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PROJECT 9536

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For The Atomic Energy Commission
H F Carroll
Chief, Declassification Branch

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Date 3-8-45

Subject Production Tests on Lanthanum Fluoride Precipitations No. 224T- 3-8

To File

From W. C. Kay

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L. Squires
J. E. Willard

HW. 9-67

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L Squires	3/21/45
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W C Johnson	3/29/45
J H Peterson	3/30/45
S G Thompson	4/3/45
H F [illegible]	4/3/45
[illegible]	[illegible]
J L Drake	4/4/45
O F Hill	4/4/45
H T Baker	4-4-45
Angerman	4/6/45
W F Schmidt	4/7/45
E H Turk	4/7/45
B E Evans	4/7/45

W A Sullivan	4/10/45
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G R Roeder	4/11/45
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INV. 9-47

MARCH 8, 1945

PRODUCTION TESTS ON LANTHANUM FLUORIDE PRECIPITATIONS
NO. 224T-3-5

Objective

To decrease LaF_3 product precipitation losses (E-3-45 wastes).

Basis

The present standard procedure for the LaF_3 cycle in Bldg. 224 is as follows:

(a) LaF_3 by-product - two 125 mg/liter preformed La strikes in 0.2N HF with centrifugations at approx. 17000 and 5 minutes retention time, followed by a 500 lb. 6% HNO_3 wash split into 2 centrifuge batches;

(b) LaF_3 product - 35°C reduction with 0.04M $H_2C_2O_4$ and $0.004Mn^{++}$ (from $KMnO_4$ oxidation in Cell 3), followed by two 50 mg/liter preformed La strikes in 0.5N HF with centrifugations at approx. 17000 and 5 minutes retention time, and a 500 lb. 6% HNO_3 wash split into 2 centrifuge batches.

The LaF_3 product precipitation wastes (E-3-45) from this process have almost consistently exceeded the standard loss of 1.5%, ranging up to 7% in several cases. Many wastes have had to be reworked by recentrifugation and the addition of extra La carrier. Several test modifications of the product strike procedure have been tried, such as:

(a) The use of 1.0N HF
(b) Elimination of the product cake HNO_3 wash
(c) 8 minutes centrifuge retention time (Clinton practice) instead of 5 minutes in the second centrifugation
(d) slow second strike (40 minutes) instead of the usual 20 minute strike
(e) A quiescent flocculation period of 2 hours in the second strike,
(f) The use of steam condensate instead of sanitary water throughout the entire procedure. A summary of these process changes and their effects is given in Table I.

Since none of these changes has reduced product losses appreciably, a temporary expedient of making 3 strikes with the same total La (2.5 lbs.) is being used at present. While this procedure has been keeping product losses down to a lower level than anticipated for the standard process, losses are still excessive and the time cycle is naturally extremely long.

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Extensive sampling and analysis throughout the LaF_3 cycle have made it appear that a small amount of LaF_3 fines leaking through the centrifuge is responsible for the product losses, rather than incomplete reduction or carrying of product. Additional evidence indicates that a leak-through of fines from the LaF_3 by-product centrifugation, most probably LaF_3, picks up 25-75% of the product during reduction and before the first regular product strike. In an effort to reduce the LaF_3 fines production or carry-over, certain test procedures are being proposed for the March series of runs in Bldg. 224-T.

TABLE I

SUMMARY OF LaF_3 PRODUCT PRECIPITATION PROCESS CONDITIONS AND WASTE LOSSES

Run No.	HF N	La Strikes: No. Strks	La Strikes: La Mg/l.	La Strikes: Strike Time, Min	Cent. Rate Lbs/min	Spec. Condit.	K-398 Loss %	Recent of K-398: Procedure	Recent of K-398: % Loss
T-4-12-B-5	0.5	2	50	20	110	None	3.3	None	-
" B-6	0.5	2	50	20	110	"	0.6	"	-
" B-6B	0.5	2	50	20	110	"	4.9	25% extra La	2.0
" B-6D	0.5	2	50	20	110	"	2.1	50% extra La 1 N HF 25% extra $H_2C_2O_4$	0.4
" B-7	1.0	2	50	20	110	"	1.5	Recent.	0.4
T-5-1-B-1	1.0	2	50	20	110	No KNO_3 wash	3.7	"	2.2
" B-2	1.0	2	50	(1) 20 (2) 40	110	No KNO_3 wash	3.0	None	-
" KC	1.0	2	50	20	(1) 110 (2) 70	None	2.9	"	-
" B-3	1.0	2	50	20	(1) 110 70	"	4.9	Recent (70 lbs/min.)	2.2
T-4-12-B-6C	1.0	2	50	20	110	"	3.7	Recent	2.6
T-5-1-BA-1	0.5	2	50	20	110	"	7.5	100% extra La	0.6
T-5-2-B-1	0.5	2	50	20	110	"	6.9	Recent	4.5
" B-2	0.5	3	33	13	110	"	0.4	None	-
" B-3	0.5	3	33	13	110	"	3.0	"	-
" B-4	0.5	3	33	13	110	Steam cond.	2.6	"	-
" B-5	0.5	3	33	13	110	Steam cond.	3.5	"	-
" B-6	0.5	3	33	13	110	Steam cond.	3.6	"	-
" BB-1	0.5	3	33	13	110	Steam cond.	5.1	100% extra La	-

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Procedure

It is proposed that the test procedures listed in Table II be carried out in the March series of runs in Bldg. 22[illegible]-T. Summarized briefly the main variable changes and the objectives of each change are given below:

Test No.	Run Nos.	Main Variable Change	Objectives
1	T-5-3-[illegible]-2 T-5-3-8-1	Prod. La strike #1 m[illegible] before retention	Flocculation of LaF_3 before product pick-[illegible]
2	T-5-3-[illegible]-2 T-5-3-8-3	Prod. La strikes #2 and #3 made before return of effluent to precipitator	Flocculation of fines from first centrif[illegible]tion
3	T-5-3-8-4 T-5-3-8-5	Eliminate HNO_3 wash in by-product or make 0.5N HF by-product	Decrease fines carry-over from by-product
4	T-5-3-[illegible]-4	All LaF_3 centrifugations at 8 minutes retention time (70 lbs./min.)	Complete check of Clinton centrifugation conditions

It is also proposed that if any one pair of test runs produces low B-3-[illegible] losses at least one more run shall be made with the same procedure to establish its reproducibility more definitely. Test No. 3 will be chosen from the two alternatives quoted as governed by whether or not Test Nos. 1 and 2 show that the HNO_3 wash of the by-product cake contributes LaF_3 fines to the B-3-[illegible].

TABLE II
PROPOSED TEST SCHEDULE FOR LaF_3 PRECIPITATIONS

		Cell D						Cell E							
									La Strikes				Cent.		
Test No.	Run No.	HF N	No. La Strikes	La Mg/l	Cent. Rate lbs/min	HNO_3 Wash	Special analyses	HF N	#1	#2	#3	Mg/l Str.	#	lbs/ Min.	Special analyses
1	T-5-2-H-2 T-5-3-B-1	0.2	2	125	110	Yes	Lab. red'n & cent. of D-3-OS before & after HNO_3 wash	0.5	Be- fore red'n	Reg.	Reg.	33	3	110	E-1-PS & E-3-MS samples for AY and ent. cent. in lab
2	T-5-3-B-2 T-5-3-B-3	0.2	2	125	110	Yes	"	0.5	Reg.	To E-1 head bef. E-3 ret.	To E-1 head bef. E-3 ret.	33	3	110	"
3	T-5-3-B-4	(a) 0.2	2	125	110	No	(a) Same for reg. D-3-OS	0.5	Reg.	Reg.	Reg.	33	3	110	"
	T-5-3-B-5	(b) 0.5	2	125	110	Yes	(b) Same as 1 & 2								
4	T-5-3-B-6	0.2	2	125	70	Yes	Same as 1 & 2	0.5	Reg.	Reg.	Reg.	33	3	70	"

NOTE: It is also proposed that any beneficial effects in the early parts of the cycle as found from the beginning of the test series be carried along as cumulative process changes, although not shown in Table II.

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Data

The Technical Department Plant Assistance Group III will assist Operations in collecting data and monitoring process conditions. Assistance may be requested from the Process Research Section in carrying out laboratory centrifugations of process samples immediately when obtained.

Equipment

No additional equipment required.

Responsibility

Technical Department Plant Assistance:

M.F. Acken - Area Supervisor
R.H. Beaton - Senior Supervisor, Group III

Estimated Completion

These tests will cover the span of the March runs in Bldg. 224-T.

Approval

[illegible] Date 3/[illegible]/45
Operating Department

L. Squires Date 3/8/45
Technical Department

B H Mackey Date 3/10
Assistant Manager

RKB/ss

[redacted]