

product losses. Since the present cycle involves three centrifugations utilizing 6% of the process time, considerable time can be saved by modifying the centrifugation standards. A reduction of the process volume and an increase in reagent addition rate also shorten the cycle.

Two general schemes appear to be feasible: (1) A reduction in process volumes coupled with an increase in centrifugation rate for the three precipitation processes, and (2) elimination of the third precipitation and centrifugation at present standard feed rates. Process volume reduction and the increased reagent addition rate may be applicable to both schemes if waste losses are not increased excessively.

Since both general schemes are expected to increase waste losses this test is designed to achieve the desired cycle with a minimum loss increase. It is possible that losses may be further minimized by increasing the hydrofluoric acid concentration. Process volume reduction also tends to lower product losses.

The bases for the elements of these schemes are given below:

Increased concentration of oxalic acid reagent

The solubility of oxalic acid is approximately 70 g per 100 ml water (41%) at 65° C (Handbook of Chemistry and Physics, 23rd Edition, p. 920). Therefore, a concentration of 25% and a weight reduction to 475 lbs. is feasible for the process reagent. The solubility of oxalic acid is 33 g per 100 ml water (25%) at approximately 35° C. Present standards require 1230 lbs. of 9.6% oxalic acid to be added for the lanthanum fluoride product reduction. In preparation the solution is heated to 65° C. A solution 26.4% in oxalic acid was made up in E-1-C scale tank at T Plant. All the acid was in solution at 65° C. Some crystallized out at 20° C. A possible slight decrease in waste loss is anticipated as the result of the decrease in volume.

Increased concentration of lanthanum salt reagent

A 10% solution of lanthanum salt is successfully used for the lanthanum fluoride by-product precipitation. Present standards require 1000 lbs. of a 1.5% lanthanum salt solution for the lanthanum fluoride product precipitation. This dilute solution is used in order to maintain a uniform addition rate. Comparable uniformity can be obtained with the more concentrated solution by the use of the proper type discharge valve from the scale tank. A possible slight decrease in waste losses is expected to result from this change due to the decrease in volume if there are no adverse effects from the increased reagent concentration.

More rapid addition rate of lanthanum

At Clinton Laboratories lanthanum for the lanthanum fluoride product precipitation was added at 3% per minute. Good separation was obtained (CN-2010, The Flocculation of Lanthanum Fluoride, p. 10, from J. B. Sutton to File). The rate of 2% per minute has been used at the Hanford

Concentration Buildings. A slight waste loss improvement was thought to result from a slower rate and 1% per minute was adopted as standard. It is proposed, therefore, to return to the rate of 2% per minute.

Processing runs at higher nitric acid concentration

When additional acid is added for the cake removal in the second cycle product precipitation, no correction is made in the Concentration Building until 400 additional pounds of 60% nitric acid have been added. Consequently some runs are processed through the Concentration Building at higher nitric acid concentration. Eleven runs have been thus processed so far. The results are given in Table I.

These data indicate that waste losses may increase with nitric acid concentration. However, runs processed to date have shown no direct correlation with the nitric acid concentration.

Increased rate of centrifugation

Present standards require a centrifuge feed rate of 60 lbs. per minute. Feed rate for the lanthanum fluoride product precipitation have been maintained at 110 lbs. per minute and 70 lbs. per minute in the past. Feed rates, estimated product loss, and time savings are listed below for three precipitations and three centrifugations.

Feed Rate	Estimated % LaF ₃ Product Loss	Minutes Saved
60	0.11	
70	0.18	69
80	0.26	115
90	0.37	150
(Based on present volumes and concentrations)		

Elimination of third centrifugation

The effluent of the second centrifugation under present standards contains approximately 0.49% of the product of the run. If the volume were reduced 10% and all lanthanum used in two centrifugations the estimated product loss would be 0.40%. This would produce a time cycle approximating the cycle with present volumes and concentrations and three centrifugations at 90 lbs. per minute.

Increased concentration of hydrofluoric acid

Tests were run at Clinton Laboratories in 1944 to determine whether hydrofluoric acid normality for the lanthanum fluoride product precipi-

tation could be lowered from 1.0 to 0.5N. The product in the effluent was approximately twice as great for 0.5N as for 1.0N hydrofluoric acid solutions. (CN-2010, The Flocculation of Lanthanum Fluoride, p. 10-12, from J. B. Sutton to File.) The standard of 0.5N was established at that time due to corrosion rate and failure of 1.0N hydrofluoric acid to yield a one centrifugation process. Corrosion tests at Clinton in 1944 showed a corrosion rate of 0.1 mils to 0.9 mils per month could be expected with 1.0N hydrofluoric acid on 25-12 stainless steel. Concentrations of 0.5N showed a rate of 0.05 to 0.1 mils per month (CN-1628, Corrosion Tests, F. S. Apple to File). This process change will result in no time savings, but may counteract increases in waste losses due to adoption of shorter cycle.

Procedure

Procedures which result in waste losses from 0.50 to 0.70% on initial runs will be tested on only 5 runs. Procedures which result in waste losses greater than 0.70 on initial runs will be tested on only two runs.

Waste losses from 0.20 to 0.30 will be reworked by an additional centrifugation. Waste losses from 0.31 to 0.70 will be reworked by an extra precipitation with 15% of standard lanthanum and repeated centrifugation. Waste losses greater than 0.70% will be reworked by a method to be determined by Plant Assistance.

Procedures of items 1 through 5 which give an average waste loss greater than 0.25% will not be used in later tests. Procedures of items 6 through 7 which yield waste losses greater than 0.50 will not be used in later testing.

The following order of testing procedures will be used:

1. Ten runs will be processed at 1.0N hydrofluoric acid concentration. Waste samples (E-3 and B-3-WS) will be analyzed for iron content.
2. Ten standard runs will be processed. Waste samples will be analyzed for iron content.
3. Ten runs will be processed using 25% oxalic acid reagent concentration.
4. Ten runs will be processed using the procedure of item 3. In addition, the lanthanum salt will be added at 1% per minute.
5. Ten runs will be processed using the procedure of item 4. In addition, 10% lanthanum salt reagent concentration will be used.
6. Ten runs will be processed using the procedure of item 5. In addition, a centrifugation rate (80 to 90 lbs. per minute) necessary to yield a 9 hour cycle will be employed.

7. Ten standard runs will be processed for control purposes.
8. Ten runs will be processed using the procedure of item 5. In addition, (a) 1.0N nitric acid concentration will be obtained for the lanthanum fluoride by-product precipitation by adding less dilution water in the bismuth phosphate cross over step and (b) a centrifugation rate (70 to 80 lbs. per minute) necessary to yield a 9 hour cycle will be employed.
9. Ten runs will be processed using the procedure of item 5. In addition, (a) a 1.1N nitric acid concentration for the lanthanum fluoride product precipitation will be obtained by adding less dilution water in the bismuth phosphate cross over step, and (b) the centrifugation rate (65 to 75 lbs. per minute) necessary to yield a nine hour cycle will be employed.
10. Ten standard runs will be processed for control purposes. The effluent of the second centrifugation will be assayed for product.
11. Ten runs will be processed using the procedure of item 5. In addition, only two precipitations and centrifugations will be made. One-half of the lanthanum will be used for each precipitation. Centrifugation rate will be standard.
12. Depending on plant performance of runs in item 1 and on the results of corrosion tests with 1.0N hydrofluoric acid, ten runs will be processed at 1.0N hydrofluoric acid concentration. In addition, the procedure of item 6, 8, 9, or 11 which gave the lowest average waste loss will be employed.

Data

Routine product assays of lanthanum fluoride product wastes will be observed. Iron analyses of waste solution from runs using 1.0N hydrofluoric acid concentration and from 10 standard runs will be required. If 1.0N hydrofluoric acid shows a product saving, corrosion tests on 25-12 stainless steel will be required. Product assay of the effluent of the second centrifugation will be required on 10 standard runs.

Equipment

Valves to permit a uniform addition rate of 3 lbs. per minute through a one inch line from the E-1-C and B-1-A scale tanks will be installed.

Responsibility

Responsibility for making the test runs will rest with the "S" Division. Test procedures will be prepared and data assembled by B. E. Kirkendall. Conclusions will be drawn jointly by "S" and Technical Divisions.

Estimated Completion

The estimated date of completion for the test is November 1, 1950.

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200 AREAS PROGRAM COMMITTEE

K.C.V.T. "S" Division 6/11/50 Date
J. Z. ... Separation Technology Division 6/17/50 Date

200 AREAS STEERING COMMITTEE

[Signature] "S" Division 6/15/50 Date
R.W. ... Separation Technology Division 6/16/50 Date

MANAGEMENT

[Signature] Mgr., Production Division 6/11/50 Date

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APPENDIX

TABLE I--Effect of Nitric Acid Concentration

Run Number	Nitric Acid Normality		% Waste Loss					
	BiPO ₄ Cross	LaF ₃ Prod.	BiPO ₄ Cross	LaF ₃ By-Prod.	LaF ₃ E-3	Prod B-3	Meta-thesis	Final IIF
B-10-03-B-22	1.66	1.10	0.08	0.04	--	0.22	0.06	7.25
B-10-04-F-04	2.55	1.03	.09	.05	--	.14	.03	7.21
F-16	1.66	1.14	.09	.04	--	.20	.05	7.60
B-22	1.60	1.07	.12	.03	--	.21	.03	7.33
B-26	1.60	1.11	.05	.03	--	.29	.04	7.47
B-28	1.61	1.07	.03	.06	--	.15	.04	7.25
B-31	1.76	1.07	.13	.03	0.19	--	.05	7.41
B-34	1.62	1.16	.09	.04	--	.18	.04	7.45
Typical (10 runs)	1.4	0.9	.09	.05	.14	.15	.06	7.48
T-10-03-B-16	1.60	1.03	0.08	0.06	--	0.14	0.09	7.51
F-31	1.80	1.12	.10	.05	0.12	--	.07	7.52
F-27	1.73	1.08	.08	.07	.03	--	.04	7.43
Typical (10 runs)	1.4	0.9	.07	.07	.03	.13	.07	7.42

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