ORNL-2212
Engineering cy. 4

PROGRESS REPORT OF PROCESS TEST SECTION

FOR JUNE, 1956

A. D. Ryon
K. O. Johnsson

OAK RIDGE NATIONAL LABORATORY
OPERATED BY
UNION CARBIDE NUCLEAR COMPANY
A Division of Union Carbide and Carbon Corporation

POST OFFICE BOX X · OAK RIDGE, TENNESSEE

Printed in USA. Price 15 cents. Available from the
Office of Technical Services
U. S. Department of Commerce
Washington 25, D. C.

ORNL-2212

Contract No. W-7405-eng-26

CHEMICAL TECHNOLOGY DIVISION

PROGRESS REPORT OF PROCESS TEST SECTION FOR JUNE, 1956

A. D. Ryon and K. O. Johnsson

Date Issued

DEC 11 1956

OAK RIDGE NATIONAL LABORATORY
Operated by
UNION CARBIDE NUCLEAR COMPANY
Post Office Box X
Oak Ridge, Tennessee

INTERNAL DISTRIBUTION

1. C. E. Center
2. Biology Library
3. Health Physics Library
4-5. Central Research Library
6. Reactor Experimental Engineering Library
7-26. Laboratory Records Department
27. Laboratory Records, ORNL R.C.
28. A. M. Weinberg
29. L. B. Emlet (K-25)
30. J. P. Murray (Y-12)
31. J. A. Swartout
32. E. H. Taylor
33. E. D. Shipley
34-35. F. L. Culler
36. M. L. Nelson
37. W. H. Jordan
38. C. P. Keim
39. J. H. Frye, Jr.
40. S. C. Lind
41. A. H. Snell
42. A. Hollaender
43. K. Z. Morgan
44. M. T. Kelley
45. T. A. Lincoln
46. R. S. Livingston
47. A. S. Householder
48. C. S. Harrill
49. C. E. Winters
50. D. W. Cardwell
51. E. M. King
52. W. K. Eister
53. F. R. Bruce
54. D. E. Ferguson
55. R. B. Lindauer
56. H. E. Goeller
57. D. D. Cowen
58. R. A. Charpie
59. J. A. Lane
60. M. J. Skinner
61. R. E. Blanco
62. G. E. Boyd
63. W. E. Unger
64. R. R. Dickison
65. A. T. Gresky
66. C. E. Guthrie
67. J. W. Ullmann
68. K. B. Brown
69. K. O. Johnsson
70. H. M. McLeod
71. J. C. Bresee
72. H. F. Bauman
73. N. C. Bradley
74. J. R. Buchanan
75. D. J. Crouse
76. R. S. Lowrie
77. R. H. Guymon
78. E. F. Joseph
79. B. B. Klima
80. A. D. Ryon
81. R. R. Wiethaup
82. E. D. Arnold
83. F. L. Daley
84. J. C. Chu (consultant)
85. M. E. Wadsworth (consultant)
86. M. Benedict (consultant)
87. D. L. Katz (consultant)
88. C. E. Larson (consultant)
89. G. T. Seaborg (consultant)
90. W. W. Weinrich (consultant)
91. M. D. Peterson (consultant)
92. ORNL - Y-12 Technical Library, Document Reference Section

EXTERNAL DISTRIBUTION

93. D. R. Miller, Atomic Energy Commission, Washington
94. G. G. Marvin, Atomic Energy Commission, Washington
95. R. B. Martin, Atomic Energy Commission, Oak Ridge
96. L. Squires, duPont Company, Wilmington
97. R. F. Bacher, California Institute of Technology
98. Division of Research and Development, AEC, ORO
99-674. Given distribution as shown in TID-4500 under Engineering category (200 copies - OTS)

CONTENTS

ABSTRACT

Leaching Tests: Roasted Ores. Samples of Marysvale and Temple Mountain ores were roasted to determine if this would improve the uranium leaching from these ores. Roasting the Marysvale ore resulted in a significant improvement in leaching efficiency but the uranium leaching of Temple Mountain ore did not appear to be affected under the conditions of the test.

Slurry Extraction. Studies were completed on the effect of some of the mixing variables on the extraction of uranium from 30%, 40%, and 50% Utex slurry by the Dapex process. Phase ratio, feed throughput, mixer speed, and turbine diameter were studied to determine their effect on both the extraction efficiency and the primary and final entrainment of the extractant phase in the raffinate stream.

NOTICE

The data presented in this report are preliminary, and are published in a formal report only to permit rapid dissemination of information to interested persons.

LEACHING TESTS: ROASTED ORE

J. R. Buchanan

Marysvale and Temple Mt. ores have been leach tested after roasting. The uranium leached from these ores in previous tests (ORNL-2112) was well below 95% of the total percent. Roasting should improve the leaching efficiency since any uranium tied up in organic or reduced mineral complexes would be oxidized to a readily leachable state.

Both ores were roasted at 500°C for 16 hours. The leaching conditions were identical to those used in the previous study.

The effect of the amount of sulfuric acid on the uranium and vanadium leaching is shown in Table 1. The leaching effi-

TABLE 1. EFFECT OF AMOUNT OF ACID ON LEACHING OF URANIUM AND VANADIUM FROM ROASTED ORE

Ore	H_2SO_4* (lb/ton)	Leached Residue Roasted Ore U_3O_8 (%)	Leached Residue Roasted Ore V_2O_5 (%)	Percent Leached Uranium Raw Ore**	Percent Leached Uranium Roasted Ore	Percent Leached Vanadium Raw Ore**	Percent Leached Vanadium Roasted Ore
Marysvale	100	0.019	0.066	76	89	-	20
	150	.013	.057	78	91	50	20
	200	.012	.055	83	92	42	20
	250	.010	.050	81	93	50	20
Temple Mt.	100	0.081	1.03	80	72	32	15
	150	.047	.89	82	83	32	15
	200	.045	.71	84	84	40	15
	250	.043	.71	85	85	44	15

*Conditions: 16 hours at 50-60°C.
**From ORNL-2112, p. 6.

ciency is based on the analyses of the ore samples and leached residues. A ten percent or greater increase in the uranium leaching of the Marysvale ore as a result of having been roasted was exhibited for each acid to ore ratio. This was sufficient to give acceptable leaching. Roasting of the Temple Mt. ore had no apparent effect on the uranium leaching.

As mentioned in the previous report the vanadium leaching was of secondary importance and was only included to give its order of magnitude. Less vanadium was leached from the roasted than similar raw ore. Typical leach liquor compositions are presented in Table 2.

No further leaching tests are planned.

TABLE 2. COMPOSITION OF LEACH LIQUORS

Concentrations in g/l unless noted

Ore	Marysvale	Temple Mt.
H_2SO_4 used, lb/ton	200	200
U	1.7	2.9
V	.1	2.6
Fe (total)	9.9	1.5
Fe^{+2}	.8	< .02
Al	8.8	6.6
Mo	.4	.002
SO_4	93	77
pH	1.7	1.4
Sp. Gr.	1.11	1.08

SLURRY EXTRACTION

F. L. Daley

Studies have been completed on the effect of some of the mixing variables on the extraction of uranium from 30%, 40%, and 50% Utex slurry by the Dapex process. Phase ratio, feed throughput, mixer speed, and turbine diameter were studied to determine their effect on both the extraction efficiency and the primary and final entrainment of the extractant phase in the raffinate stream.

The equipment consisted of a mixer-settler unit as was shown in Fig. 5, report ORNL-CF-56-8-30. Samples were taken after 4, 8, 10, 12 volume changes of the raffinate in the settler. Each of the primary entrainment results given is an average of eight determinations.

The final entrainment data was collected by diluting the slurry raffinate to 5 wt % with water, discarding the sands, and introducing the slime-slurry into a standard dilution cell, as described in ORNL-2035. The dilution cell results are an average of two determinations.

A summary of the feed analysis for the three different solid concentrations is given in Table 3.

TABLE 3. SUMMARY OF THE FEED ANALYSIS*

% Solids	pH	grams per liter							
		U	V	Mo	Al	SO_4	Fe	Fe^{++}	Si
50	1.4	2.3	1.6	0.024	2.8	33.0	3.9	3.1	0.53
40	1.5	1.7	1.3	.021	1.6	33.0	3.4	2.9	.40
30	1.5	1.1	.8	.015	1.6	26.0	3.0	2.6	.35

*Analysis of filtrate from slurry.

50% Utex Slurry

The data in Table 4 shows the effect of phase ratio on the organic entrainment in the raffinate stream. An increase in phase ratio (organic/aqueous) tends to decrease the primary entrainment and extraction efficiency, but has little effect on the final entrainment.

TABLE 4. EFFECT OF PHASE RATIO

Run No.	Mixer Speed (rpm)	Slurry feed (cc/min)	Phase Ratio O/A	Extraction Eff. (%)	Organic Entrainment (gal/ton of ore): Primary	After Dilution Cell**: 1 hr	After Dilution Cell**: 4 hr	After Dilution Cell**: 16 hr	After Centrifugation: 5%	After Centrifugation: 50%
43$_A^*$	700	200	2/1							
43$_B^*$	700	200	5/1							
42	700	200	10/1	99.8	195	10.0	6.2	2.5		
41	700	200	30/1	98.6	60	8.2	5.3	3.8		
43	700	200	60/1	91.2	30	3.7	3.0	2.2	1.6	

*Unable to run due to emulsion build-up in mixer.
**In the range of 0-3 gal/ton of ore the 95% confidence level is ±62.9% of the reported value. In the range of 3 - >3 gal/ton of ore the 95% confidence level is ±31.6% of the reported value.

It is noted from the data in Table 5 that an increase in the mixer speed from 700 rpm to 1500 rpm decreases the primary entrainment, but increases the final entrainment. At mixer speeds below 700 rpm the emulsion build-up in the mixer became so excessive that the runs were terminated.

TABLE 5. EFFECT OF MIXER SPEED

Run No.	Mixer Speed (rpm)	Slurry feed (cc/min)	Phase Ratio O/A	Extraction Eff. (%)	Organic Entrainment (gal/ton of ore) Primary	After Dilution Cell 1 hr	After Dilution Cell 4 hr	After Dilution Cell 16 hr	After Centrifugation 5%	After Centrifugation 50%
46A*	300	200	10/1							
46B*	300	200	30/1							
48	500	200	5/1							
42	700	200	10/1	99.8	195	10.0	6.0	2.5		
48	1500	200	5/1	98.7	119	30.0	15.9	5.0	2.7	19.3
47	1500	200	10/1	98.7	79	23.2	10.8	6.0	3.3	

*Unable to run due to emulsion build-up in mixer.

An increase in the slurry throughput, from 200 c/min to 1000 cc/min, decreased the primary entrainment but increased the final entrainment. At 1000 cc/min throughput the extraction efficiency fell to 77.8%. The data for these runs is found in Table 6.

TABLE 6. EFFECT OF SLURRY FEED THROUGHPUT

Run No.	Mixer Speed (rpm)	Slurry Feed (cc/min)	Phase Radio O/A	Extraction Eff. (%)	Organic Entrainment (gal/ton of ore) Primary	After Dilution Cell 1 hr	After Dilution Cell 4 hr	After Dilution Cell 16 hr	After Centrifugation 5	After Centrifugation 50%
42	700	200	10/1	99.8	195	10.0	6.2	2.5		
45	700	1000	10/1	77.8	91	7.9	4.3	4.2	1.03	

The mixer was equipped, in turn, with turbines of 2", 3", and 4" diameters to study the effect of turbine diameter on the organic entrainment. Table 7 gives the results of these runs. The 3" impeller at a power level of 0.031 hp/gal gave the lowest final entrainment and highest extraction efficiency. It was necessary to operate at a power level of 0.31 hp/gal in the cases of the 2" and 4" turbines before the emulsion build-up could be controlled enough to allow the run to be made.

40% Utex Slurry

Three runs were made on the 40% Utex slurry. The results of these runs followed the same pattern as that of the corresponding 50% slurry runs. Table 8 contains the data for these runs.

30% Utex Slurry

One run was made on the 30% Utex slurry. It was necessary to feed the slurry at 1000 cc/min due to fall-out of sands in the feed line to the mixer. The primary entrainment was higher but the final entrainment was slightly lower than for the corresponding 50% slurry run. Table 9 contains the data for this run.

Centrifugation

A series of samples were taken to study the effect of centrifugation on the recovery of the organic from the raffinate. Five samples were prepared, using the same procedure as used for the dilution cell, and rotated in a laboratory centrifuge for 10 min at 1400 rpm. The solids were collected and submitted for organic entrainment. In each case the centrifuged sample gave a lower organic content (in the order of one-half) than that of the corresponding sample run for 16 hours in the dilution cell.

TABLE 7. EFFECT OF TURBINE DIAMETER

Slurry Feed (200 cc/min)					Phase Ratio O:A = 10:1 Organic Entrainment (gal/ton of ore)					
						After Dilution Cell			After Centrifugation	
Run No.	Turbine Diameter	Mixer Speed (rpm)	Power Input (hp/gal)	Extraction Eff. (%)	Primary	1 hr	4 hr	16 hr	5%	50%
50A*	2"	1380	0.031							
50B	2"	2400	.31 **	95.1	99	29.0	12.7	7.4		
42	3"	700	.031	99.8	195	10.0	6.2	2.5		
47	3"	1500	.31	98.7	79	23.2	10.8	6.0	3.3	
49A*	4"	430	.031							
49B	4"	930	.31	93.8	87	20.0	9.2	4.1		12.6

*Unable to make run due to emulsion build-up in mixer.

**Mixer rpm of 2950 needed for 0.31 power level.

TABLE 8. 40% SLURRY RESULTS

Run No.	Mixer Speed (rpm)	Slurry Feed (cc/min)	Phase Ratio (o/a)	Extraction Eff. (%)	Organic Entrainment (gal/ton of ore)					
					Primary	After Dilution Cell			After Centrifugation	
						1 hr	4 hr	16 hr	5%	50%
52	700	200	10/1	99.4	123	6.0	3.0	2.2	1.8	
53	700	200	60/1	88.3	24	3.1	1.6	1.2		
54	500	200	10/1	Would not run						

TABLE 9. 30% SLURRY RESULTS

55	700	200	10/1	Slurry feed would not flow from tanks				
55	700	1000	10/1	98.4	128	2.8	2.7	2.3

Future Work

Considering the encouraging results obtained in the reduction of final entrainment by batch centrifugation it seemed that a continuous centrifuge might be employed for the final separation. Arrangements have been made with the Sharples Corporation of Philadelphia to test, in their laboratory, the recovery of organic from an aqueous raffinate in their "Nozljector" continuous centrifuge.

Plans are being made for a few preliminary tests utilizing a spray tower as the contactor for the extraction of uranium from slurries.