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PROGRESS REPORT OF PILOT PLANT SECTION

RAW MATERIALS PROCESS TESTING

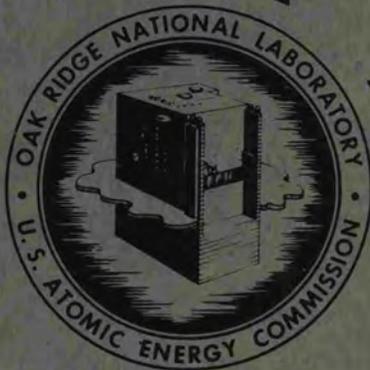
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PROGRESS REPORT OF PILOT PLANT SECTION
RAW MATERIALS PROCESS TESTING

January, 1955

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PROGRESS REPORT OF PILOT PLANT SECTIONRAW MATERIALS PROCESS TESTING

January, 1955

1. EXTRACTION OF URANIUM AND VANADIUM
WITH AN AMINE REAGENT

Studies by the Uranium Chemistry of Raw Materials Section on the amine extraction process have shown that the process has possible application for recovery of both uranium and vanadium from sulfuric acid leach liquors. Therefore, three runs have been made to test this application in bench-scale countercurrent mixer-settlers.

The runs were carried out in a mixer-settler test array which contained an extraction section, a uranium stripping section, and a vanadium stripping section as shown in Fig. 1. The two different size mixer-settler units employed in the test array are shown in Fig. 2, and their arrangement is given in Table 1 together with other operating conditions.

The head liquor for runs 1 and 2 was prepared by adding uranium concentrate to the raffinate from previous runs on a leach liquor from Plant A (runs 16 and 17, ORNL-1914). The head liquor for run 3 was a leach liquor from Plant A. Prior to starting the runs the liquors were treated with sufficient manganese dioxide to oxidize the vanadium to the pentavalent state and the pH values were adjusted to about 2.0 in order to increase the vanadium extraction coefficients (ORNL-1734). In making the runs, uranium and vanadium were extracted together and each was separately stripped from the organic extract in the following manner: first the uranium was removed by contacting the organic extract with an aqueous sodium chloride solution; the vanadium was then stripped by contacting the organic with an aqueous sodium carbonate solution. At the end of a run each mixer-settler unit was sampled to obtain stagewise concentrations (gradients) of the major components. The analysis of the head liquors, organic phase and strip solutions are shown in Table 2. The operating data are reported in Table 3. Table 4 shows an analysis of the uranium and vanadium products that were recovered for each run.

Emulsion Formation. An emulsion formed in the extraction section in all three runs and in the uranium stripping section in run 3. It was less severe in run 1 than in runs 2 and 3. Varying the agitator speed from 250 to 600 rpm had no appreciable effect.

In duplicate runs made previously with Plant A liquors at a pH of 1.1, only a small amount of emulsion was observed at the raffinate end of the extraction section in one run and none with the other. Since the pH was 1.7 in run 1 and about 2.1 in runs 2 and 3, it appears that emulsions may form more readily as the pH is increased.*

Some of the emulsion from the uranium stripping section in run 3 was collected and centrifuged, yielding a small quantity of solids. A spectrographic analysis of the solids after they were washed with 1 molar sodium chloride solution, water, and then dried, showed that they contained:

Aluminum	0.5%
Iron	5
Silicon	5
Uranium	7
Vanadium	5

The high silicon content indicates that the emulsion was due to a carryover of that formed in the extraction section. Because of the emulsion, the data collected from the stripping were not reported for run 3.

*The uranium distribution coefficients are large even with head liquors at a pH less than 1.0. Therefore, for uranium extraction, it will not be necessary to adjust to pH values greater than 1.0.

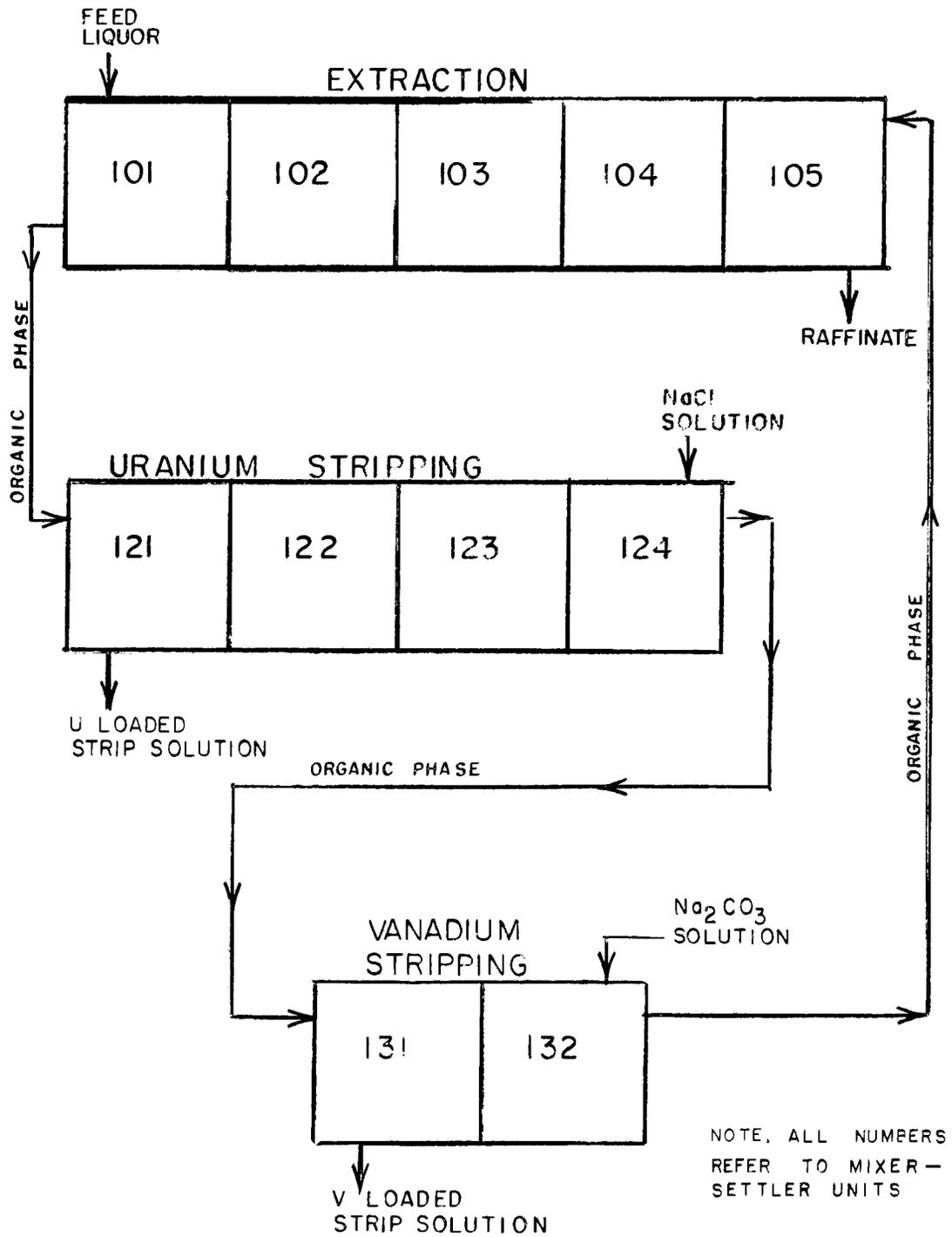
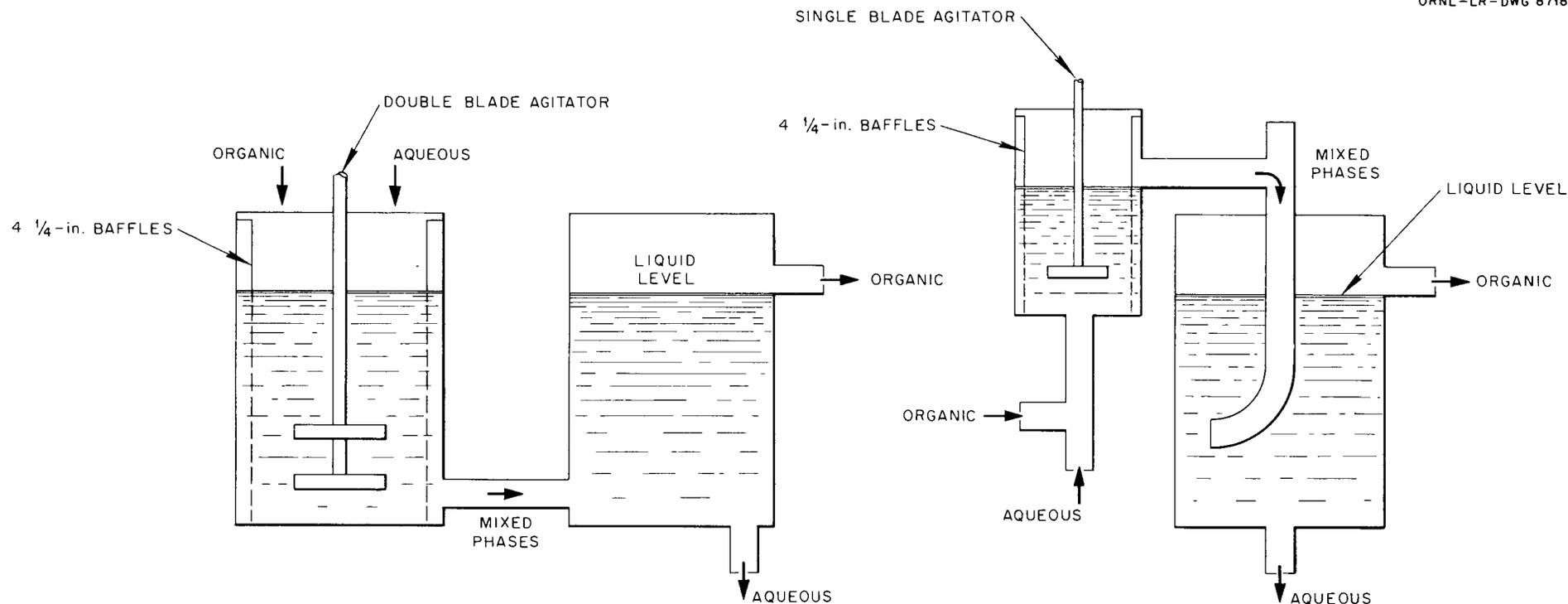


FIG. 1. FLOW DIAGRAM FOR RUNS 1 THROUGH 3.



TYPE A

MIXER: 500 ml TALL FORM BEAKER
OPERATING VOLUME 375 ml

SETTLER: 500 ml TALL FORM BEAKER
OPERATING VOLUME 375 ml

TYPE B

MIXER: 200 ml TALL FORM BEAKER
OPERATING VOLUME 125 ml

SETTLER: 500 ml TALL FORM BEAKER
OPERATING VOLUME 375 ml

Fig. 2. Types of Mixer-Settlers.

TABLE 1. OPERATING CONDITIONS

Run number	1	2	3
Operating time, hours	6	2	6
<u>Flow rates, ml/min</u>			
Head liquor	50	50	50
Organic phase	25	25	25
U strip solution	5		
V strip solution	5		
<u>Extraction section</u>			
Number of units	4	4	5
Size (Fig. 2)	A	A	A
Agitator speed, rpm	470	470	470
<u>U stripping section</u>			
Number of units	4		
Size	B		
Agitator speed, rpm	870		
<u>V stripping section</u>			
Number of units	2		
Size, unit 131	B		
Size, unit 132	A		
Agitator speed, unit 131, rpm	1050		
Agitator speed, unit 132, rpm	210		

TABLE 2. COMPOSITION OF THE FEED STREAMS

(All concentrations in grams per liter unless otherwise noted)

	Concentration		
	Run 1	Run 2	Run 3
<u>Head Liquor*</u>			
pH	1.7	2.1	2.2
Uranium	1.12	1.09	1.22
Iron	2.3	2.2	2.2
Vanadium, V ⁺⁵	0.65	0.63	0.63
Total vanadium	0.66	0.64	0.67
Aluminum	2.6	2.6	2.6
Molybdenum	0.008	0.008	0.08
Titanium	<0.005	<0.005	0.03
Calcium	0.66	0.66	0.65
Silicon	0.13	0.13	0.26
Phosphate	0.87	0.87	0.70
Sulfate	47.5	41.5	42.0
Chloride	0.43	0.43	0.41
Nitrate	0.3	0.3	0.29
Fluoride	0.25	0.25	
<u>Organic Phase</u>			
Rohm and Haas amine EB 765-2, molarity	0.099	0.099	0.099
Uranium	0.001	0.004	0.001
Iron	0.001	0.001	0.001
<u>U Strip Solution</u>			
Sodium chloride	58		
<u>V Strip Solution</u>			
Sodium carbonate	92.1		

*Liquor for runs 1 and 2 was prepared by adding uranium concentrate to the raffinate from previous runs on Plant A liquor (runs 16 and 17, ORNL-1914). Plant A leach liquor was used in run 3.

TABLE 3. OPERATING DATA, EXTRACTION SECTION

Run No.	Volumetric Phase Ratio in Mixer aq/org	Organic Entrainment in Raffinate ml/l.	Mixer-Settler Number	Aqueous, g/l.				Organic, g/l.			Distribution org/aq		
				pH*	U	V**	V ⁺	U	V	Fe	U	V	Fe
1	1.96	0.1	101	1.7	0.100	0.63	0.60	2.38	0.82	0.08	24	1.3	0.03
			102	1.7	0.001	0.53	0.53	0.15	0.75	0.24	150	1.4	0.11
			103	1.7	0.001	0.48	0.48	0.004	0.69	0.36	-	1.4	0.17
			104	2.0	0.001	0.21	0.19	0.001	0.61	0.46	-	2.9	0.22
2	2.08	-	101	2.2	0.072	0.22	0.17	2.28	1.4	0.06	32	6.4	0.03
			102	2.2	0.002	0.18	0.14	0.32	0.62	0.27	160	3.4	0.12
			103	2.1	0.001	0.16	0.13	0.050	0.29	0.32	-	1.8	0.15
			104	2.0	0.001	0.12	0.11	0.04	0.23	0.28	-	1.9	0.13
3	2.36	0.07	101	2.4	0.51	0.19	0.12	2.46	1.95	0.05	5	10.	0.02
			102	2.5	0.009	0.13	0.10	1.35	1.13	0.11	150	8.7	0.05
			103	2.5	0.001	0.12	0.07	0.060	0.68	-	-	5.7	-
			104	2.4	0.001	0.11	0.06	0.005	0.39	0.28	-	3.5	0.12
			105	2.4	0.001	0.08	0.06	0.004	0.19	0.32	-	2.4	0.14

*In runs 2 and 3 the stripped organic phase was contacted with dilute sulfuric acid prior to pumping it back to the extraction cycle in order to maintain a more nearly constant pH in the extraction section.

**Total vanadium.

TABLE 3. OPERATING DATA, STRIPPING SECTION

Run No.	Strip Section	Mixer-Settler Number	Aqueous, g/l.						Organic, g/l.				Distribution org/aq	
			pH	U	V	Fe	Cl	SO ₄	U	V	Cl	SO ₄	U	V
1	U	121	1.6	**11.0	0.90	0.24	28.7	17.2	0.028	0.77	3.2	<0.5	0.003	0.86
		122	1.8	0.57	0.35	0.06	36.7	3.6	0.001	0.77	3.2	<0.5	0.002	2.2
		123	2.1	0.018	0.15	0.02	36.3	<0.5	<0.001	0.75	3.3	<0.5		5.0
		124	2.5	0.003	0.055	0.006	35.7	<0.5	<0.001	0.75	3.2	<0.5		13.6
	V	131	8.9	0.006	**3.35		16.8		<0.001	0.01	<0.1			
		132	0.8	<0.001	0.1	-	0.3	-	<0.001	<0.01	<0.1	-		

*No stripping data were obtained during run 2 because of the short duration of the run. The data collected during run 3 are not reported because of emulsion difficulties in the uranium stripping section.

**Average of last two hourly samples.

TABLE 4. ANALYSES OF URANIUM AND
VANADIUM PRODUCTS; RUN 1

(Basis: Product dried 24 hrs at 130°C)

	Uranium Product (%)	Vanadium Product (%)
Uranium	59.9	<0.001
Uranium as U ₃ O ₈	70.6	-
Vanadium	4.2	44.8
Vanadium as V ₂ O ₅	7.5	80.0
Iron	1.5	-
Chloride	<0.04	-
Carbon	0.08	-
Phosphate	-	0.110
Molybdenum	-	2.3

NOTE: Spectrographic analyses of the products showed that the concentration of trace elements (other than Na) was less than 1%.

Uranium Extraction. Uranium extraction was satisfactory in all three runs; the uranium concentration in the raffinate was below 0.003 grams per liter in less than two stages in runs 1 and 2 and between two and three stages in run 3. As expected, the uranium distribution coefficients were higher in these runs than those normally obtained when the pH of the head liquor was 1.0 (ORNL-1734). The coefficients averaged about 28 in unit 101 in runs 1 and 2 while it was only 5 in run 3, probably because the emulsion was more severe during run 3. In all three runs the coefficients ranged between 150 and 160 in unit 102.

Vanadium Extraction. Vanadium extraction was not complete in any run; 29% of the V⁺⁵ remained unextracted in run 1 after four mixer-settler units, 19% in run 2, and 17% in run 3. With all five units in run 3, 12% remained unextracted. These results show the increase in vanadium extraction as the pH of the feed liquor was increased from 1.7 in run 1 to 2.2 in run 3.

In laboratory studies, vanadium extraction coefficients as high as 80 were obtained when extracting vanadium from an acidic aqueous phase with dilaurylamine. The liquor contained vanadium sulfate (V^{+5}), sodium sulfate and sulfuric acid only, and the sulfate concentration and pH were adjusted to 0.5 M and 2.0, respectively, prior to the extraction.

In runs 2 and 3 with a sulfate concentration in the leach liquor of about 0.5 M and a pH of 2.0, the vanadium extraction coefficients ranged from only 2 to 10. The low extraction coefficients may have been caused by either the effects of ions other than sulfate, the use of a different amine as the extraction agent, or by a combination of these factors.

Uranium Stripping and Product Purity. Essentially complete stripping of uranium from the organic phase was obtained in two stages in run 1; however, a significant amount of vanadium was also stripped along with uranium. The data from Tables 2 and 3 show that essentially all of the uranium and 15 percent of the vanadium (V^{+5}) in the head liquor was contained in the loaded strip solution. A chemical and spectrographic analysis of the washed and dried uranium product, which was precipitated as sodium diuranate, is given in Table 4. The uranium content as U_3O_8 was about 71 percent while that of the principal impurities, vanadium and iron, was 7.5 percent V_2O_5 and 2.1 percent Fe_2O_3 . Loss on ignition was not determined.

Because of the short duration of run 2 (2 hours), the data collected from the stripping section were not reported.

Vanadium Stripping and Product Purity. Less than two stages were needed to strip vanadium from the organic phase (<0.01 g/l. V in stripped organic). An analysis of the vanadium product which was precipitated as "red cake" is given in Table 4. The V_2O_5 content of the product was about 80 percent and the principal impurity was Mo which amounted to about 2.3 percent. The over-all vanadium recovery amounted to about 70 percent of that contained in the head liquor.

Future Work

The present study represents the first continuous counter-current test of the applicability of the amine process to recovery of both uranium and vanadium by simultaneous extraction with separate stripping. This variation of the amine process appears promising, but further pilot-scale tests should await additional laboratory studies of leach liquors containing high concentrations of vanadium. Specifically, studies must be directed toward solving the problems of emulsion formation with higher pH of leach liquors and increasing the vanadium extraction efficiency.

2. MIXING AND PHASE SEPARATION STUDIES

Previously (ORNL-1914) an investigation was started to determine the effect of the mixing variables upon the extraction efficiency, the rate of settling and the amount of phase entrainment. Also a horizontal settler was constructed to study the effect of settler geometry upon the separation of phases and entrainment. The equipment has been tested and several revisions made to improve the flow pattern in the settler.

Some preliminary results have been obtained using a centrifugal pump and two sizes of orifices as mixers.

Equipment

The equipment layout is shown in Fig. 3. The aqueous and organic phases were metered from their respective storage tanks to the mixing unit. The dispersed phases leaving the mixer were separated in a continuous settler which consisted of an 84-inch length of 6-inch glass pipe with a 6-in. x 2-in. bell reducer on the inlet end and a 6-inch tee on the discharge end. The effective length of the settler was changed by sliding the inlet tube in or out. A 3-inch section of York Demister screen distributed the high velocity discharge stream of the inlet tube and prevented eddy currents in the settler. A Tygon gasket around the end of the inlet tube blocked off the unused portion of the settler. The interface position was controlled by a gravity leg in the aqueous discharge line.

Testing Procedure

To start a run, the aqueous and organic flows were adjusted to the desired values and the position of the settler inlet tube was fixed. The system was then allowed to operate until the interface in the settler held a constant level; this level was adjusted so that the linear velocities of the individual phases in the settler were approximately the same. When the desired conditions were established, time was allowed for about 10 volume changes in the system after which entrainment samples were taken at the phase discharge points.

The effect of additional settling time upon entrainment was measured by collecting a large volume of the aqueous

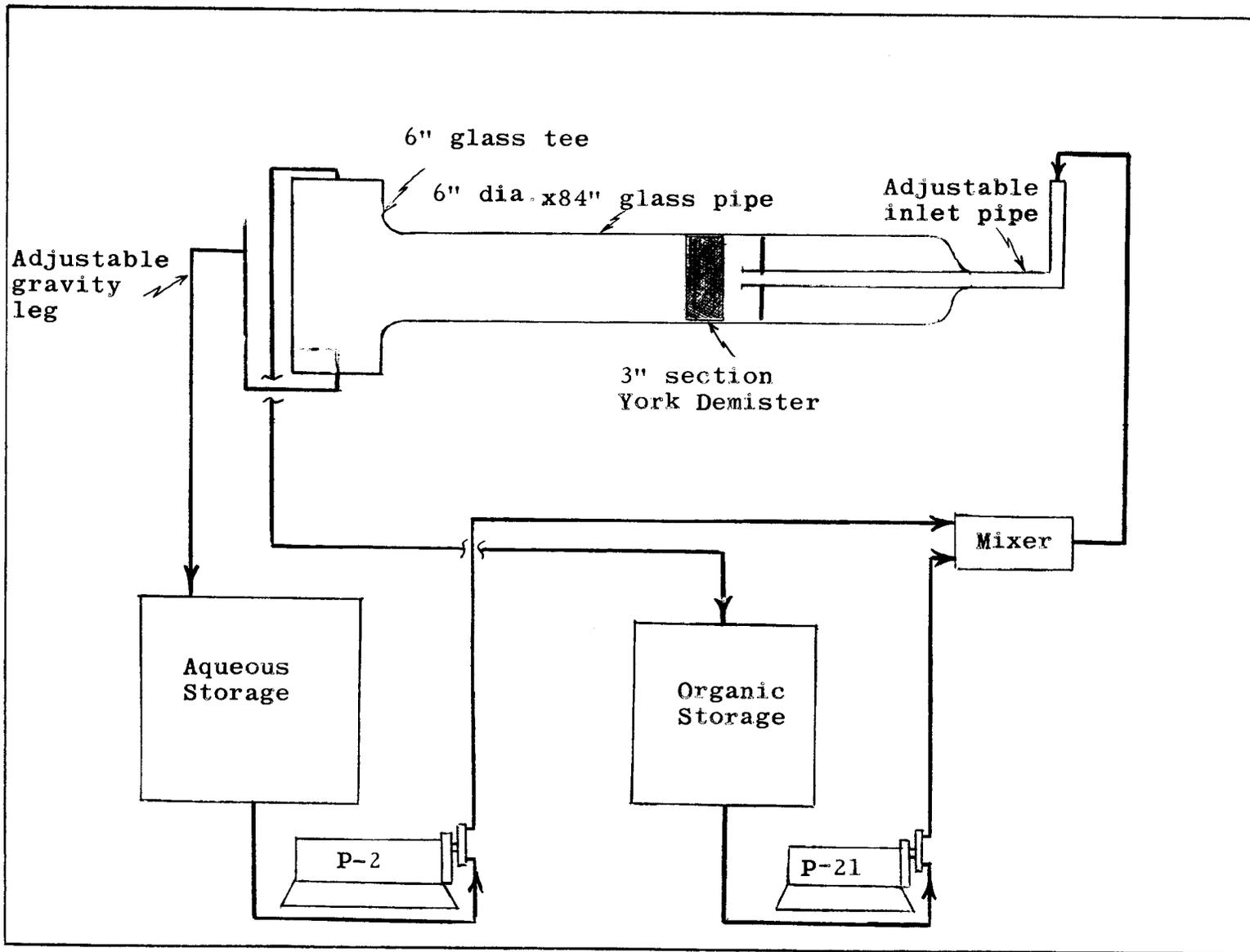


Figure 3. EQUIPMENT FOR STUDY OF MIXING AND PHASE SEPARATION

phase and withdrawing samples after desired time intervals. Entrainment of aqueous in the organic phase was determined volumetrically, while entrainment of the organic phase in the aqueous was measured by steam distillation.

After the samples were collected, the flow from the mixer was diverted from the horizontal settler to a 6-in. diameter batch settling tank, and the settling rate was determined by filling the tank to a depth of 18 in. and then noting the rate at which the emulsion band diminished.

Operating Conditions

The aqueous phase for all runs was a synthetic Marysville liquor from which the uranium had been extracted so that the mixing and settling tests simulated the conditions at the raffinate end of an extraction plant where a low entrainment of the organic phase is desired. The organic phase was a 0.1 M solution of Rohm and Haas amine (EB 765-2) in kerosene. In runs 1 through 3, the organic phase contained 5% capryl alcohol by volume. All runs were made at room temperature (20° to 25°C).

Pump Mixer. When the centrifugal pump (Eastern Model A-1) was used as a mixer, the aqueous and organic phases were fed to the pump by the respective metering pumps. The speed of the centrifugal pump was about 4000 rpm and the discharge pressure was maintained at 10 psig by a throttling valve. Under these conditions the degree of dispersion was higher than that normally produced by tank type mixers.

Orifice Mixers. When either the 1/8-in. or 1/4-in. orifice was used as the mixer the phases were metered to the orifice by separate pumps. The Reynolds numbers for the 1/4-in. orifice were: 10,000 at 1.0 gpm; 23,000 at 2.3 gpm; and 47,000 at 4.7 gpm. For the 1/8-in. orifice the Reynolds numbers at 1.0 and 2.3 gpm total flow were 21,000 and 46,000, respectively. The 1/8-in. orifice was not tested at 4.7 gpm because of the high head required at this flow.

Results and Discussion

The results of the tests of two different types of mixers (centrifugal pump and orifice) are shown in Tables 5 and 6. For each, the initial separation into two discrete layers,

TABLE 5. PHASE SEPARATION IN A PUMP MIXER-SETTLER

Run Number	Total Flow Rate (gpm)	Phase Ratio aq/org	Batch Settling Rate (in./min)	Entrainment of Organic in Aqueous Phase (ml/liter)							Entrainment Aqueous in Organic (ml/liter)
				Continuous Settler: length,		After Additional Static Settling for:					
				48 in.	80 in.	10 min	20 min	30 min	60 min	4 hrs	
1*	1.0	2.5	9.2	0.8	1.7	-	-	-	-	-	14
2*	2.3	2.5	10.2	0.6	1.0	-	-	-	-	-	18
3*	4.7	2.5	10.2	0.9	1.3	-	-	-	-	-	17
4	2.3	2.5	7.4	0.5	0.4	-	-	-	-	-	28
5	4.7	2.5	-	1.8	-	0.7	0.5	0.4	0.2	<0.1	5
6	1.0	0.2	3.8	0.1	0.6	1.0	0.8	0.7	<0.1	<0.1	3
7	1.0	5	10.2	1.6	2.8	1.6	0.8	0.7	0.1**	<0.1	1

*Organic phase contained 5% capryl alcohol

**Sample taken after 2.5 hours.

TABLE 6. PHASE SEPARATION IN AN ORIFICE MIXER-SETTLER

Run Number	Total Flow Rate (gpm)	Re* (mixer)	Phase Ratio aq/org	Batch Settling Rate (in./min)	Entrainment of Organic in Aqueous Phase, ml/l.							Entrain- ment Aqueous in Organic (ml/liter)
					Continuous Settler Length		After Additional Batch Settling for:					
					48 in.	80 in.	10 min	20 min	30 min	60 min	4 hrs	
1/4 in. diameter orifice												
8	1.0	10,400	2.5	9.9	0.9	0.2	-	-	-	-	-	1
9	2.3	22,800	2.5	7.0	0.9	0.7	-	-	-	-	-	14
10	4.7	46,500	2.5	7.7	6.9	2.5	0.7	0.6	0.5	0.2	0.1	100
1/8 in. diameter orifice												
11	1.0	20,800	2.5	8.5	3.3	1.0	-	-	-	-	-	11
12	2.3	45,600	2.5	8.2	2.5	2.9	1.7	1.3	0.9	0.6	0.2	5

*Reynolds number.

(sometimes called the primary break), and the residual entrainment of fine droplets of each phase in the other were evaluated. The former is shown in the data by the batch settling rate, and the latter by the measured entrainment in the continuous settler effluent supplemented with additional settling time.

Settling Rate. The results show that the settling rate of the mixed phases was very nearly constant (7 to 10 in. per min) under a variety of conditions. The settling rate was independent of the type of mixer as well as the phase flow rates. Also no effect of the presence of capryl alcohol was noted. The only significant change in the rate of settling was observed in run 6 where the organic to aqueous ratio was 5 to 1. In this run the settling rate was about 4 in. per min, about half that observed with the phases inverted.

Aqueous Entrainment in Organic. The entrainment of aqueous in the organic effluent from the horizontal settler ranged from 1 to 100 ml per liter and averaged 10 ml per liter of organic. There was no definite correlation with any operating conditions.

Organic Entrainment in Aqueous Phase. The entrainment of organic in the aqueous phase as it was discharged from the settler was measured for settler lengths of 48 and 80 in. In the first four runs entrainment was high; moreover increasing the settler length from 48 to 80 in. did not reduce the amount of entrainment. Therefore, in subsequent experiments, samples of the aqueous phase were allowed an additional static settling time to show the effect of increased time upon entrainment. The tests have shown that, in general, between 30 and 60 min. settling time was required to obtain an entrainment of less than 0.2 ml of organic per liter of aqueous phase.

Future Work

The residual organic entrainment experienced with either a pump mixer or an orifice has been greater than desired. Therefore, a turbine mixer, in which the mixing intensity and the power input may be controlled, will be used to continue the studies of the effect of mixing variables on the amount of phase entrainment. In the first tests, emphasis will be placed on evaluating the effect of varying the power input while holding the flow rate and the impeller diameter constant.

3. SLURRY EXTRACTION

The exploratory study of a process for recovery of uranium from sulfuric acid leach slurries by solvent extraction (ORNL-1914) was continued with emphasis placed on testing the utility of a laboratory centrifugal pump as a mixer in a single-stage mixer-settler unit.

Preliminary calculations indicated that, with an organic entrainment in the slurry raffinate amounting to as much as 20 gallons per ton of ore, only about five mixer-settler units were needed to extract essentially all of the soluble uranium from leached slurry provided the extraction efficiency was near 100 percent. Because of greater differences in density, it appeared likely that lower organic entrainment would be obtained with leach slurries containing 50 to 60 wt % solids than with more dilute slurries (except for extreme dilution). Consequently, the tests were made using slurry direct from the leach tank.

A number of runs were made and the equipment revised in an attempt to improve the continuity of operations. In one run operation was satisfactory over a period long enough to achieve steady state. The data showed that the extraction efficiency was only 60 percent and the organic entrainment was equivalent to about 25 gallons per ton of ore. More complete details on this and subsequent runs will be reported when this phase of the exploratory studies has been completed.