UNCLASSIFIED

ORNL
CENTRAL FILES NUMBER

Date: April 26, 1951

Subject: Uranium Ores Recovery Pulse Column Tests

CF-51-4-155

To: F. L. Steahly

From: A. C. Jealous

Before reading, please sign and date below:

CLASSIFICATION CANCELLED
DATE FEB 15 1957 *ml*
For The Atomic Energy Commission
H. R. Canale
Chief, Declassification Branch

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

Price \$ 0.25

Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

2065 1

UNCLASSIFIED

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

UNCLASSIFIED

~~CONFIDENTIAL~~

~~SECRET~~

-2-

April 26, 1951

1.0 Introduction

The scrub and strip operations of the Slurrex Process for the recovery of uranium from ore were briefly tested in a 2 in. I.D true-bore glass pulse column 8 ft. high, using a feed solution received from the Mallinckrodt pilot plant and using conditions agreed upon with the Catalytic Construction Company, architect-engineer for the new ore refinery. The tests were conducted by the ORNL Unit Operations solvent extraction group.

The purpose of the pulse column tests was to check flooding rates and column operability using specified flowsheet conditions. In addition, the effect of temperature elevation on (1) flooding, (2) amount of uranium transferred (as percent uranium stripped), and, in the case of the scrub column only, (3) the concentration of molybdenum in the scrub aqueous raffinate was observed. These tests were limited to one set of pulse column conditions for reasons of available time and test material.

The feed material for the pulse column tests consisted of 150 gallons of organic effluent from the Mallinckrodt pilot plant extraction column. The effluent was 30% TBP - 70% Gulf BT containing ~ 80 g U/l. Prior to use, this solution was diluted to 13.5% TBP with additional Gulf BT, to provide sufficient material for two scrub and six strip tests.

~~CAUTION~~

This document contains information affecting the National Defense of the United States. Its transmission or the disclosure of its contents in any manner to an unauthorized person is prohibited and may result in severe criminal penalties under applicable Federal laws.

~~SECRET~~

CLASSIFICATION CANCELLED

DATE FEB 15 1957 *ml*

For The Atomic Energy Commission

H. F. Canale

Chief, Declassification Branch

~~RESTRICTED DATA~~

This document contains restricted data as defined in the Atomic Energy Act of 1946

UNCLASSIFIED

2025

~~CONFIDENTIAL~~

~~SECRET~~

-3-

April 26, 1951

All analytical results reported were provided by the ORNL Analytical Chemistry Division. Effluent samples from the two scrub runs were shipped to New Brunswick for complete analysis to determine the history of molybdenum and other contaminants carried in these streams.

2.0 Summary

The pulse column tests of the scrub and strip operations of the Slurrer Process indicated that this type of contactor is operable at total flow rates of greater than 1200 gallons per square foot per hour. Operation at elevated temperatures ($\sim 50^{\circ}\text{C}$) increased the separation of molybdenum from uranium in scrubbing by a factor of three, and decreased the uranium strip loss. The flooding rate of the strip column was increased by 30% by operation with a continuous organic phase.

3.0 Equipment

The test pulse column for the uranium ore processing scrub and strip tests consisted of an 8 ft. length of 1.916" I.D. true-bore glass pipe (see picture). Forty-five perforated stainless steel plates with 0.125 in. holes and 23% free area were spaced 2 in. apart on a 0.25 in. supporting rod, and the cartridge was installed in the glass column. This is the "standard" cartridge presently used in the ORNL pilot plant.

CAUTION

This document contains information affecting the National Defense of the United States. Its transmission or the disclosure of its contents in any manner to an unauthorized person is prohibited and may result in severe criminal penalties under applicable Federal laws.

~~RESTRICTED DATA~~

This document contains restricted data as defined in the Atomic Energy Act of 1946.

~~SECRET~~

DECLASSIFIED

2005 3

~~CONFIDENTIAL~~

~~SECRET~~
PHOTO 7987

FIG. 1
PULSE COLUMN WITH 8ft x 1.916in. I.D.
TRUE-BORE GLASS SECTION CONTAINING
47 STAINLESS STEEL PERFORATED PLATES,
USED FOR SCRUBBING AND STRIPPING TESTS

~~CONFIDENTIAL~~

~~SECRET~~

-4-

April 26, 1951

Two Milton Roy Constametric (straight-line flow) pumps were used: one to feed the uranium-bearing organic solution, and one to remove the aqueous raffinate and control the interface level. A modified Milton Roy simplex pump was used for pulsing the liquid contents of the column. Calibrated sight glasses on the feed tanks served as burets to check feed rates; 4 ft. sections of 3" glass pipe served for effluent flow rate checks.

A heat exchanger was provided on the organic feed line, and a steam coil was installed in the water feed tank. Instrument control (Wheelco indicator-controllers and solenoid steam valves) was used to heat feed streams such that temperature of the column contents would be raised to about 60°C maximum.

4.0 Scrub Tests

The scrub investigation (see Table I) indicated that the combined effect of increasing the solution temperature from 20° to about 60°C and increasing the flow ratio (organic:aqueous) from 10 to 20 decreased the flooding rate from greater than 2500 to less than 1800 gal./ft.²-hr. These changes in conditions had little effect on the percent of uranium transferred from the organic phase; however, the concentration of molybdenum in the aqueous scrub effluent was increased from 10 to 26 ppm of uranium in this effluent.

The flooding results, particularly of the second run, are somewhat doubtful. In this run, the column operated smoothly for 3.5 hours at which time it flooded perhaps due to a decrease in temperature. By increasing the pulse amplitude the

~~CONFIDENTIAL~~

~~SECRET~~

~~CONFIDENTIAL~~

2005

5

~~CONFIDENTIAL~~

~~SECRET~~

-5-

April 26, 1951

flooding condition was eradicated, and it is felt that the run would have been generally more satisfactory had it been run at an amplitude higher than 0.5 in.

Table I

Scrub Tests in Pulse Column Slurrex Process

Pulse column: plate section: 8 ft. x 1.916 in. I.D. true-bore glass pipe

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

Plate spacing: 2 in.

Pulse amplitude: 0.5 in.

Pulse frequency: 70 strokes/minute

Organic phase continuous

Feed solution: organic sample from St. Louis pilot plant which was diluted with additional Gulf BT to 13.5% TBP (dielectric constant method), 37 g U/l and 0.20 N HNO₃

Run	Temp.	Flow Ratio O/A	Total Flowrate gal/ft ² -hr.	Run Duration Hrs.	Aqueous Effluent U as % of feed	Scrub Mo conc. ppm U	Flooding Rate gal/ft ² -hr
MF-1	20	10	1750	4.6	10	10	2500*
MF-2	60	20	1350	3.5**	7	26	1800

* Limiting rate of pumps

** Flooded after 3.5 hours; this was perhaps due to large temperature change in building since run started at 4 PM and finished (flooded) 1.5 hours after sundown. A short test in which the amplitude was increased to 0.9 in. eradicated the flooding condition.

~~CONFIDENTIAL~~

~~SECRET~~

DECLASSIFIED

2005

6

~~CONFIDENTIAL~~

~~SECRET~~

-6-

April 26, 1951

5.0 Strip Tests

The strip investigation (see Table II) indicated that changing from a continuous aqueous to a continuous organic phase increased the flooding rate from 2400 to 3000 gal./ft.²-hr. at 20°C. With the organic phase continuous, the flooding rate was not affected by elevating the temperature to 50°C and simultaneously increasing the organic to aqueous flow ratio from 1 to 1.67. However, under roughly similar conditions with the aqueous phase continuous the flooding rate was decreased from 2400 to 1200; material was not available for recheck.

The uranium strip loss was decreased by elevating the temperature with either aqueous or organic phase continuous, in spite of using / ^{higher} organic to aqueous flow ratios at the higher temperature. It also appears that continuous organic phase reduces uranium loss slightly; however, it is not known how much the loss is affected by the considerably higher throughput rates used in the run with the continuous organic phase.

Teflon perforated plates, used in one run with the organic phase continuous and a column temperature of about 50°, did not give significantly different flooding or stripping results as compared with the use of stainless steel plates. (See Run CC-5 Table II).

~~CONFIDENTIAL~~

~~SECRET~~

DECLASSIFIED

2005 7

~~CONFIDENTIAL~~

~~SECRET~~

-7-

April 26, 1951

Table II

Strip Tests in Pulse Column Slurrex Process

Pulse column: plate section: 8 ft. x 1.916 in. I.D. true-bore glass pipe
Plates: 18 ga. stainless steel except as noted; 0.125 in. holes, 23% free area
Plate spacing: 2 in.
Pulse amplitude: 0.5 in.
Pulse frequency: 70 strokes/min.
Feed solution: organic product solution from scrub column tests; analyzed ~33 g U/l
and 0.1 - 0.14 N HNO₃

Run No.	Continuous Phase	Temp °C	Flow Ratio O/A	Total Flowrate gal/ft ² -hr	Run Duration hrs.	% U Loss	Flooding Rate gal/ft ² -hr
CC-1	Aqueous	20	1	640	1	25	2400
CC-2	Aqueous	60	2	950	1	12	1200
CC-2	Organic	20	1	2200	0.5	19	3000
CC-6	Organic	50	1.67	2150	0.7	12	3000
CC-5*	Organic	50	1.67	2150	0.7	16	2900

* Teflon plates substituted for stainless steel; 1/16th in. thick, otherwise same dimensions as stainless plates.

~~CONFIDENTIAL~~

~~SECRET~~

DECLASSIFIED

2005 8

~~CONFIDENTIAL~~

~~SECRET~~

-8-

April 26, 1951

6.0 Appendix

Table III

Uranium and Acid Analyses

Run	Sample	Feed		Product		Raffinate	
		U g/l	H ⁺ N	U g/l	H ⁺ N	U g/l	H ⁺ N
MF-1	30 Minute	37.9	0.15	36.9	1.0	30.0	0.05
	45 "			35.0	1.0	31.5	0.08
	60 "			31.1	1.2	31.5	0.08
	75 "			32.9	1.3		
	90 "			32.4	1.4	31.5	0.10
	105 "			32.4	1.4		
	120 "			31.8	1.4	31.5	0.10
	135 "			32.1	1.4		
	150 "			32.3	1.4	32.9	0.08
	275 "			31.5	1.5	34.0	0.08
MF-2	15 "			36.8	1.1		
	30 "			41.5	1.3	34.6	0.14
	45 "			44.7	1.5		
	60 "			45.0	1.6	34.6	0.16
	75 "			50.4	1.6		
	90 "	36.9	0.17	51.7	1.6	34.6	0.14
	105 "			47.1	1.6		
	120 "			49.4	1.7	35.0	0.11
	135 "	36.9	0.20	47.4	1.6		
	150 "			53.3	1.6	35.8	0.14
	165 "	37.0	0.22	52.7	1.7		
	180 "			51.0	1.7	35.0	0.11
	195 "			51.8	1.7		
	210 "			54.0	1.7	35.0	0.14
CC-1	25 "	30.3		23.2	0.14	7.7	0.01
	35 "			23.4	0.15	7.5	"
	45 "			24.1	0.15	7.8	"
	55 "			24.7	0.14	7.4	"
	60 "			24.0	0.14	7.5	"
CC-2	20 "	33.4		25.0	0.14	6.5	0.01
	25 "			26.0	0.13	6.3	0.01
	30 "			26.8	0.13	6.0	"
	35 "			27.3	0.14	6.1	"

~~SECRET~~
DECLASSIFIED

~~CONFIDENTIAL~~

~~SECRET~~

10
-9-

April 26, 1951

Table III (continued)

Run	Sample	Feed		Product		Raffinate	
		U g/l	H ⁺ N	U g/l	H ⁺ N	U g/l	H ⁺ N
CC-3	30 Minutes	34.5	0.15	64.7	0.3	4.3	0.00
	40 "			62.5	0.3	3.9	"
	50 "			62.0	0.3	3.7	"
	60 "			62.7	0.3	"	"
CC-6	20 "	33.0	0.08	43.5	0.13	4.2	0.00
	30 "			42.1	0.09	4.0	"
	35 "			41.3	0.09	3.9	"
	40 "			44.1	0.09	4.2	"
CC-5	20 "	36.0	0.07	45.0	-	4.7	0.01
	30 "			46.5	0.14	5.5	0.00
	35 "			46.0	0.14	5.4	-
	40 "			44.5	0.15	5.4	0.02

~~CONFIDENTIAL~~

~~SECRET~~

DECLASSIFIED

2065 10

Table IV

SPECTROGRAPHIC LABORATORY
706-A
REPORT OF ANALYSIS

VS = very strong
S = strong
M = moderate
W = weak
VW = Very weak

T = trace
FT = faint trace
- = sought not found
= not sought
Numerals = microgm./ml.

Film No. 3245
Date 3/25/51
For A. C. Jealous

Material	MF - 1	MF - 1	MF - 1	MF - 2	MF - 2	MF - 2
AL	FT	FT	FT	FT	FT	FT
B	VFT	VFT	VFT	VFT	VFT	VFT
Ca	T	T	T	T	T	T
Cr	FT	FT	FT	VFT	VFT	VFT
Cu	FT	FT	FT	FT	FT	FT
Fe	VW	VW	VW	FT	FT	FT
La	-	-	-	-	-	-
Mg	T	T	T	T	T	T
Mn	FT	FT	FT	VFT	VFT	VFT
Mo	0.34	0.30	0.30	1.37	1.35	1.71
Na	-	-	-	-	-	-
Ni	-	-	-	-	-	-
Pb	-	-	-	-	-	-
Sc	VFT	VFT	VFT	VFT	VFT	VFT
Si	T	T	T	FT	FT	FT
Zr	FT	FT	FT	FT	T	FT