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FINAL REPORT

PRODUCTION TEST 224-T-14

LANTHANUM FLUORIDE PRODUCT PRECIPITATION TIME CYCLE REDUCTION

SECRET
SECURITY INFORMATION

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I. INTRODUCTION

The time required to process runs through the lanthanum fluoride product precipitation in the Concentration Building is 23 hours. Since two cells (B and E) are used for this operation, the effective time cycle is 11.5 hours. The projected production schedule requires a time cycle of nine hours or less. It was believed that the desired reduction in time cycle could be effected by (1) modifying the centrifugation procedure which specified three passes at a controlled rate that accounted for 65 per cent of the process time, (2) reducing the process volume to result in shorter total centrifugation times, and (3) increasing reagent addition rates. Production Test 224-T-14, Document HW-18080, defined the conditions for investigating these possibilities. The results obtained from this investigation are documented in this report.

II. OBJECTIVE

The objective of the production test was to evaluate procedures designed to reduce the lanthanum fluoride product precipitation effective time cycle to nine hours or less with a minimum increase in product losses.

III. SUMMARY AND CONCLUSIONS

A total of 93 runs, divided into ten separate phases to evaluate individual variables and combinations of conditions, were made in the 224-T Building to establish and demonstrate the feasibility of reducing the time cycle. A time cycle of nine hours was achieved with an increase in product loss of 0.05 per cent of an average run by reducing process volumes, through increased reagent concentration and smaller dilution factors in the bismuth phosphate by-product step, increased reagent addition rates and faster centrifugation through-put. Under the test conditions the process volume during the lanthanum fluoride product precipitation was reduced to result in an increase in nitric acid normality to 1.1 normal from approximately 0.9 normal. This was accomplished by increasing the oxalic acid concentration to 25 per cent from 9.4 per cent, the lanthanum ammonium nitrate concentration to 10 per cent from 1.5 per cent and by decreasing the dilution in the bismuth phosphate by-product precipitation. The lanthanum addition rate was increased to 2 per cent per minute from 1 per cent per minute and the centrifugation rate was increased to 70 pounds per minute from 60 pounds per minute.

A seven hour cycle was demonstrated with an increase in product loss of 0.24 per cent of a run. This was achieved with two passes through the centrifuge at a rate of 60 pounds per minute utilizing the advantages of the volume reduction determined under the nine hour cycle process.

Increasing the hydrofluoric acid concentration in the lanthanum fluoride product precipitation to 1.0 normal from 0.5 normal did not significantly lower the product loss.

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On the basis of the time cycle requirements at the conclusion of the test it was recommended that a ten hour cycle, based upon two cells in parallel operation, and utilizing a nitric acid concentration of 1.1 normal for the lanthanum fluoride product precipitation, 25 per cent oxalic acid and 10 per cent lanthanum salt as reagent chemicals, and a lanthanum salt addition rate of 2 per cent per minute be made standard. It was further recommended that any future time cycle reduction be accomplished by modifying the centrifugation rate or eliminating the third pass through the centrifuge as required to meet a specific cycle.

IV. EXPERIMENTAL

Two general schemes appeared to be feasible: (1) reduction in process volumes coupled with an increase in centrifugation rate for the three precipitation process, and (2) elimination of the third precipitation and centrifugation at present standard feed rates. Process volume reduction and increased reagent addition rate may be applicable to both schemes if waste losses are not increased excessively. Since both general schemes were expected to increase waste losses this test was designed to achieve the desired cycle with a minimum loss increase. It was believed that losses might be further minimized by increasing the hydrofluoric acid concentration. Process volume reduction would also tend to lower product losses.

The bases for the elements of the test are given below:

Increased concentration of oxalic acid reagent - Present standards require 1230 pounds of 9.6 per cent oxalic acid to be added for the lanthanum fluoride product reduction. In preparation the solution is heated to 65°C. The solubility of oxalic acid is approximately 41 per cent at 65°C and 25 per cent at 35°C. Therefore, a concentration of 25 per cent and a weight reduction to 475 pounds is feasible for this process reagent.

Increased concentration of lanthanum salt reagent - Present standards require 1000 pounds of 1.5 per cent lanthanum salt solution to be added 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 a more concentrated solution by the use of the proper type discharge valve on the scale tank. A 10 per cent solution of lanthanum salt is successfully used for the lanthanum fluoride by-product precipitation.

More rapid addition rate of lanthanum - Present standards require that the lanthanum salt reagent be added at 1 per cent per minute. A rate of 2 per cent per minute had previously been used for the lanthanum fluoride product precipitation. At Clinton Laboratories the lanthanum was added at 3 per cent per minute and good separation was obtained (CN-2010).

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 per cent nitric acid have been added. On the basis of eleven runs processed in this manner, the data indicate no direct correlation of waste losses with nitric acid concentration. Present standards require a nitric acid concentration of 0.9 normal for the lanthanum fluoride product precipitation.

Increased rate of centrifugation - Present standards require a centrifuge feed rate of 60 pounds per minute. Feed rates of 110 and 70 pounds per minute have formerly been maintained with increased waste losses.

Elimination of third centrifugation - The effluent of the second centrifugation under present standards contains approximately 0.49 per cent of the product of the run. If the volume were reduced 10 per cent and all the lanthanum used in two precipitations and centrifugations the estimated product loss would be 0.40 per cent. A time cycle approximating the cycle with present volume and concentrations and three centrifugations at 90 pounds per minute would result.

Increased concentration of hydrofluoric acid - Present standards require a hydrofluoric acid concentration of 0.5 normal. Tests at Clinton Laboratories to determine whether the hydrofluoric acid normality could be lowered from 1.0 to 0.5 normal, indicated that the product in the effluent was approximately twice as great for the 0.5 normal condition. The standard of 0.5 normal was established at that time due to the corrosion rate and failure of the 1.0 normal hydrofluoric acid concentration to yield a one centrifugation process. (CN-2010).

The following procedures were used during the production test:

Phase 1. A series of ten runs were processed with a hydrofluoric acid concentration of 1.0 normal in the lanthanum fluoride product precipitation.

Phase 2. A series of ten standard control runs were processed.

Phase 3. A series of ten runs were processed with an oxalic acid concentration of 25 per cent.

Phase 4. A series of ten runs were processed using 25 per cent oxalic acid and the lanthanum salt reagent added at 2 per cent per minute.

Phase 5. A series of ten runs were processed using 25 per cent oxalic acid, lanthanum salt added at 2 per cent per minute and with a lanthanum salt reagent concentration of 10 per cent.

Phase 6. A series of twelve runs were processed using 25 per cent oxalic acid, 10 per cent lanthanum salt added at 2 per cent per minute, and a centrifugation rate of 80 pounds per minute.

Phase 7. A series of six standard control runs were processed.

Phase 8. A series of ten runs were processed using 25 per cent oxalic acid, 10 per cent lanthanum salt added at 2 per cent per minute, with a nitric acid concentration of 1.1 normal and a centrifugation rate of 70 pounds per minute.

Phase 9. A series of ten standard control runs were processed.

Phase 10. A series of five runs were processed using 25 per cent oxalic acid, 10 per cent lanthanum salt added at 2 per cent per minute and in addition only two precipitations and two centrifugations. One-half of the lanthanum was used for each precipitation. Centrifugation rate was standard at 60 pounds per minute.

V. DISCUSSION

The first phase of the production test involved testing an increase in the hydrofluoric acid concentration for the lanthanum fluoride product precipitation from 0.5 to 1.0 normal. An indicated increase in corrosion of about 70 per cent resulted with a reduction in waste loss of 0.02 per cent of an average run.

A nine hour effective time cycle was demonstrated during phase eight of the test. Phase eight included testing of 25 per cent oxalic acid concentration, 10 per cent lanthanum salt concentration, lanthanum salt addition rate of 2 per cent per minute, nitric acid concentration of 1.1 normal and centrifugation rate of 70 pounds per minute. The lanthanum fluoride product waste loss was 0.17 per cent compared to 0.12 per cent of an average run for the standard process.

An effective time cycle of seven hours was obtained during phase ten of the test through elimination of the third precipitation and centrifugation. The waste loss averaged 0.36 per cent of an average run. The test data are summarized in Table I. Complete production test data are presented in Tables II through XI.

The time cycle requirement for the lanthanum fluoride product cell upon completion of the test was ten hours for two cells in parallel operation. On the basis of test results the process that best fitted a ten hour cycle and yielded the lowest waste loss was chosen. This process consisted of 25 per cent oxalic acid concentration, 10 per cent lanthanum salt concentration, lanthanum salt addition rate of 2 per cent per minute and a nitric acid concentration of 1.1 normal for precipitation and three centrifugations at the standard rate of 60 pounds per minute. The average

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lanthanum fluoride product waste loss using this procedure was 0.16 per cent of an average run for 20 runs, T-10-10-F-43 through D-56, B-58 through T-10-11-B-4.

VI. FUTURE WORK

Subsequent to the completion of this series of tests it has become evident that a further reduction in the time cycle would be desirable. Therefore, a series of production tests with even broader limits has been instituted under Production Test 221-T-16, "Four Hour Time Cycle In The Canyon and Concentration Building", in Document HW-24716.

Table I - Summary of Test Results - Production Test 224-T-14

Phase	I	II	III	IV	V	VI	VII	VIII	IX	X
Number of Runs	10	10	10	10	10	12	6	10	10	5
Average % La Product Waste Loss	0.10	0.11	0.09	0.11	0.14	0.21	0.12	0.17	0.12	0.36
Time Cycle Hours	11.5	11.5	11.2	10.5	10.5	9.0	11.4	9.0	11.6	7.0

Note: The phase numbers used in the table are defined in the experimental section of this report.

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Table II - Production Test 224-T-14 - Phase I - Hydrofluoric Acid 1.0 N

Run Number	B-3WS Per Cent	E-3WS Per Cent	Iron g/l	Isolation Recycle Per Cent	P-1 Normality
T-10-06-D-22	0.13		0.13	3.1	2.01
T-10-06-D-23		0.10	0.14	6.7	2.02
T-10-06-D-24	0.09		0.12	4.7	2.40
T-10-06-D-25		0.07	0.15	15.1	1.68
T-10-06-D-26	0.07		0.13	11.3	3.00
T-10-06-D-27		0.13	0.12	4.8	2.36
T-10-06-D-28	0.11		0.08	5.8	1.87
T-10-06-D-29		0.10	0.12	4.7	2.07
T-10-06-D-30	0.11		0.11	6.0	1.92
T-10-06-D-31		0.07	0.10	5.7	2.54
Average	0.10	0.09	0.12	6.8	2.19
Average T-10-01-B-23 thru F-36 (10 standard runs)	0.18	0.12	0.07	4.1	2.17

Table III - Production Test 224-T-14 - Phase II - Standard Control Runs

Run Number	B-3WS Per Cent	E-3WS Per Cent	Iron g/l	Isolation Recycle Per Cent	P-1 Normality
T-10-06-D-32	0.18		0.48	9.7	2.40
T-10-06-B-33		0.13	0.10	3.9	2.07
T-10-06-B-34	0.09		0.10	4.4	1.95
T-10-06-B-35		0.06	0.05	5.9	2.91
T-10-07-B-1	0.06		0.04	5.1	2.15
T-10-07-D-2		0.10	0.05	8.0	2.27
T-10-07-D-3	0.15		0.05	[illegible]	[illegible]
T-10-07-D-4		0.08	0.14	4.8	1.92
T-10-07-D-5	0.06		0.06	6.8	2.03
T-10-07-[illegible]6		0.15	0.06	4.8	1.93
Average	0.11	0.10	0.11	5.8	2.18

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Table IV - Production Test 224-T-14 Phase III - Oxalic Acid Concentration 25 Per Cent

Run Number	B-3WS Per Cent	E-3WS Per Cent	Time Cycle Hours
T-10-07-D-7	0.10		12.0
T-10-07-D-8		0.10	11.4
T-10-07-D-9	0.10		11.7
T-10-07-D-10		0.07	11.2
T-10-07-D-11	0.10		16.2
T-10-07-D-12		0.07	12.9
T-10-07-F-13	0.09		12.6
T-10-07-F-14		0.09	12.0
T-10-07-F-15	0.07		12.3
T-10-07-F-16		0.07	12.4
Average	0.09	0.08	12.5

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Table V - Production Test 224-T-14 Phase IV - Oxalic Acid Concentration - 25 Per Cent Lanthanum Addition - 2 Per Cent Per Minute

Run Number	B-3WS Per Cent	E-3WS Per Cent	Time Cycle Hours
T-10-07-F-17	0.09		11.4
T-10-07-AW-1		0.08	
T-10-07-F-18	0.09		13.7
T-10-07-F-19		0.15	12.8
T-10-07-F-20	0.15		12.9
T-10-07-F-21		0.08	11.4
T-10-07-F-22	0.14		12.1
T-10-07-F-23		0.11	11.8
T-10-07-F-24	0.10		10.5
T-10-07-F-25		0.12	11.6
T-10-07-F-26	0.11		11.4
Average	0.11	0.11	12.0

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Table VI - Production Test 224-T-14 Phase V - Oxalic Acid Concentration - 25 Per Cent, Lanthanum Salt Concentration - 10 Per Cent, Lanthanum Addition 2 Per Cent per Minute.

Run Number	B-3WS Per Cent	E-3WS Per Cent	Time Cycle Hours
T-10-07-F-27		0.14	10.8
T-10-07-F-28	0.15		10.7
T-10-07-D-29		0.19	10.5
T-10-07-D-30	0.08		11.0
T-10-07-D-31		0.13	10.7
T-10-07-D-32	0.10		10.6
T-10-07-D-33		0.14	11.0
T-10-07-D-34	0.[illegible]		10.8
T-10-07-D-35		0.16	11.4
T-10-07-D-36	0.11		10.8
Average	0.13	0.15	10.8

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Table VII - Production Test 224-T-14 Phase VI - Oxalic Acid Concentration - 25 Per Cent, Lanthanum Salt Concentration - 10 Per Cent, Lanthanum Addition Rate - 2 Per Cent Per Minute, Centrifugation Rate - 80 Pounds Per Minute

Run Number	B-3WS Per Cent	E-3WS Per Cent	Time Cycle Hours
T-10-07-D-37		0.13	9.1
T-10-07-D-38	0.22		9.0
T-10-07-D-39		0.15	9.4
T-10-07-D-40	0.27		9.7
T-10-07-D-41		0.19	9.8
T-10-07-F-42	0.14		9.8
T-10-07-F-43		0.16	9.3
T-10-07-F-44	0.23		9.2
T-10-07-F-45		0.17	9.2
T-10-07-F-46	0.26		9.2
T-10-08-B-3	0.34		
T-10-08-B-4		0.21	
Average	0.24	0.17	9.4

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Table VIII - Production Test 224-T-14 Phase VII - Standard Control Runs

Run Number	B-3WS Per Cent	E-3WS Per Cent	Time Cycle Hours
T-10-07-F-47		0.11	11.8
T-10-07-F-48	0.14		11.4
T-10-07-F-49		0.07	11.7
T-10-07-B-50	0.16		12.8
T-10-08-B-1	0.13		13.2
T-10-08-B-2		0.12	12.2
Average	0.14	0.10	12.2

Table IX - Production Test 224-T-14 Phase VIII - Nitric Acid Concentration - 1.1 N, Oxalic acid concentration - 25 Per Cent, Lanthanum Concentration - 10 Per Cent, Lanthanum Addition Rate - 2 Per Cent Per Minute, Centrifugation Rate - 70 Pounds Per Minute

Run Number	B-3WS Per Cent	E-3WS Per Cent	Time Cycle Hours	A-4BP Per Cent	D-4BP Per Cent
T-10-08-B-5	0.23		9.0	0.14	0.06
T-10-08-B-6		0.18	9.5	0.09	0.05
T-10-08-B-7	0.24		9.3	0.08	0,06
T-10-08-F-8		0.17	9.4	0.10	0.07
T-10-08-F-9	0.18		9.1	0.14	0.07
T-10-08-F-10		0.13	9.6	0.11	0.07
T-10-08-F-11	0.19		9.5	0.18	0.07
T-10-08-F-12		0.11	10.0	0.11	0.08
T-10-08-F-13	0.15		9.0	0.09	0.08
T-10-08-F-14		0.12	9.9	0.06	0.08
Average	0.2	0.14	9.4	0.11	0.07

Table X - Production Test 224-T-14 Phase IX - Standard Control Runs

Run Number	B-3WS Per Cent	E-3WS Per Cent	B-3W Per Cent	E-3W Per Cent	Time Cycle Hours
T-10-03-F-15	0.12		0.92		12.2
T-10-08-F-16		0.09		0.59	12.4
T-10-08-F-17	0.09		0.50		11.6
T-10-08-F-18		0.10		0.47	13.1
T-10-08-F-19	0.16		0.64		12.6
T-10-08-F-20		0.15		0.93	13.0
T-10-03-B-21	0.08		0.55		13.3
T-10-08-B-22		0.14		0.54	13.0
T-10-08-B-23	0.14		0.89		12.1
T-10-08-B-24		0.11		0.66	12.2
Average	0.12	0.12	0.70	0.64	12.5

Table XI - Production Test 224-T-14 Phase X - Phase V Procedure
Two Precipitations, Two Centrifugations.

Run Number	B-3WS Per Cent	E-3WS Per Cent	Time Cycle Hours
T-10-08-H-25	0.44		7.5
T-10-08-H-26		0.40	8.6
T-10-08-H-27	0.37		7.0
T-10-08-H-28		0.30	8.5
T-10-08-H-29	0.28		7.0
Average	0.36	0.35	7.7

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