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TECHNOLOGY—FEED MATERIALS

Subject: Additional Pulse Column Tests
for Uranium Ores Recovery Process

To: F. L. Steahly

From: A. C. Jealous

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For The Atomic Energy Commission

H. R. Cansell
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1.0 Introduction

Catalytic Construction Company, architect-engineer for the Feed Materials Processing Center, requested* that two additional scrub tests be made in the ORNL Unit Operations pulse column, using a sample of uranium extract from the extraction column in the Mallinckrodt pilot plant. The purpose of the additional scrub tests was to determine the degree of uranium decontamination using conditions differing somewhat from the previous scrub runs. In the latter runs, the organic phase was dispersed, whereas previously the aqueous phase was dispersed. The column length was 25-feet, or about three times the length used in the first scrub runs. The previous scrub and strip tests were reported in ORNL CF# 51-4-155.

2.0 Summary

Two additional scrub pulse column tests for the Slurrex Process, made in a 25 ft. x 2 in. I.D. test column, showed that the conditions selected were satisfactorily operable in that no symptoms of flooding were observed at any time during either run. The effectiveness of the scrubbing operations, with respect to the removal of uranium contaminants, was somewhat inconclusive, since the organic extract supplied for the tests contained uranium of an exceptionally high degree of purity.

* Letter, April 18, 1951, to N. Woodruff, AEC, Oak Ridge, from F. M. Belmore, Director, Production Division, AEC, New York; EPD:NFS:bt; ORNL Particulation in the New Refinery Research and Development Program.

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3.0 Scrub Tests and Results

Temperature was the sole variant in the two scrub runs: one run was made at 20°C and the other at 50°C. The aqueous phase was continuous for both runs. The column was 25 feet long, and except for additional plates, was otherwise the same as the 8 ft. column previously described in ORNL CF# 51-4-155.

Pulsing conditions for both runs were 0.7 in. amplitude and 50 strokes per minute frequency. Total throughput rate was 800 gal./sq. ft.-hr. in each case. Duration of the runs was five hours for the first and 6.5 for the second.

3.1 Operational Results

Both runs were operationally satisfactory in that, at no time, was there any evidence of flooding or near-flooding. Material balances indicate that for the run at 20°C, about 8% of the feed uranium was carried off in the aqueous effluent; and for the heated run (50°C), about 14% of the feed uranium was carried by the scrub aqueous (see Table I). Some minor operating difficulties (such as pumping an aqueous layer from the feed drums into the column) disturbed the column equilibrium somewhat, particularly during the second run.

From the uranium and acid analyses, it appears that column equilibrium was reached in the first scrub run (MF-3) in about 2 - 3 hours. However, in the second run, dynamic equilibrium may not have been reached. Although the acid analysis indicate that the maximum possible acid concentration (determined by material balance) in the aqueous stream was attained at the end of the run, on the other hand, the exit uranium values are quite low compared with values indicated for a 100% material balance (see Table II).

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3.2 Uranium Decontamination

Spectrographic analysis (see Table III) of run samples submitted to the AEC New Brunswick Laboratory indicate that the feed sample from Mallinckrodt contained insufficient impurities to permit clear evaluation of contaminant removal in either of the two scrub tests. This suggests that use of the scrubbing step in the Slurrex Process may not always be necessary on a routine basis, and that its use may be governed by the type of ore being processed.

4.0 Comparison of the Pulse Column Scrub Runs with a Batch Countercurrent Run

A comparison of pulse column runs MF-3 with a laboratory batch countercurrent run at room temperature indicates that the 25-foot column was equivalent to about 5 batch countercurrent stages, based on the uranium and acid concentrations in the aqueous raffinate (recycle) stream. The data from both of the column runs, and also the batch countercurrent run, indicate that an appreciable build-up of uranium occurs in the middle region of either contactor (see Table IV). The latter effect results from the change in distribution coefficient due, among other things, to the presence of some nitric acid.

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Table I

Scrub Tests in Pulse Column for Slurrex Process

Pulse column: plate section: 25 ft. x 1.916 in. I.D. true-bore glass pipe

Plates: 18 ga. stainless steel; 0.125 in. holes, 23% free area

Plate spacing: 2 in.

Pulse amplitude: 0.7 in.

Pulse frequency: 50 strokes/minute

Aqueous phase continuous

Flow ratio (O/A): 13:1

Total throughput rate: 800 gal/sq. ft.-hr.

Feed solution: organic sample from St. Louis pilot plant: 22.5% TBP -
77.5% Shell Dispersol, ~75 g U/l

<u>Run</u>	<u>Temperature</u>	<u>Uranium (as % of feed) in Aqueous Scrub Effluent</u>
MF-3	~20	~8
MF-4	~50	~14

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Table II

Uranium and Acid Analyses of Stream Samples from Pulse Column Scrub Runs

Run	Sample	Feed		Product		Raffinate	
		U g/l	H ⁺ N	U g/l	H ⁺ N	U g/l	H ⁺ N
MF-3*	0.5 hr.	75	0.1	48	0.2	61	0.4
	1.0			60	0.01	77	0.7
	1.5			66	"	81	0.9
	2.0			63	"	84	1.2
	2.5			73	0.04	86	1.4
	3.0			73	0.05	84	1.6
	3.5			70	0.03	82	1.8
	4.0			-	0.04	79	1.9
	4.5			70	0.04	81	1.9
	5.0			70	0.03	83	2.0
MF-4	0.5	76	0.1	52	0.01	97	1.0
	1.0			50	"	125	0.8
	1.5			60	"	87	0.7
	2.0			56	"	90	1.0
	2.5			55	"	106	1.0
	3.0	77	0.14	54	"	120	1.0
	3.5			55	"	121	1.2
	4.0			57	"	126	1.2
	4.5			56	"	136	1.2
	5.0			63	"	146	1.0
	5.5			60	"	140	1.3
	6.0			65	"	129	1.6
	6.5			61	"	126	1.7

* All HNO₃ values for MF-3 (except for Feed Organic) are considered slightly high. Material balance based on ~ 0.1 N HNO₃ in Feed Organic would not permit over 1.7 N HNO₃ in the Raffinate.

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Table III
(Condensed)

Spectrographic Analysis of Feed and Effluent Samples

from Scrub Runs

(Reported as ppm on a Uranium basis)

Element	Run MF-3			Run MF-4		
	Feed	Organic Effluent	Aqueous Raffinate	Feed	Organic Effluent	Aqueous Raffinate
Ag.	0.2	0.1	0.1	1	0.1	0.1
Al	10	10	20	10	10	10
B	0.8	1.2	0.8	1.1	1.8	0.6
Co	-	-	200	-	-	5
Cu	1	1	100	1	1	1
Fe	5	5	200	5	5	10
Mg	5	5	200	5	5	8
Mn	-	-	5	-	-	-
Mo	-	-	100	-	-	10
Ni	-	-	200	-	-	20
P	50	50	50	50	50	-
Pb	1	1	50	1	1	10
Si	30	20	25	75	100	20
Sn	-	-	5	-	-	-
As	-	-	-	-	-	-

NOTE: indicates "none detected".

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Table IV
Uranium and Acid Stream Concentrations for Pulse Column
and Batch Countercurrent Scrub Runs

1. Batch Countercurrent Run

	<u>Aqueous</u>		<u>Organic</u>	
	<u>U</u> <u>g/l</u>	<u>H⁺</u> <u>N</u>	<u>U</u> <u>g/l</u>	<u>H⁺</u> <u>N</u>
Feed	-	-	77	0.03
Stage 1	117	0.05	81	0.03
" 3	153	0.25	81	0.03
" 5	74	1.78	71	0.01

2. Pulse Column Runs

<u>Run</u>	<u>Aqueous</u>		<u>Organic</u>	
	<u>U</u> <u>g/l</u>	<u>H⁺</u> <u>N</u>	<u>U</u> <u>g/l</u>	<u>H⁺</u> <u>N</u>
MF-3 Feed	-	-	75	0.1
"Mid-point"*	112	-		
Effluents	83	1.8	70	0.03
MF-4 Feed	-	-	76	0.1
"Mid-point"*	168	-		
Effluents	130	1.5	62	0.01

* "Mid-point" sample was an aqueous sample drawn from the column 4 ft. above the bottom end of the column.

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