.028	1.	-	-	-	14
		104	1.3	<0.001	-	-	0.007	1.	-	-	-	-
		105	1.3	<0.001	-	-	0.006	1.	-	-	-	-
3	2.38	101	0.9	0.90	2.8	0.18	2.5	0.03	0.1	-	0.02	2.8
		102	0.9	0.097	2.9	0.09	2.2	0.1	0.2	0.4	0.02	23
		103	0.9	0.007	2.9	0.05	0.37	0.4	0.5	0.3	0.02	53
		104	0.9	<0.001	2.9	0.04	0.026	0.6	0.5	0.09	0.02	-
		105	0.9	<0.001	2.7	0.03	0.003	0.6	0.4	0.04	0.02	-
4	2.41	101	0.5	0.79	3.0	0.29	2.7	0.03	0.2	1.9	0.04	3.4
		102	0.5	0.12	3.0	0.13	2.0	0.08	0.1	0.5	0.06	17
		103	0.5	0.017	3.0	0.10	0.41	0.2	0.2	0.2	0.06	24
		104	0.5	0.004	2.9	0.07	0.067	0.2	0.2	0.1	0.05	17
		105	0.4	<0.001	2.8	0.05	0.011	0.2	0.2	0.05	0.05	-

*From measured flow rates.

TABLE 3 (continued)

OPERATING DATA, STRIPPING SECTION

Run No.	Ratio: * Organic/ Strip	Mixer-Settler Number	Aqueous, g/l**						Organic, g/l				
			U	Fe	V	Mo	Ti	pH	U	Fe	V	Mo	Ti
1	7.1	121	22.	0.12	-	-	-	-	0.17	0.01	-	-	-
		122	0.96	0.06	-	-	-	-	0.001	0.004	-	-	-
2	7.3	121	20.	0.21	-	-	-	-	0.37	0.02	-	-	-
		122	1.6	0.06	-	-	-	-	0.001	0.001	-	-	-
3	7.1	121	20.	0.31	2.4	11.0	0.14	7.6	0.17	0.004	<0.03	0.21	<0.01
		122	0.86	0.03	0.53	0.76	0.02	8.9	<0.001	0.008	<0.03	0.04	<0.01
4	7.1	121	19.	0.14	2.2	11.0	-	7.0	0.17	0.005	<0.03	0.26	<0.01
		122	0.99	0.11	0.27	0.81	0.02	8.8	0.001	0.005	<0.03	0.03	<0.01

*From measured flow rates.

**Filtered prior to sampling.

TABLE 4. COMPOSITION OF URANIUM PRODUCTS*

(Basis: Dried at 130°C for 24 hours)

Run Number:	Percent by weight 1	2	3	4
Component				
Uranium as U	73.7	72.1	57.7	59.7
as U_3O_8	86.9	85.0	68.0	70.4
Iron (Fe)	0.18	0.17	0.58	0.17
Carbon (C)	0.14	0.18	0.10	0.12
Molybdenum (Mo)	-	-	0.35	1.1
Titanium (Ti)	-	-	0.42	< 0.1
Vanadium (V)	-	-	5.2	3.2
Phosphate (PO_4)	-	-	1.1	-
Loss on ignition (600°C)			6.3	
U on ignited product			61.6	

*Precipitated from the filtered loaded strip solutions that were collected over the final two hours for each run.

TABLE 5. URANIUM MATERIAL BALANCES

(Basis: 100 parts U in head liquor)

Run Number:	1	2	3	4
In				
Feed	100	100	100	100
Organic	< 0.1	< 0.1	< 0.1	< 0.1
Total in	100	100	100	100
Out, Extraction				
Raffinate	< 0.1	< 0.1	< 0.1	< 0.1
Organic extract	93	102	95	93
Out, Over-all				
Raffinate	< 0.1	< 0.1	< 0.1	< 0.1
Stripped organic	< 0.1	< 0.1	< 0.1	< 0.1
Loaded strip	104	93	107	93

varying widely, the number of mixer-settler units needed to reduce the uranium content of the raffinate to 0.003 g/l ranged from 2 in run 1 to 5 in run 4.

Iron extraction was appreciable only in run 2 which was made with a head liquor containing high concentration of iron (15 g/l Fe^3) without fluoride or phosphate*. Three mixer-settler units were needed for complete extraction of uranium in run 2 as compared to two units in run 1.

The effect on the uranium extraction coefficients of several anions and metals that are the most likely components of sulfuric acid leach liquor obtained from processing Colorado Plateau ores are shown by comparing the data for runs 1, 3, and 4. The concentrations of the components common to all three head liquors were much higher in runs 3 and 4 than they were in run 1. Also, head liquors in runs 3 and 4 contained high concentrations of vanadium, molybdenum, and titanium, differing only in that the pH was 1.0 in run 3 and 0.5 in run 4. The results show that uranium extraction coefficients were markedly lowered in runs 3 and 4 and that in addition to uranium, only molybdenum was extracted in appreciable amounts. Molybdenum extraction was about 95% complete in both runs. The over-all effect was an increase in the number of mixer-settler units needed for satisfactory uranium recovery from 2 in run 1 to 4 in run 3 (pH = 1.0) and 5 in run 4 (pH = 0.5).

Uranium Stripping and Product Purity

The data in Table 3 shows that essentially complete stripping of uranium, iron, vanadium, molybdenum, and titanium from the organic extract was obtained in the strip operation. The analysis of uranium products precipitated from the loaded strip solutions (Table 4) shows that the products obtained from runs 1 and 2 assayed about 85% U_3O_8 while those from runs 3 and 4 were about 70%.

Uranium Material Balance

A uranium material balance is shown in Table 5 for each run. For convenience, the calculations were made on the basis of 100 parts (grams, pounds, etc.) of uranium entering in the head liquors. In all runs the balance between uranium input and output, over-all, was within ±10%.

---

*Laboratory studies have shown that either F or PO_4 will depress the extraction of Fe^3 by amines from acidic sulfate solution.

Effect of Phase Ratio

Laboratory studies have shown that an increase in uranium concentration in the organic extract brought about by an increase in the aqueous to organic phase ratio, depresses the extraction of contaminating ions such as iron and vanadium. To check this effect in a continuous, countercurrent system two runs were made in which the aqueous to organic phase flow ratios were 3 to 1 in one run and 3.5 to 1 in the other.

The runs were carried out in a mixer-settler test array with the same equipment arrangement and under essentially the same operating conditions as those employed in runs 1 through 4.

The head liquor for run 5 was the Vitro leach liquor used in previous tests (runs 16 and 17, ORNL-1914); for run 6 it was the same as that used in run 1. In both runs the composition and concentrations of the organic phase and the strip solution were the same as for runs 1 through 4. The operating conditions, composition of the feed streams, and the operating data are shown in Tables 6, 7, and 8, respectively. The composition of uranium products is shown in Table 9.

The data in Tables 8 and 9 compared with that for run 1 shows that, with increased loading: (1) the extraction of iron (Fe^3) and other contaminants was reduced significantly from that obtained with an aqueous to organic phase ratio of 2.5 (run 1); and (2) the number of stages needed to extract essentially all of the uranium increased from 2 with an aqueous to organic phase ratio of 2.5/1 (run 1) to 4 with a ratio of about 3.5/1 (run 6). The U_3O_8 content of the products dried for 24 hours at 130°C from runs 5 and 6 was 81 and 77%, respectively.

Future Work

Tests have been started which are a part of studies of the effects of operating variables on the rates and degrees of completion of the stripping reactions involving aqueous solutions of sodium carbonate, sodium chloride, and ammonium nitrate. In the first tests the effects of both reagent excess and mixing variables will be evaluated in batch type equipment.

TABLE 6. OPERATING CONDITIONS

Run number	5	6
Operating time, hours	4	4
Flow rates, ml/min		
Head liquor	53.9	57.4
Organic phase	18.0	16.1
Strip solution*	2.99	2.66
Extraction section		
Number of units	5	5
Size**	A	A
Agitator speed, unit 101, rpm	450	700
Agitator speed, units 102-105, rpm	450	450
Stripping section		
Number of units	2	2
Size**, unit 121	B	B
Size**, unit 122	A	A
Agitator speed, unit 121, rpm	450	450
Agitator speed, unit 122, rpm	450	450

*50% in excess of the theoretical Na_2CO_3 requirements which are: 3 moles per mole of uranium and 0.5 mole per mole of amine.

**Fig. 2, ORNL-1970.

TABLE 7. COMPOSITION OF THE FEED STREAMS

(All concentrations in grams per liter unless otherwise noted)

	Concentration	
	Run 5*	Run 6
Head Liquor		
pH	1.1	1.0
Uranium	1.1	1.2
Iron Fe^3)	1.6	5.4
Iron (Total)	2.3	5.6
Vanadium (Total)	0.76	0
Vanadium (V^5)	0	0
Titanium	0.03	0
Molybdenum	0.08	0
Aluminum	2.7	3.1
Sulfate	42.0	63.
Phosphate	0.79	2.1
Fluoride	0.2	1.0
Calcium	0.64	0
Silicon	0.21	0
Chloride	0.32	0
Nitrate	0.30	0
Organic Phase		
Amine 9D-178 molarity	0.1	0.1
Uranium	0.001	0.001
Strip Solution		
Na_2CO_3	97.	97.

*Vitro leach liquor.

TABLE 8. OPERATING DATA, EXTRACTION SECTION

Run No.	Ratio: Head Liquor/ Organic*	Mixer-Settler Number	Aqueous, g/l			Organic, g/l		
			pH	U	Mo	U	Fe	Mo
5	2.99	101	1.3	0.36	-	3.3	0.04	0.5
		102	1.3	0.011	-	0.98	0.08	-
		103	1.3	<0.001	-	0.026	0.1	-
		104	1.3	<0.001	-	0.001	0.2	-
		105	1.4	<0.001	<0.01	0.001	0.2	-
6	3.57	101	1.1	1.0	-	3.8	0.01	-
		102	1.1	0.43	-	3.3	0.04	-
		103	1.0	0.015	-	1.7	0.3	-
		104	1.0	<0.001	-	0.050	0.5	-
		105	1.1	<0.001	-	0.002	0.6	-

*From measured flow rates.

TABLE 8 (continued)

OPERATING DATA, STRIPPING SECTION

Run No.	Ratio:* Organic/ Strip	Mixer-Settler Number	Aqueous, g/l U	Aqueous, g/l Fe	Aqueous, g/l pH	Organic, g/l U	Organic, g/l Fe
5	6.0	121	20.	0.04	8.3	0.005	0.009
		122	0.14	0.006	9.9	<0.001	0.009
6	6.1	121	22.	0.04	7.9	0.003	0.03
		122	0.12	0.005	9.4	<0.001	0.01

*From measured flow rates.

TABLE 9. COMPOSITION OF URANIUM PRODUCTS

(Basis: Dried at 130°C for 24 hours)

Run Number:	Percent by Weight 5	Percent by Weight 6
Component		
Uranium as U	68.7	65.0
U_3O_8	81.0	76.6
Iron	0.25	0.23
Carbon	0.18	0.26
Molybdenum	0.10	-
Titanium	-	-
Vanadium	0.11	-
Phosphate	0.55	0.49
Loss on ignition (600°C)	4.4	

Effect of Turbulence on Phase Entrainment

In operation of a large-scale plant for the recovery of uranium by the amine extraction process, the principal loss of organic phase will come from entrainment in the raffinate (waste stream). The organic is entrained as a fog-like dispersion in the aqueous phase that persists after the primary break.* The time required for coalescence and separation of the dispersion is long in comparison with the primary break time; therefore, it is important to know how to predict the size of a settler to obtain a primary break and how much additional settler length will be required in order to recover a part or all of the residual entrainment.

The results of exploratory tests carried out under conditions simulating the raffinate end of an extraction section (ORNL-1970) indicated that, when sharp-edged orifices were used as the mixing device, the amount of entrainment in the raffinate discharged from a settler was dependent on the degree of turbulence in the mixing operation (as measured by the Reynolds number). To more closely determine the effect of turbulence on the time required for the primary break and on the amount of entrainment, tests were made in a single mixer-settler unit in which the speed of the impeller was varied from 200 to 900 rpm for each of three flow rates in the range of 1 to 4.7 gpm using a constant phase ratio. The effect of phase ratio on entrainment was also measured at a flow rate of 4 gpm.

The mixer used in the tests was a 6-in. dia. mixer column containing 5 baffled compartments with a 3-in. dia. turbine-type impeller located at the center of each compartment on a common shaft (ORNL-1914).** The compartmented column was used instead of a baffled tank because it appeared likely that variations in the degree of dispersion with flow rate would be minimized by operating it with cocurrent phase flows.

---

*Separation into discrete phases with a distinct interface.

**Construction of the mixer-column was essentially the same as that employed by Rushton and Oldshue, Chem. Engr. Progress, Vol. 48, No. 6, June, 1952, pages 297-306.

The aqueous and organic phases were metered at the desired phase ratio into the bottom of the mixer column and overflowed into the horizontal settler in which the length could be varied as shown in Fig. 2. The aqueous phase was a synthetic raffinate solution.* The organic phase was a 0.1 molar solution of amine 9D-178 (Rohm and Haas) in kerosene.

The separation rate which determines the time required for the primary break was obtained by withdrawing samples from the mixer column discharge line at a point adjacent to the column and measuring the separation rate in a 6-in. dia. container, 24-in. deep. Samples for determination of phase entrainment were collected at the discharge end of the settler. Organic entrainment in aqueous was determined by steam distillation; aqueous entrained in organic was obtained by measuring the volume collected in the bottom of a calibrated separatory funnel after 24 hours of settling. Test procedures were the same as those employed in previous tests (ORNL-1970). Experimental data are reported in Tables 10 and 11. Observations and measurements made during the tests to describe action in the settler are shown in Fig. 3.

Primary Break

The rate at which the phases separated to give the primary break with aqueous to organic phase flow ratios of 2.5:1 (Table 10) and 5:1 (Table 11) averaged about 6 in. per minute over a range of flow rates varying from 1 to 4.7 gpm (total) and impeller speeds varying from 200 to 900 rpm. These results show that with an aqueous to organic phase flow ratio of 2.5:1, the separation rate was virtually independent of the flow rate and impeller speed over the ranges employed.

The data in Table 11 show that with a flow rate of 4 gpm and impeller speeds of 200 and 900 rpm, the rate of phase separation was about 3 in. per min with an aqueous to organic ratio of 1:5 as compared to 6 in. per min with a 5:1 ratio. Since the viscosity of the organic phase was about twice that of the aqueous (1.855 and 0.989 cp., respectively, at 25°C), it appears likely that the rate of separation is largely controlled by the viscosity of the continuous phase.

---

*U, <0.1 g/l; Al, 3 g/l; Fe, 5 g/l; F, 1.5 g/l; SO_4, 40 g/l; pH = 1.0.

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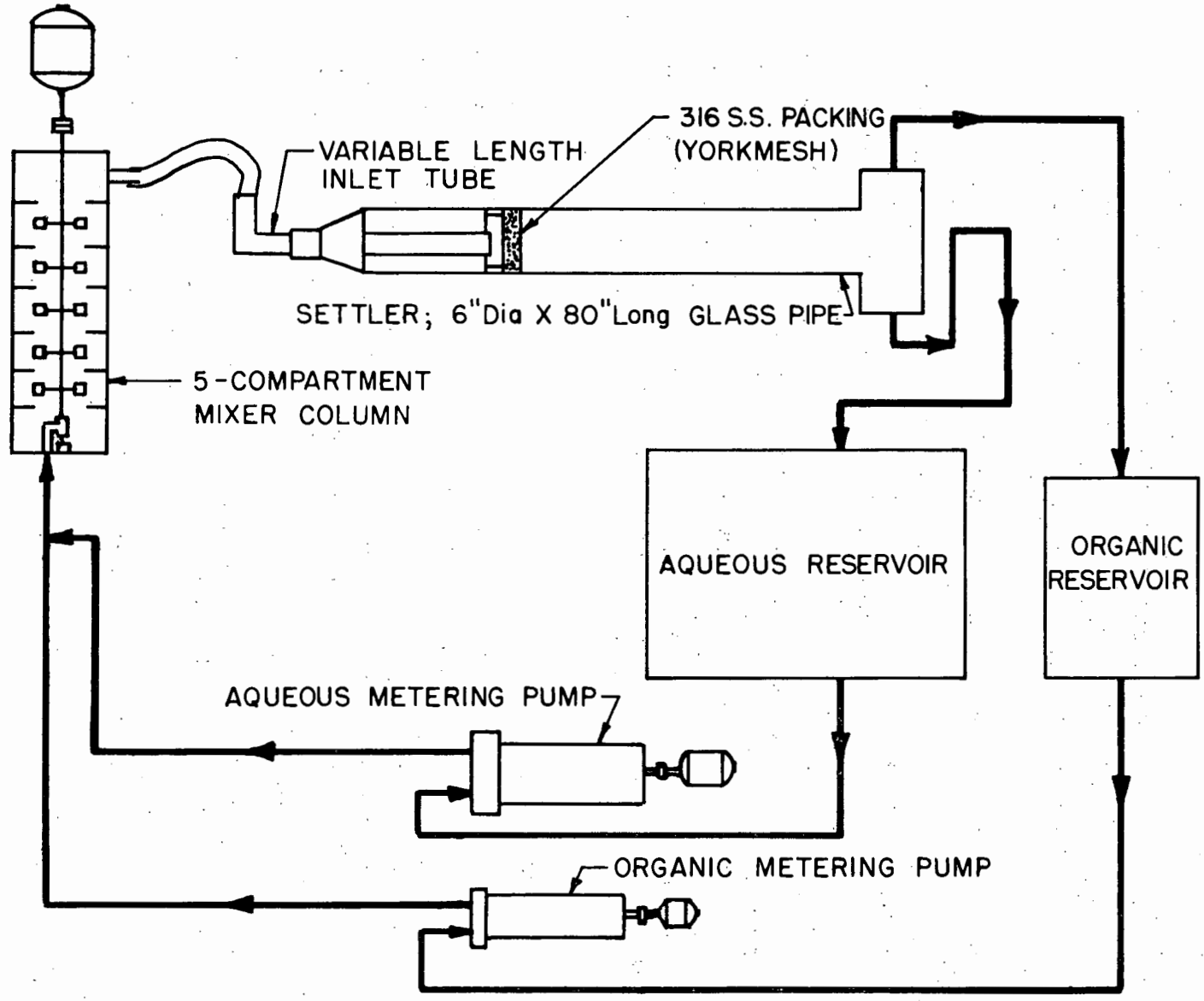

FIGURE 2. EQUIPMENT LAYOUT FOR MIXER-SETTLER PHASE SEPARATION STUDIES

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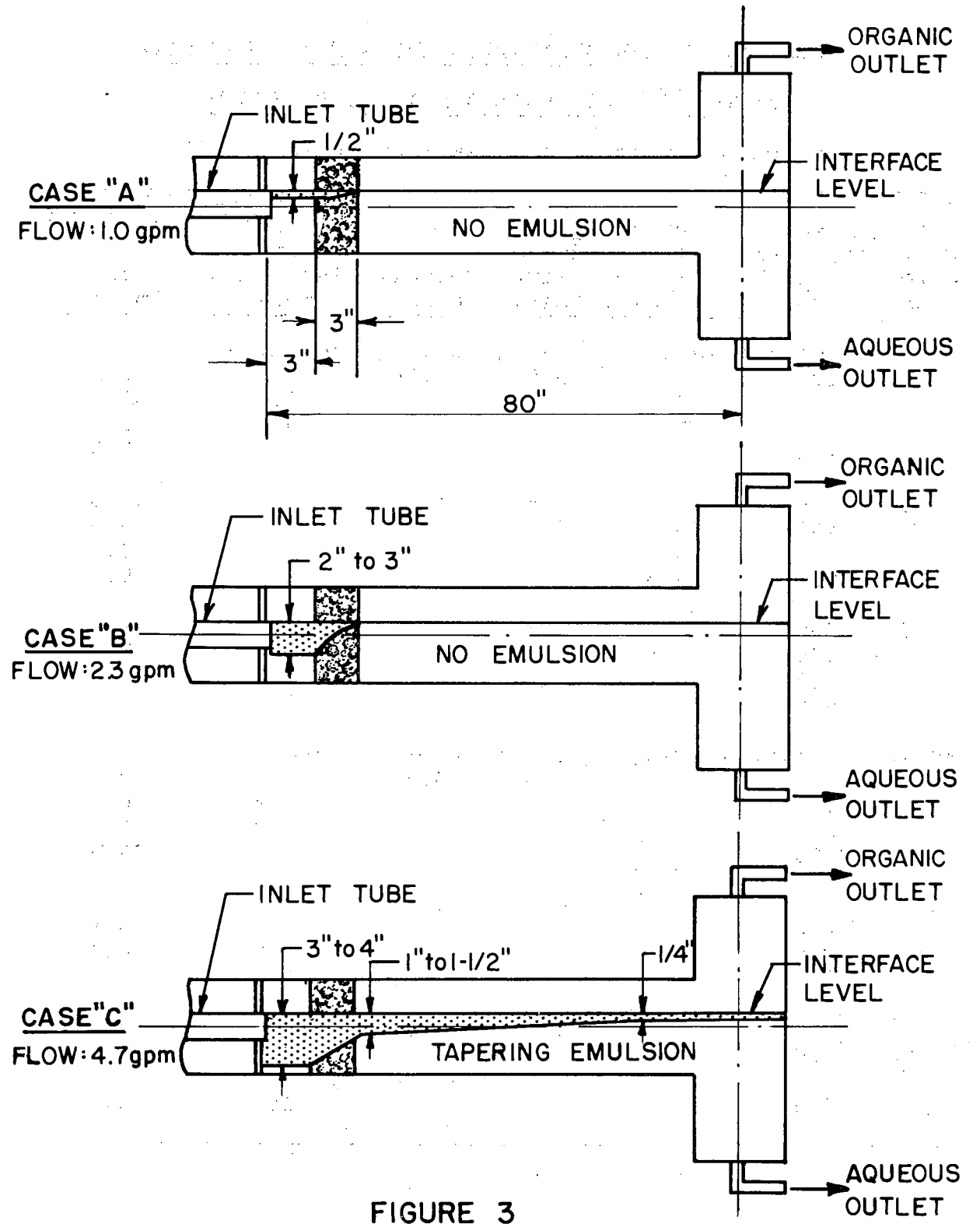

FIGURE 3

EMULSION CONDITIONS IN SETTLER AT VARIOUS FLOW RATES

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TABLE 10. PHASE SEPARATION AND ENTRAINMENT DATA

EFFECT OF FLOW RATE AND IMPELLER SPEED

(Aqueous to organic phase flow ratio 2.5:1)

Total Flow Rate (gpm)	Mixer Impeller Speed (rpm)	Separation Rate (in./min)	Settler Holdup Time (min)	Settler Velocity (fpm)	Entrainment Organic in Raffinate (ml/l)	Entrainment Raffinate in Organic (ml/l)
48.-in. settler length; 1.8 sq ft interface area						
1	200	-	5.9	0.66	0.1	<0.5
	400	5.5			0.2	<0.5
	600	5.0			0.6	<0.5
	900	5.9			0.7	<0.5
2.3	200	5.5	2.6	1.5	1.	<0.5
	400	6.4			1.	<0.5
	600	6.3			1.	<0.5
	900	6.5			2.	9.
4.7	200	6.7	1.3	3.1	4.	1.
	400	6.5			3.	1.
	600	6.4			2.	4.
	900	6.1			2.	12.
80-in. settler length; 3 sq ft interface area						
1	200		9.8	0.66	<0.1	<0.5
	400				0.2	<0.5
	600				0.2	<0.5
	900				0.4	<0.5
2.3	200		4.3	1.5	0.8	<0.5
	400				0.6	<0.5
	600				0.9	<0.5
	900				1.0	6.
4.7	200		2.1	3.1	3.	4.
	400				2.	6.
	600				1.	12.
	900				1.	18.

TABLE 11. PHASE SEPARATION AND ENTRAINMENT DATA

EFFECT OF PHASE RATIO

Total flow rate, 4 gpm
Linear velocity in settler, 2.7 fpm

Impeller speed (rpm)	Phase Flow Ratio, aq:org	Separation Rate, (in./min)	Settler Length (in.)	Settler Holdup Time (min)	Entrainment org in aq (ml/l)
200	5:1	6.5	48	1.5	4.
			80	2.4	3.
	1:5	2.9	48	1.5	0.2
			80	2.4	0.2
900	5:1	6.1	48	1.5	6.
			80	2.4	4.
	1:5	2.4	48	1.5	0.4
			80	2.4	0.4

*Settling area: 1.5 sq ft for 48-in. settler and 2.6 sq ft for 80-in. settler.

Organic Entrainment

The effects of flow rate and impeller speed for the 48-in. settler length are shown in Fig. 4 which is a plot of entrainment, E, and linear velocity in the settler, V, for each impeller speed.

With a constant impeller speed, E increased as V was increased and no apparent functional relationship between V and E exists. There was a marked difference in the effect of impeller speed on E as V was increased from 0.66 to 3.1 fpm. With V at 0.66 fpm, there was a well-defined increase in E as the impeller speed was increased; while with V at 3.1 fpm, E decreased as the impeller speed was increased. Similar trends are shown by the data for the 80-in. settler length and, in addition, the larger settler was from 25 to 100% more effective in reducing the amount of entrainment.

Rushton* has shown that for fully developed turbulent flow in mixers, turbulence is proportional to the total head, H, which in turn is proportional to N^2D^2 where N and D are the impeller speed and diameter, respectively. It follows that the totality of small droplets that represent entrainment as well as the droplet size distribution are proportional to H and that an increase in H would be accompanied by a corresponding increase in entrainment. The contradictory results obtained when the velocity was 3.1 fpm indicates that between 1.5 and 3.1 a critical velocity exists above which streamline settling cannot occur. In this connection, it is estimated that flow in the settler would have become turbulent with V at 4 fpm.

For purposes of cost estimating, mixing impeller speed and linear velocity in the settler have been set tentatively at 400 rpm and 0.66 fpm, respectively.

Effect of Phase Ratio. The data in Table 11 show that with the average velocity in the settler at 2.67 fpm and mixer impeller speeds of 200 and 900 rpm, entrainment ranged from 3 to 6 ml/l with an aqueous to organic phase flow ratio of 5 to 1 and from 0.2 to 0.4 ml/l with a phase flow ratio of 1 to 5. However, the volumetric flow rate of the raffinate phase will control the amount of organic lost as entrainment and at a total flow of 4 gpm with an aqueous to organic ratio of 1:5, the raffinate rate was only about 0.67 gpm. In comparison, entrainment was only 0.1 ml/l in the tests made at a total flow rate of 1 gpm and a phase ratio of 2.5:1.

*J. H. Rushton and J. Y. Oldshue, Chem. Engr. Progress, Vol. 49(1953), No. 4, pages 161-168; No. 5, pages 267-275.

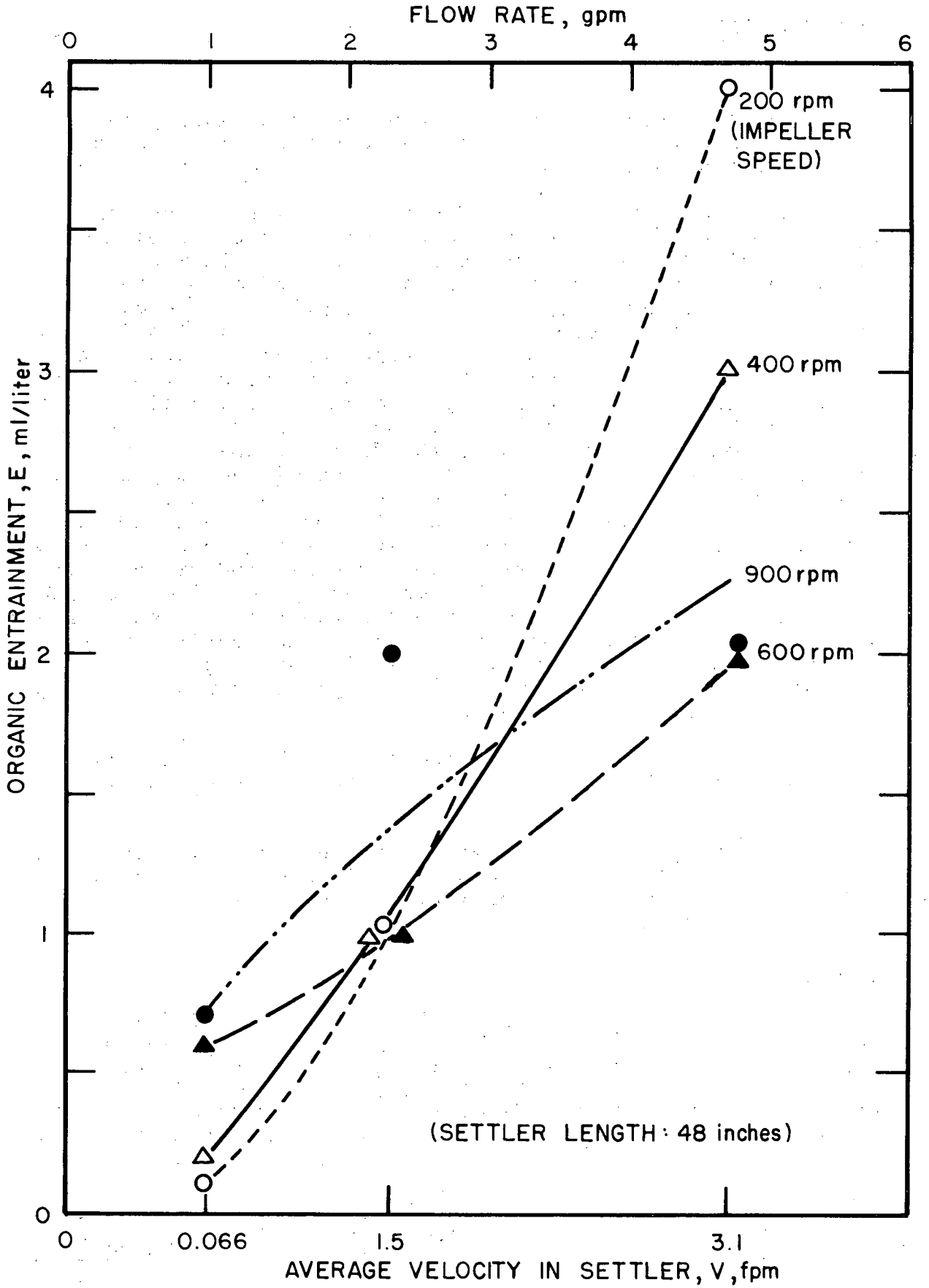

FIGURE 4. SETTLER PERFORMANCE

Consequently, operating with an aqueous to organic phase ratio of 1:5 does not appear to offer any advantage in lowering organic entrainment loss.

Static Settling Tests. The results of tests showing the effects of velocity on entrainment suggest the use of a small settler, sized to obtain the primary break, followed by one large enough so that coalescence and separation of the entrained organic can take place under near static conditions. Therefore, large batches of raffinate were collected for static settling tests from the settler discharge while carrying out the tests at 4 and 4.7 gpm flow rates described previously. The batches were large enough to fill a 10-in. dia. glass tank to a depth of about 12 in. and samples for steam distillation were withdrawn at the desired time intervals from the bottom of the tank. The volume of the samples was equivalent to 1 inch of depth in the settling tank; consequently the values for entrainment reported in Table 12 represent residual entrainment after settling for the indicated time interval. With the aqueous phase predominant, the settling time increased as the impeller speed increased. This is shown in Fig. 5 which is a plot of the residual entrainment and time for the four impeller speeds. The settling time required to reduce entrainment to 0.1 ml/l was approximately 13 min with an impeller speed of 200 rpm, 50 min with 400 rpm, and 80 min with 600 and 900 rpm.

With the organic phase predominant, the settling time was markedly lower than it was when the aqueous phase was in excess.

The data in Table 12 and Fig. 5 are useful in predicting minimum holdup time for recovery of organic from raffinate entrained under a variety of mixing conditions.

Aqueous Entrainment

The data in Table 10 show that with aqueous to organic phase ratios of 2.5:1, the amount of aqueous entrainment in organic was less than 0.5 ml/l in both settler lengths with 1 and 2.3 gpm flow rates and impeller speeds of 600 rpm or less. With impeller speeds of 900 rpm, aqueous entrainment ranged from 0.5 ml/l at 1 gpm flow rate to 18 ml/l at 4.7 gpm.

TABLE 12. STATIC SETTLING RATES

ORGANIC ENTRAINMENT

Impeller Speed (rpm)	Phase Flow Ratio, aq:org	Total Flow Rate (gpm)	Organic Entrainment (ml/l) Settling Time (min) 0	10	20	30	60	240
200	5:1	4	3.	-	0.3	0.3	<0.1	<0.1
900			4.	0.7	0.6	0.5	0.2	0.1
200	2.5:1	4.7	3.	0.2	<0.1	<0.1	<0.1	<0.1
400			2.	0.4	0.3	0.2	<0.1	<0.1
600			1.	0.5	0.3	0.3	0.2	<0.1
900			1.	0.4	0.3	0.3	0.2	<0.1
200	1:5	4	0.2	0.2	0.2	0.2	0.2	0.1
900			0.4	0.2	0.2	0.2	0.1	<0.1

UNCLASSIFIED

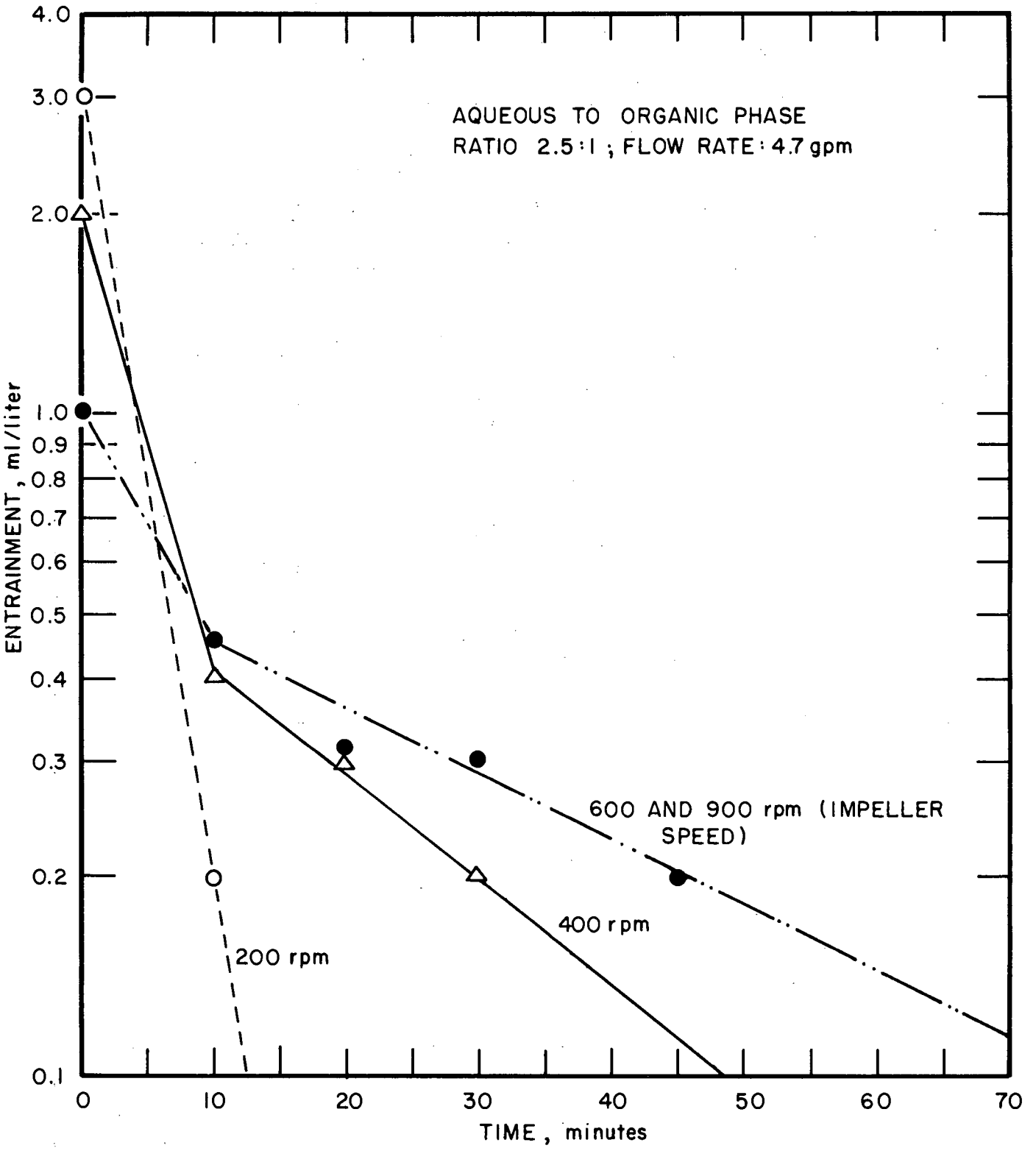

FIGURE 5. STATIC SETTLING TIME

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Entrainment of aqueous phase in organic amounting to considerably more than the maximum of 18 ml/l would not appreciably affect the uranium extraction efficiency.

Future Work

In continuation of the present study, tests are in progress to more closely define the length of settler needed to obtain the primary break with linear velocities of the separated phases ranging from 0.66 to 3.1 fpm, and to determine the amount of entrainment over a range of holdup time with a constant velocity in the settler.

SLURRY EXTRACTION

In the preceding period work was started on testing the utility of a laboratory centrifugal pump as a mixer for contacting leach slurries containing 50-60 wt % solids with organic extractant. The uranium extraction efficiency* obtained in a pump mixer-settler test unit was about 60% and organic entrainment in the slurry raffinate discharged from the settler was about 25 gal per ton of ore. In continuation of the tests, runs were carried out with several modifications in the equipment piping in attempts to increase the extraction efficiency and decrease the amount of organic entrained in the slurry raffinate. A summary of the results obtained to date for all runs is given in the following.

Details of the pump-mixer-settler test unit are shown in Fig. 6. The composition of the leach slurry and organic phase are reported in Table 13. The operating conditions are shown in Table 14.

A summary of the data and test results that were obtained after the system was at equilibrium is shown in Table 15. Extraction efficiencies were calculated from the uranium content of the raffinate solution (filtrate) at steady state and after equilibration with the organic extract*; therefore, the values reported are approximations of the true efficiencies.

The maximum extraction efficiency of about 90% was obtained in run 3 using the larger laboratory centrifugal pump. The amount of organic phase entrained in the slurry raffinate withdrawal stream ranged from 19 to 25 gal per ton of ore.

These results do not show sufficient promise to warrant continuation of the testing of pump mixers.

Tests are planned to determine the maximum amount of entrained organic that can be recovered from slurry raffinate by dilution with water and gentle agitation. It is expected that the results of these tests will show whether the entrainment is mechanical in nature or due to adsorption.

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*Extraction efficiency: $E = \frac{C_o - C}{C_o - C_e}$ where C_o = g/l U in solution in the slurry feed, C = g/l U (ave) in solution in raffinate withdrawal, and C_e = g/l U in solution in raffinate withdrawal after equilibration with organic extract.

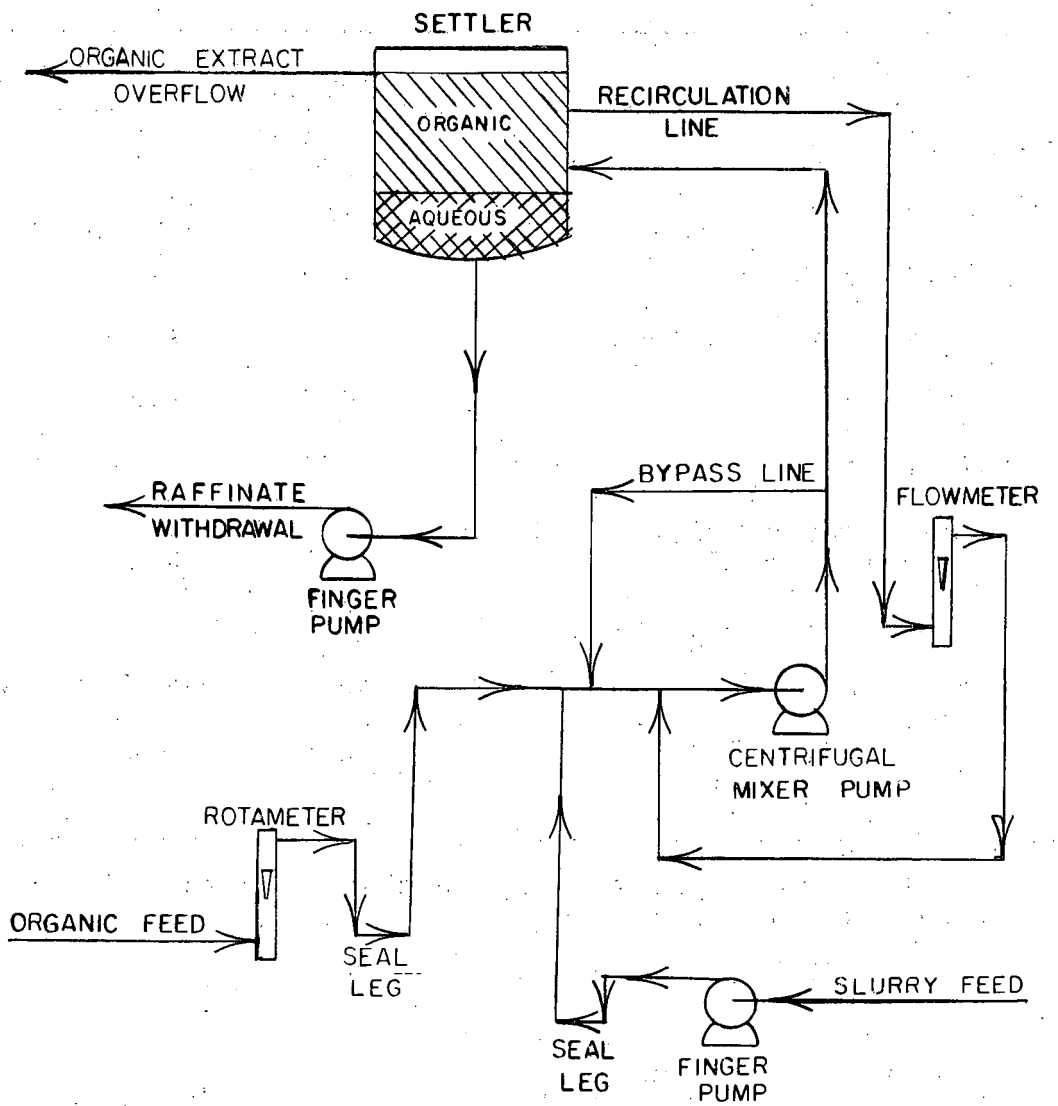

FIG.6 - PUMP MIXER-SETTLER TEST UNIT

TABLE 13. COMPOSITION OF THE FEED STREAMS

	Run 1	Run 2	Run 3
Leach slurry 1/			
Solids, wt %	55	60	50
Density, g/cc	1.57	1.65	1.5
U, g/l in solution	1.28	1.77	0.86
pH	0.9	1.3	1.5
Organic phase			
Di-2-ethylhexyl phosphoric acid, molarity	0.1	0.1	0.1
Capryl alcohol, vol %	2	2	2
Kerosene, vol %	98	98	98
Density, g/cc	0.816	0.816	0.816

1/ The slurry was prepared by leaching -20 mesh Uravan ore with sulfuric acid.

TABLE 14. SUMMARY OF OPERATING CONDITIONS

	Run 1	Run 2	Run 3
Mixer pump speed, rpm	4400 1/	4400 1/	3400 2/
Flow rates, cc/min			
Slurry feed	150	150	150
Organic feed	75	75	75
Organic recycle	7000	1500	4400
Pump bypass	0	6000	9400
Holdup, cc			
Slurry in the system	650	750	800
Organic phase in the system	2000	2000	2500
Volume of bypass line	0	340	340

1/ Eastern Model A-1, capacity: 7.6 l/min at 3 ft head and 4400 rpm.

2/ Eastern Model DB, capacity: 16 l/min, at 3 ft head and 3400 rpm.

TABLE 15. SUMMARY OF OPERATING DATA AND RESULTS

	Run 1	Run 2	Run 3
Organic extract			
U, g/l	0.71	0.60	1.12
Slurry raffinate, U, g/l in solution	0.68	0.78	0.130
At equilibrium			
U in solution, g/l	0.003	0.088	0.057
U in organic, g/l	0.82	0.69	1.32
Uranium extracted, % of feed	47	56	85
Stage efficiency, %	47	59	91
Organic entrainment in slurry raffinate			
gal/ton of ore	19	25	24