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Date 4-7-47

Subject Fred. Test SE-224-T-PI-4

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Continous Fluoride Precipitation

TID-III
PL 503
PL Eick 1-3-48

*General
 Mary
 6/10/47*

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From K. H. Beaton

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P. W. Crane

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APRIL 5, 1945

INV. 13-47

PRODUCTION TEST SB-224-T-PA-4
LANTHANUM FLUORIDE PRECIPITATION

Objective

To shorten LaF₃ by-product and product operations in order to attain practical time cycles of 24 hours or less in Cells D and E.

Basis

A LaF₃ By-Product: The present LaF₃ by-product precipitation process involves two 125 mg./l. preformed La strikes in 0.2N HF, centrifuged each time at 1700 G and 5 minutes retention time (110 lbs./min.). The "ideal" time cycle is approximately 23 hours. Stepwise decontamination factors (Bookman meter observations) are 5.0 and 3.0 for the first and second strikes respectively, averaging an overall of 15 for the cycle.

Recycling of 800 mg./l. of La to the first strike from the isolation supernatants has increased the first strike decontamination factor to approximately 10, even when centrifuged at 110 lbs./min. A single strike of 800 mg./l. of La as crystalline LaF₃ in 0.6N HF has produced a decontamination factor of 20, when centrifuged at 70 lbs./min.

Since the present two-strike LaF₃ by-product process has a time cycle too close to 24 hours to be practical, it is proposed that a single-strike process be tested. With present volumes, an ideal single-strike cycle with slow centrifugation would be about 17 hours. The background data presented above indicate that a decrease of not more than 2 in decontamination factor would be expected with flocculent LaF₃ and none for crystalline LaF₃. The reduced process volumes being proposed for reduction of the LaF₃ product cycle time would further reduce the ideal by-product cycle to approximately 15 hours.

B LaF₃ Product: The background of the LaF₃ product precipitation and the development of the procedure necessary to maintain the product waste losses (B-3-WB) below the standard of 1.5% have been traced in Production Test 224-T-3-8 and in Memorandum to M.F.Acken by R.H.Beaton on March 29. It is shown therein that it is necessary to make either two 50 mg./l. or three 33 mg./l. La product strikes, followed in each case by three 70 lbs./min. centrifugations (8 minutes retention time) at 1700 G, to maintain low B-3-WB losses. The time cycle for this process is 38-40 hours.

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PRODUCTION TEST SE-224-T-PA-4 (Cont'd) -2-

4-8-48

A preliminary plant test (Run T-5-05-B-6) of the crystalline LaF₃ process being developed by the Process Chemistry and Semi-Works Sections of the 200 Area Technical Group has produced very promising results. A single strike of 100 mg./l. La, centrifuged at 70 lbs./min., left only 4.8% product in the effluent, which was in turn reduced to 0.75% and 0.15% respectively by two additional 50 mg./l. strikes of the regular flocculent LaF₃.

In order to approach the desired practical cycle time of 24 hours for the LaF₃ product precipitation, it is proposed that the Concentration Building process solution volumes be reduced. It has been calculated that the volume of the 17-4-P solution can be reduced to 45% of the present by a series of reductions in Bi concentrations and HNO₃/Bi cake solution ratios. The three-centrifugation process would still take slightly more than 24 hours, however, so it is recommended that Cell B be fitted out for fluoride precipitations. Parallel product strikes on two separate charges at about 15% of the time will make possible a practical 24 hour cycle. Cell B should also be made versatile enough to handle fluoride by-product precipitations as a stand-by for Cell D. It is also proposed that plant investigation of the crystalline LaF₃ be continued with the reduced process volumes, with the objective of developing a still shorter time cycle to permit Cell B to be a stand-by fluoride cell at all times.

Procedure

It is proposed that the test procedures listed in Table I be carried out in the LaF₃ cycle (Cells D and E) of the Concentration Building during the April series of runs in T-Area. This series of tests is aimed at the development of shorter LaF₃ product and by-product cycles without affecting either product losses or decontamination factors appreciably.

The LaF₃ by-product tests are set up as follows:

<u>Test No.</u>	<u>Run Numbers</u>	<u>Main Variable Change</u>	<u>Objectives and Predictions</u>
1.	T-5-4-B-1 T-5-4-B-2	One 200mg./l. crystalline La strike in .5N HF, cent. at 70 lbs./min. Usual process volumes.	Check runs on T-5-3-B-6 performance, and prep. for crystalline product strikes. No decrease in D.F. expected.
2.	T-5-4-B-3 T-5-4-B-4	One 580 mg./l.* flocculent La strike in 45% of usual volume, cent. at 70 lbs./min.	Efficiency of single, high-concentration strike with regular LaF ₃ . Decrease in D.F. of no more than 2 expected.
3.	T-5-4-B-5 T-5-4-B-6	One 470 mg./l.* crystalline La strike in 45% of usual volume, cent. at 70 lbs./min.	Direct comparison of crystalline with flocculent LaF ₃ (test #2), and prep. for crystalline product strikes.
4.	T-5-4-B-7 T-5-4-B-8	One 235 mg./l. crystalline La strike in 45% of usual volume, cent. at 70 lbs./min.	Preparation for crystalline product strikes and reduction of by-product La.
5.	T-5-4-B-9	Same as Test #2, #3, or #4.	Start of reproducibility tests if choice of process can be made at this time.

* La concentration based on use of same total weight of La in 45% of usual volume, to be obtained by gradual volume reductions throughout the Canyon Building (281).

PRODUCTION TEST SE-224-T-PA-4 (Cont'd) -4-

4-8-45

The LaF_3 product precipitation tests involve comparative studies of the crystalline and flocculent types of LaF_3 , particularly in the reduced volumes to be obtained from the Canyon Building (221) which in themselves induce lower time cycles. The LaF_3 product tests are set up as follows:

<u>Test No.</u>	<u>Run Number</u>	<u>Main Variable Changes</u>	<u>Objectives</u>
1.	T-5-4-B-1	Two 50 mg./l. <u>crystalline</u> La strikes. 3 centrifugations at 70 lbs./min.	2-strike, 2-cent. Cell B process. Third centrifugation for safety.
2.	T-5-4-B-2	One 50 mg./l. <u>crystalline</u> and one 50 mg./l. <u>flocculent</u> La strike; 3 cent. at 70 lbs./min.	Simpler process if first crystalline strike lesser low enough for clean-up of <u>flocculent</u> LaF_3 .
3.	T-5-4-B-3 T-5-4-B-4	Two 115 mg./l. <u>flocculent</u> La strikes in 45% of usual volume; 3 cent. at 70 lbs./min.	2-strike process with regular LaF_3 with low enough volumes to permit slow centrifuging.
4.	T-5-4-B-5	Two 115 mg./l. <u>crystalline</u> La strikes in 45% of usual volume; 3 cent. at 70 lbs./min.	Same as Test #3 - comparison of crystalline vs. flocculent LaF_3 .
5.	T-5-4-B-6	One 115 mg./l. <u>crystalline</u> La and one 115 mg./l. <u>strike</u> in 45% of usual volume; 3 cent. at 70 lbs./min.	Same as Test #2, in reduced strike volumes.
6.	T-5-4-B-7 T-5-4-B-8	One 230 mg./l. <u>crystalline</u> La strike in 45% volumes; 2 cent. at 70 lbs./min.	Single-strike, single cent. process if test progress promising.
7.	T-5-4-B-9	To be decided from previous tests.	Choice of process, if possible.

- * La concentrations based on use of same total weight of La in 45% of usual volume, to be obtained by gradual volume reduction throughout the Canyon Building (221).

Data

The Technical Department Plant Assistance Group III will assist Operations in collecting data and setting up and monitoring process conditions.

Equipment

Since the $\text{Na}_2\text{SO}_4 \cdot \text{La}_2(\text{SO}_4)_3$ slurries for the crystalline LaF_3 tests are to be made up by the Semi-Works Section in the 300 Area, no equipment for its manufacture in Building 224-T is needed. A dolly tank and rubber hose jumper connection will be needed for adding the slurries from the control gallery. Both are available. Simple on-the-spot line changes in the pipe gallery are also needed for introduction of the slurries.

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PRODIGE TEST 9B-224-T-PA-4 (Cont'd)

4-8-46

Proposed Test Schedule Lab's Precipitations

Table I

Test No.	HF	No.	Lab's Product				Test No.	HF	No.	Lab's Product				Remarks
			La. Type	Co. No.	Rate	Notes				La. Type	Co. No.	Rate	Notes	
7-5-4-B-1	1	0.5	1	200	Cryst. 1	70	1	0.5	2	50	Cryst. 3	70	Std. Process Volumes	
7-5-4-B-1	1	0.5	1	200	Cryst. 1	70	2	0.5	2	50	Cryst. 3	70		
7-5-4-B-1	1	0.2	1	500	Place. 1	70	3	0.5	2	115	Place. 3	70	45% of usual Std. volumes	
7-5-4-B-1	1	0.5	1	470	Cryst. 1	70	4	0.5	2	0.115	Cryst. 3	70		
7-5-4-B-1	1	0.5	1	285	Cryst. 1	70	5	0.5	2	115	(1) Cryst. 3 (2) Place. 3	70	45% of usual Std. volumes	
7-5-4-B-1	1	0.5	1	285	Cryst. 1	70	6	0.5	1	250	Cryst. 3	70		
To be decided later													45% of usual Std. volumes	
To be decided later														

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PRODUCTION TEST SE-224-T-PA-4 (Cont'd) -6-

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Responsibility

Technical Department Plant Assistance: H.F. Adken - Area Supervisor
R.H. Benton - Senior Supervisor, Group III
The experimental runs will be carried out under the usual S-Department supervision.

Estimated Completion

These tests will cover the April series of runs in T-Area and will be completed by June 1, 1945.

Approval

<u>M. C. Ray</u>	Operating Department	Date <u>4/7/45</u>
<u>L. Dyer</u>	Technical Department	Date <u>4/6/45</u>
<u>B. J. Mackay</u>	Assistant Manager	Date <u>4/7/45</u>

KHB:jd

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