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UNITED STATES ATOMIC ENERGY COMMISSION

[PITCHBLENDE PROCESSING DEVELOPMENT] PRELIMINARY TESTS ON OLIVER FILTER, BATCHES S-104 INCLUSIVE

By
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[PITCHBLENDE PROCESSING DEVELOPMENT]
PRELIMINARY TESTS ON OLIVER FILTER, BATCHES S–104 INCLUSIVE

By C. F. Ritchie

INTRODUCTION

The writer's experience with the filtration of the residues from the digestion of AAA ores began about the time that the so-called No. 3 process was inaugurated in the pilot plant. This consisted of adding nitric acid to the ore slurry, followed by the addition of barium nitrate solution, then sufficient sulfuric acid to precipitate (theoretically) the added barium and the lead content of the ore. (Note: In Batch S the barium was added as $BaCO_3$ with the ore slurry.)

At the time, the experimental Oliver filter was being employed for separating the solids from the digestion liquor, and this machine was employed in all of the tests described in this memo. Later, the filtration work was transferred to the experimental Feinc filter (see subsequent memo on that work.)

All work reported in this memo was done on ore lot (P-29961-LA.)

OUTLINE OF TESTS

The subject matter of this report may be divided into three phases, viz:

1. Filtration tests of sludge at low temperatures (140-160°F)— necessitated by limitations of the then available filtration media. (Tests S, T, U.)

2. Filtration tests at higher temperatures (190-210°F)—using high temperature, acid resisting fabrics. (Tests V-Z incl.)

3. Filtration tests on an altered residue, also at high temperatures. The change of character of the residue was brought about by altering the processing steps, so that the ore was added to the acid, instead of the opposite procedure.

DISCUSSION

1. Low Temperature Filtration

At the time, the only suitable acid-resisting filter medium available was the common variety of Vinyon filter cloth, which cannot be used at temperatures above about 160°F—over that temperature, partial plasticity and profound shrinkage sets in.

In reporting these early tests, facts pertaining to the nature of the cakes, their acceptance of wash water, etc., are of greater interest than quantitative data.

The outstanding characteristic of the filter cakes produced in runs S, T, and U was the "softness" of said cakes. Attempts to apply wash water to the filter resulted in pronounced channeling or erosion of the cake—a slight excess of water easily sluiced the cake from the drum. Somewhat better results were obtained when "drag cloths" were installed to spread the water, but these smeared the cake badly, and did not stop the erosion entirely.

For the benefit of the record, it should be stated here that these sludges or residues were by no means unfilterable. On the contrary, cakes of $1/8$ to $3/16$ inch thickness were formed in about two minutes of submergence at 20″ – 24″ vacuum, and in one instance, at least, (Batch U) the projected time for the filtration was well within the time allowable; namely, 200 minutes.

The cakes were, however, almost unwashable (within times allowable) for the reasons already noted. This fact was obvious from a visual inspection of the cake being discharged from the machine. From $1/2$ to $3/4$ of the depth of the cake was still strongly colored by the entrained liquor, even when maximum quantities of wash water had been applied.

2. High Temperature Filtration

Between August 3 and August 6, 1945, several changes were made to the Oliver filter setup. A group of six mail-volume sprays (0.07 gpm each) were mounted over the filter drum on long flexible copper tubes.

These were placed so that their distances from the cake could be easily and quickly changed, and also so that their relative peripheral spacing could be altered. Each was fitted with a separate cock,—any number could be employed.

Also, there was obtained at this time a new Vinyon fabric, known as the "N" grade, capable of withstanding not only the acids involved, but also boiling temperatures. In tests, V, W & X, a very heavy tightly woven fabric called Vinyon 101N was used to dress the filter.

Means were installed to keep the contents of the filter bowl at 190–210°F, and run V performed. The resulting cake showed considerable improvement over the previous cakes formed at lower-temperature. Either by virtue of the improved cake or the new sprays (or both), the cake appeared to accept water better. It could be kept pretty well flooded with a considerably lessened tendency toward erosion. Despite this, visual inspection showed that nothing approaching complete washing was being obtained, and this was, of course, substantiated by later analyses. The best cake picked from the Oliver filter in test V showed almost 10% X, dry basis.

Rate of filtration was quite acceptable; the 77 gallon batch containing 150 pounds of X filtered in a projected time of 100 minutes. Note: The pilot filter is to the proposed plant filter as the 150 pound X batch is to the proposed plant batch, and the "safe" time allowed for filtration in the proposed plant schedule is 200 minutes.

It should be noted here for the benefit of the record, if nothing else, that these high temperature "free filtering" cakes exhibited a marked tendency toward cracking. Also it was noted that the cakes that cracked the most showed the best filtration characteristics.

Fortunately, it was found that the cracks could be sealed quite effectively by the application of the wash water. In this respect, the soft or erosive nature of the cake was of positive value, as it enabled us to sluice enough solid matter from the surface of the cake into the cracks to permit further filtration and washing. On the other hand, this operation always comprised a compromise between the application of too little wash water, resulting in profound cake cracking, and too much wash water, resulting in profound erosion. Any suggestion that these cakes be filtered and "sucked dry" (without washing) was out of the question because of this cracking tendency. Note: The latter might constitute a possible means of operation when combined with a sufficiently generous repulping operation.

Test W was a duplication of test V. However, the time (262 minutes) required to filter the batch was over twice that encountered in batch V. That this was due to blinding of the heavy 101N Vinyon cloth could be readily seen by the "spotty" nature of the cake on the various areas of the panel sections.

The average cake thickness was, of course, considerably less in test W than in test V, and as a result, somewhat better washing was encountered. The 7.3% X, dry basis, exhibited by the cake from test W is the best yet found.

The 248 pounds of wet gelatinous cake collected from the first filtration (but not including the "heal" from the Oliver bowl) was repulped in 19 gallons of water, and the resulting 40 gallons of slurry again filtered. The drum was operated at the same rate in both filtrations—6 minutes per revolution. To further study the matter of cloth blinding, the filtration was stopped during the run and the fabric thoroughly scrubbed. Cake thickness increased appreciably following said cleansing, but blinding had set in again before the run was completed.

Again, largely due to the slow filtration rate (thin cake) the washing of the repulp cake was the best yet encountered. Said cake contained only 4.4% X, dry basis. It might, under perfect conditions, have been washed as low as 1.4% X. In other words, soluble X to the extent of 3.0%, dry basis, could still have been washed out. This is decidedly not good operation.

Before making test X, the heavy 101N Vinyon cover was removed, washed and replaced. At the same drum speed employed in the prior test (6 m.p.r.), the filtration rate was not very different, considering the greater volume of this latter batch. (Some one was apparently "heavy-handed" with the water hose.) Washing was poorer than obtained on the previous run. The data sheet said "Some blinding; cake washed off."

At this point, or even before, it had become obvious that the matter of cloth blinding was most critical. In fact, unless some means were found to circumvent it, the commercial operation of the process was doomed to failure. Microscopic examination of the 101N Vinyon showed it to be composed of large cords (fibro bundles) closely woven, forming rather deep pockets in which the slime could easily lodge. Increased air pressure on the filter discharge (1.5 lb. while blowing) failed to effect a cure. Back washing or even continual brushing seemed out of the question.

In searching for a better weave, a piece of Vinyon 119N was finally obtained. This is a medium weight cloth of the same high temperature acid resisting fiber. It has a much smoother surface finish (microscopically) and it was hoped that it might be the answer to the dilemma.

Accordingly, for test Y, the Oliver was dressed with this new cloth. The filtration appeared fairly satisfactory, although subsequent analyses showed no further improvement in washing efficiency, either upon first filtration or upon repulp. Incidentally, at this point, the practice or repulping in dilute (1%-2%) nitric acid was inaugurated.

In test Z, the drum speed was reduced somewhat to allow more time for washing, as the previous test had shown this new setup capable of filtering the batch well within the allowable 200 minutes. At said lower speed, a fairly well washed first cake was obtained, —3.2% X.

Upon repulp the X content was further reduced to 1.6%, which is the best recorded result to date. However, it will be noted that, even at the high drum speed of 3 minutes per revolution, an excessive filtration time was again encountered. This again indicated cloth blinding.

3. Changing the Character of the Residue

To this point, considerable progress had been made. Use of higher filtration temperatures and the selection of a more suitable cover had resulted in improved filtration behavior. Despite this, it was apparent that the problem of cover blinding was still with us. Notwithstanding the better surface of the 119N fabric, severe blinding was again encountered upon the fourth use of the new fabric.

Likewise, while considerable improvement had been made in the quality and washability of the cake, there was still much to be desired. While the high-temperature cake accepted wash water better, a wash approaching perfection was seldom achieved, even with a clean free-filtering cover. Furthermore, a point not shown by the unadorned data sheets: Even this improved high temperature cake was far from foolproof. In other words, it was a soft, gelatinous cake, which could be easily eroded and washed off the drum by the application of excessive quantities of water. With the urgency for complete washing which exists, this is an unhappy feature to translate into full scale usage.

With those facts in mind, it was decided to conduct a series of experiments to the end of altering the nature of the precipitate, specifically, to try to eliminate its bulky, gelatinous characteristics. Apparently, much of the silicious matter of AAA ore is reprecipitated during the digestion as silicic acid. In past practice a pronounced thickening of the slurry was invariably noted as the latter part of the nitric acid charge was added to the slurry of ground ore and water.

It was postulated that the addition of the ore to nitric acid should result in a denser form of precipitated silicic acid. A denser cake should hold less X values, purely by virtue of mechanical entrainment, it should be more amenable to the application of water, and it might possibly wash better and cling to the cloth less.

With these desiderata in mind, tests 101-103 were made, adding the ore to the nitric acid. Upon completion of the digestion, entire freedom from the thickening phenomenon was apparent.

To show conclusively that the reduced bulk of the precipitate, and of the filter cake, were the results of procedural changes, rather than of some change in the ore, batch 104 was made up as a duplicate of batches Y and Z. As expected, the gelatinous bulky cake of prior practice resulted.

As just indicated, cakes formed on the filter in tests 101-103 inclusive were much less bulky and much more amenable to the mechanical application of wash water than cakes of prior practice. Visually, the cakes, even from the first (digestion) filtration, were well washed. All data sheets stated that the color of the liquor was well displaced. Because of poor manipulation of the 100 cc samples, poor laboratory results, or "reversion" peculiarities, a rigorous comparison of the washing efficiency (analytically) is impossible. If we can take the "insoluble X" value of 5.1% (step 5) of test 101 as gospel, the washing upon first filtration was complete in this test. Also, in test 102, we find that the first filtration cake had only 0.9% X, against a base of 0.4%. Just why the 100 cc samples of the repulp showed 2.6% X with a washed repulp cake of 2.5% X can only be explained by poor sampling, bad analyses or reversion. The latter is entirely possible. It is the moreso when it is remembered that this experimental Oliver "spit-back" an awful lot of strong liquor with the discharge cake. (Cake samples, for record, avoided this spit-back.)

Despite the fact that the new type precipitate behaved much better than the former gelatinous precipitate (a fact more apparent from observation than from recorded results) the rate of filtration still left considerable to be desired. Not one of these digestion slurries filtered within the allotted 200 minutes.

It was the firm belief of those in charge of operations that this cake should filter at a reasonable rate. Failure to do so was believed to lie solely in the phenomenon already discussed at considerable length: cloth blinding. So, rather than pursue that problem further in connection with the Oliver filter (time was short) it was decided to transfer the test work to the Feinc filter.

The Feinc filter had previously been ruled out because its cake rollers, wash belt, string discharge, etc., would not operate with the soft gelatinous cake of prior practice. The new precipitate would, however, undoubtedly behave on the Feinc. Given a chance of "mechanical" success, insofar as cake formation and discharge were concerned, it was believed that the Feinc had a very good chance of solving the one remaining filtration stumbling block—cloth blinding. This subsequently proved to be the case.

SUMMARY AND CONCLUSIONS

The Oliver filter was tested quite extensively on two types of precipitate resulting from the digestion of AAA ore, and its subsequent treatments.

The solids resulting from digestions wherein the acid was added to the ore slurry were of a bulky gelatinous nature.

When this precipitate was filtered at lower temperature (140-180°F) good cakes were formed, but these were so very soft that application of wash water was virtually impossible.

By filtering the same sludge at higher temperature (190-210°F) some improvement was attained, with respect to the acceptance of wash water. However, the improvement was not sufficient to guarantee full freedom from the cake erosion difficulties encountered when applying wash water to the cake on the drum.

Even if operations could have been further improved with respect to completeness of washing, it was felt that this type of precipitate would not be of commercial utility. Between the minimum quantity of water required to keep cake cracks sealed, and the maximum quantity of water which resulted in severe cake erosion, there existed only a relatively small leeway.

A second type of precipitate was produced by adding the ore to the acid. This formed a much denser non-gelatinous filter cake which had much less cracking tendency and accepted water better. Being much less "soft" application of wash water had far less tendency to erode this cake, i.e., to wash it off the drum.

Throughout all of these tests, the problem of filter cloth blinding or sliming was recognized as an outstanding stumbling block. Several types of cloth were tested and some improvement was noted. However, neither different cloths nor improvements of the type of precipitate eliminated the blinding troubles sufficiently for us to feel secure in handing the process over to plant operation. All cloths operating on all precipitates blended badly after a few short runs.

Experience in the industry is the effect that sliming (cloth blinding) precipitates are best handled on very light weight cloths. Such fabric is impractical on a filter of the Oliver type, where cake discharge depends upon back-blowing to loosen the cake, followed by its removal against a "doctor knife" on a taught wire.

On the other hand, the Feinc filter is organized to operate with light weight cloths, —no back blow or doctor knives are employed. Previously, the Feinc string discharge filter had been ruled out for handling the soft precipitate, but with the discovery of the technique for preparing a dense precipitate, it was believed that the Feinc could now be made to operate.

If the Feinc could be made to handle the precipitate (mechanically) there was a very good chance that, by virtue of its different organization from the Oliver, we might be able to solve the cloth blinding problem. To this problem, there seemed to be no practical solution in connection with the Oliver.

7-30-45

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

1ST BATCH ON "NEW YALE" OR "NO 3" PROCESS
$BaCO_3$ ADDED WITH ORE. OLIVER LOW TEMP

Batch 5

Nitric	Dilution	Barium	Sulfuric	Ore
199# TOTAL 171# FOR ORE HNO_3/ORE = 0.56 RATE: 0.5 G.P.M.	30 GALS	24¾# $BaCO_3$	21.5# 66°	300# X = 35.02% P_2O = 6.4 % MoO_3 = 0.99% My = 1.7X10⁻ g/g
Order 4	Order 1	Order 2	Order 5	Order 3

Digestion
#4 VOL. 45 GAL.
FOLLOWING 5, ADDED 15 GAL COLD WATER
TEMPS: 209-212
Time: start 4 to stop 5
140 MIN.
Vol. 55 gal.

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric (AFTER DILUTION)	Ore
SOL. 24.3g/100			SOL. = 27.2g/100	
X = 50.6 g/l			X = 46.5 g/l	
HNO_3 = 00 "			MoO_3 = 0.45 "	
MoO_3 = .05 "			My = 2.8X10⁻ g/cc	
			X = 11.8%	
			My = 3.0X10⁻ g/g	

Wash water 9¼ gals net
Temp. 160°F

Filter Dressed LOW TEMP. VINYON
WATER CHANNELS CAKE. 1/8 - 3/16 CAKE. VERY MUSHY

6.0 MPR
22" VAC
OLIVER

Time, Minutes
Actual = 160
Projected = 460

Filtrate I
Sp.Gr.
Vol. 24 gal.
X = 82 g/l
MoO_3 = 0.75 g/l
My = 5.4X10⁻ g/ml
SO_4 = 3.9 g/l

Cake I
Wt. 126 lbs.
X - 8.4% 5.8%
H_2O - 36.5 39.9%
X(DRY) 13.0 - 9.6%

Repulp Fluid
33 GAL. H_2O

Pounds Heel =

THIS GANGUE FILTERED IN SPERRY P+F PRESS

Wash Cake
Gal. net

Repulp Cake 212°F
Total Cake
Wt. 275 lbs.
Vol. gal.
100 cc sample

MPR

Filtrate II
Sp.G.
Vol. 76 gal.

Time, Minutes
Actual =
Projected =

Cake II
Wt. 188 lbs.
X = 3.4%
H_2O = 38.8%
My = 3.3X10⁻⁹ g/g
X(DRY) 5.6%

Pounds Heel =

Combined Filtrates
gal.
X=
My=

Chemical GLC
Total Cake lbs.
Gross Cake lbs.
X content lbs.

281 010

DECLASSIFIED

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

Batch *I*

NO 3 PROCESS – HI ACID – OLIVER
BARIUM AS $Ba(NO_3)_2$ FROM HERE ON

Digestion	Nitric	Dilution	Barium	Sulfuric	Ore
4 – 73 GAL. *5 – 70 GAL.* *TEMPS. 196–221*	*244# 38°*	*30 GALS.*	*24.5# $BaCO_3$* *28.5# HNO_3 38°* *25 GAL. H_2O*	*20.5# 66°* *2.5 GAL. H_2O*	*300#* *X = 41.4%* *P_2O_5 = 5.7%* *MoO_3 = 0.57%* *My = 1.8×10^{-7} g/g*
	Order *3*	Order *1*	Order *4*	Order *5*	Order *2*

Time: start stop *3* to stop *5*
132 MIN
Vol. *63* gal.

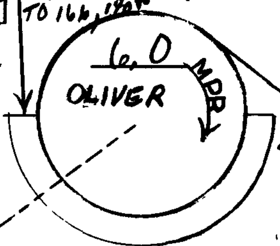
Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric	Ore
SOL. = 21 g/lim cc *X = 67.4 g/l* *HNO_3 = 9 g/l* *MoO_3 = 0.045 g/l*			*SOL. 20.5 g/lim cc* *X = 50.6 g/l* *My = 4.5×10^{-11} g/cc* *X = 0.28%* *My = 3.3×10^{-9} g/g*	

Wash water ______ gals net
Temp. ______

FLOODED? *COOLED 1 HR TO 166, 170*

Filter Dressed *LOW TEMP VINYON? NEW?*
ADDED "DRAG CLOTH"
CAKE 1/4"



Filtrate I
Sp.Gr. *1.46*
Vol. *35* gal.
SOL. 0.2 g/l

Time, Minutes
Actual = *120*
Projected = *194*

← 142°F END OF RUN TEMP ROSE AND CAKE + WASH IMPROVED

Pounds Heel = *112# HEEL*

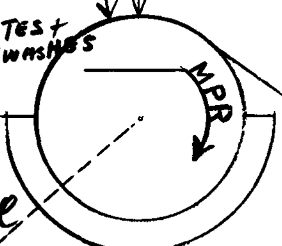
Cake I
Wt. *181* lbs.
X = 6.4%
H_2O = 52.0%
X = 13.3% DRY

Repulp Fluid
35 GAL. H_2O

REPULP FILTERED ON SPERRY

Wash Cake ______ Gal. net

POLISHED FILTERATES + WASHES



Filtrate II
Sp.G. *1.185*
Vol. *140* gal.
X = 90.3 g/l
MoO_3 1.0 g/l

Time, Minutes
Actual =
Projected =

Pounds Heel=

WASHED IN SPERRY
Cake II
Wt. *207* lbs.
X = 0.85%
H_2O = 46.9%
Ba = 7.5%
DRY X = 1.6%
DRY My = 5.2×10^{-7} g/g

Repulp Cake
Total Cake
Wt. *293* lbs.
Vol. ______ gal.
100 cc sample

Combined Filtrates
______ gal.
X= ______
My= ______

Chemical GLC
Total Cake ______ lbs.
Gross Cake ______ lbs.
X ... ______ lbs.

281 011

8-3-45

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

DUPLICATE OF "T"

Batch U

Digestion
3- 70 GAL
4- 79 "
5- 81 "
202-206°F
Time: startstop
3 to stop 5
126 MIN
Vol. 81 gal.

Nitric
244# 38°
HNO_3 / ORE = 0.81
Order 3

Dilution
30 GAL
Order 1

Barium
24# $BaCO_3$
28.5# HNO_3 38°
10 GAL H_2O
"SLIGHTLY ACID"
Order 4

Sulfuric
20.5# 66°
Order 5

Ore
122# X
300#
X = 40.86%
H_2O = 3.7%
M_oO_3 = 1.29%
P_2O_5 = 1.55%
Order 2

SCREEN	%
+20	8.6
50	27.7
80	17.2
100	6.8
200	27.8
-200	11.9

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric	Ore
X = 48.6 g/l			X = 40.9 g/l	
HNO_3 = 8. g/l			P_2O = 1.0 "	
P_2O = 51 g/l			My = 3.1 X 10^{-10} g/cc	
			X = 5.3%	
			My = 3.9 X 10^{-7} g/g (ORY)	

Filter Dressed

Wash water
FLOODED ~~gals~~
Temp. 160°F net

Filtrate I
Sp.Gr. 1.35 LAB / 1.32 PLANT
Vol. 53 gal.
SOL. 0.4 g/l

3.0 MPR
OLIVER

Time, Minutes
Actual = 90
Projected = 117

1/4" CAKE WASHED 1/2
17" VAC
DRY

Cake I
VERY SLOPPY
Wt. 300 lbs.
X = 6.6%
H_2O = 51.4%
DRY X = 14.2%

Repulp Fluid
30 GAL. H_2O

Pounds Heel = 92#

HEEL WASHED TO X - 5.3%, H_2O - 35.8%, X DRY 8.2%

2000 F
REPULP FILTERED ON SPERRY

Wash Cake
_____ Gal. net

Filtrate II
Sp.G. _____
Vol. _____ gal.
TAKEN FROM RESLURRY TANK

MPR

Time, Minutes
Actual =
Projected =

Cake II
Wt. 235 lbs.

Repulp Cake
Total Cake
Wt. 392 lbs.
Vol. 68 gal.
100 cc sample

Pounds Heel =

Combined Filtrates
140 gal.
X = 99.0 g/l
~~My~~ = SO_4 3.27 g/l
SpGr. 1.185

Chemical GLC
Total Cake _____ lbs.
Gross Cake _____ lbs.
Z ... ment _____ lbs.

281 012

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

8-6-45

1ST HI TEMP RUN ON OLIVER

Batch V

Digestion
2- 42 GAL
3- CONSIDERABLE FOAMING
3. 64 GAL
4. 86 "
4- 91 "
5- 80 "
200-220°F
Time: start stop 3 to stop 5
157
Vol. 77 gal.

Nitric
296 # 38°
HNO_3 / ORE = 0.85
Order 3

Dilution
30 GAL.
Order 1

Barium
39.2# $BaCO_3$
33# HNO_3 38°
10 GAL H_2O
DILUTED TO 25 GALS.
Order 4

Sulfuric
27# 66°
3 GAL H_2O
Order 5

Ore 150#
348#
X = 43.06%
H_2O 4.2
PbO 6.44
MoO_3 0.57
SiO_2 19.6
P_2O_5 1.20
Order 2

SCREEN	%
+ 20	5.4
50	19.9
80	12.1
100	8.9
200	29.0
-200	24.7

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Sulfuric
SOL. 16.5 g/100cc	SOL. 22 g/100cc
X = 43.9 g/l	X = 46.3 g/l
HNO_3 6 g/l	PbO = 0.84 g/l
MoO_3 = 0.045 g/l	MoO_3 = 0.6 "
	My = 2.5 X 10⁻¹¹ g/cc
	X = 2.7% → LATER REPULPED AND WASHED AGAIN. X = 1.36%
	My = 3.6 X 10⁻⁷ g/g

Filter Dressed VINYON 101 N COVER (HEAVY "DUCK" WEAVE)
PLACED 6 ADJUSTABLE SPRAYS ON FILTER

Wash water 16 gals net
Temp. 4 SPRAYS
200°F

OLIVER 6 MPR

Filtrate I
Sp.Gr.
Vol. 44 gal

Time, Minutes
Actual = 77
Projected = 109

CAKE 1/4 - 5/16 VERY WET

Pounds Heel = 120

Cake I
Wt. 278 lbs.
X = 6.8 4.9%
H_2O = 55.0 54.7%
X 14.6 9.8%

Repulp Fluid
30 GALS.

"CLOTH ABOUT 75% PLUGGED"

Wash Cake
11 Gal. net

Filtrate II
Sp.G.
Vol. 13 gal.

3.0 MPR
170°F

Time, Minutes
Actual = 45
Projected = 205

1/4" CAKE
VAC. 20-25"

Pounds Heel = 232

Cake II
VERY WET
Wt. 65 lbs.
X = 2.5%
H_2O = 65%
X = 7.2% D.
My = 3.5 X 10⁻⁷ g/g A

Repulp Cake 214°F
Total Cake Wt. 398 lbs.
Vol. 61 gal.
100 cc sample
X = 4.3% D.

Combined Filtrates
BOILED 180 → 130 gal.
X = 142. g/l
My = 4.9 X 10⁻⁸ g/l
MoO_3 = 1.3 g/l

Chemical GLC
Total Cake 297 lbs.
Gross Cake ______ lbs.
X ... ent ______ lbs.

281 013

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

8-8-45

DUPLICATE OF V (CLOTH BLINDING)

Batch W

Digestion
2. 43 GAL.
3. 67 GAL.
MIX CURDY AND GELATINOUS
4. 86 GAL.
5. 78 GAL.
200 - 220 °F
Time: start stop 3 to stop 5 INCL.
175 MIN
Vol. 78 gal.

Nitric
296 # 38°
HNO_3 = 0.85
ORE
Order 3

Dilution
30 GAL.
Order 1

Barium
29.2 # $BaCO_3$
33 # HNO_3 38°
10 GAL. H_2O
DILUTED TO 25 GAL.
Order 4

Sulfuric
24.5 # 66°
3 GAL. H_2O
Order 5

Ore 150 # X
348 #
SEE "V"
Order 2

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric	Ore
		SOL. 15.5 g/100cc	SOL. 20.8 g/100cc	
		X = 44.2 g/l	X = 45.4 g/l	
		HNO_3 9.2 g/l	P_2O = 1.8 g/l	
		MoO_3 0.03 g/l	MoO_3 = 1.9 g/l	
			M_4 = 7.0 X 10^{-11} g/cc	
			DRY X = 1.4%	
			DRY P_2O = 4.2%	
			DRY MoO_3 0.19%	

Filter Dressed VINYON 101 N
HOODED DRUM TO KEEP WASH SPRAY HOT

Wash water
56 gals net
Temp. 4 SPRAYS

Filtrate I
Sp.Gr.
Vol. 80 gal

OLIVER 6 MPR
183°F - 192°F
"1/2 WASHED"
VAC 17 - 22"

Time, Minutes
Actual = 200
Projected = 262

Pounds Heel = 78

Cake I SAMPLE "80% WASHED"
Wt. 248 lbs.
X = 2.9%
H_2O - 60.3%
X = 7.3%

Repulp Fluid
19 GAL

CLOTH BLINDING. BRUSHED DURING RUN - OK.
BLINDING AGAIN AT END.

Wash Cake
60 Gal. net

Filtrate II
Sp.G. 1.08
Vol. 28 gal.
SOL. = 0.1 g/l

6 MPR
196°F

Time, Minutes
Actual = 180
Projected = 300

Pounds Heel = 83

Cake II
Wt. 128 lbs.
X = 1.6%
H_2O = 64.1%
D.X = 4.4%

Repulp Cake OLIVER ONLY
Total Cake
Wt. 248 lbs.
Vol. 40 gal.
100 cc sample
BACK COMPLETED
X = 6.1 g/l

Combined Filtrates
BOILED TO 134 gal.
X = 141 g/l
My = 1.3 X 10^{-10} g/cc
P_2O = 4.76 g/l

Chemical GLC
Total Cake 211 lbs.
Gross Cake ______ lbs.
X content ______ lbs.

281 014

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

8-10-45

Batch X

ANOTHER "DUPLICATE"

Digestion	Nitric	Dilution	Barium	Sulfuric	Ore
2- 51 GAL. 3- 80 GAL. 4- 90 " 5- 90 " 200-214	296# 38° HNO_3/ORE = 0.85	30 GAL	29.2# $BaCO_3$ 33# HNO_3 38° 10 GAL. H_2O	25.5# 66° 3 GAL. H_2O	150# X 348# SAME AS "V"
Time: start 3 to stop 5 151 MIN Vol. 90 gal.	Order 3	Order 1	Order 4	Order 5	Order 2

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric	Ore
SOL. 13 g/100cc X = 40.3 g/l HNO_3 = 13 g/l P_2O = 1.36 "-?			SOL. 21 g/100cc X = 40.8 g/l My = 5.0×10^{-9} g/g DRY My = 3.2×10^{-7} g/g X = 1.1 %	

Wash water 60± gals net
Temp. ______

Filter Dressed: REMOVED VINYON 101 N, WASHED + REPLACED. TOOK OUT STRETCH + FIXED CLEATS. TRACED + INSULATED BOWL

Filtrate I
Sp.Gr. 1.395
Vol. 52 gal.
SOL. = 0.9 g/l

OLIVER 6 MPR 196°F

1 1/2# BLOW. SOME BLINDING.

Time, Minutes
Actual = 226
Projected = 294
CAKE WASH OFF

Cake I
Wt. 295 lbs.
X = 5.4 %
H_2O = 49.8 %
X = 10.8 % D.

Repulp Fluid
30 GAL

Pounds Heel = 89# Heel washed TO 1.0% X d.

SOME BLINDING.

Wash Cake
9 Gal. net

Filtrate II
Sp.G. ______
Vol. 18 gal.
SOL. = 0.06 g/l

3.3 MPR

Time, Minutes
Actual = 130
Projected = 180

7/16" CAKE

Cake II
Wt. 249 lbs.
X = 4.2 %
H_2O = 57.3 %
X = 10.0 % D.

Repulp Cake 220°F
Total Cake
Wt. 384 lbs.
Vol. 57 gal.
100 cc sample

Pounds Heel = 94-WASHED TO 2.2 % XD.

Combined Filtrates
______ gal.
X = 94.7 g/l
My = 2.4×10^{-11} g/cc
P_2O 3.02 g/l
P_2O_5 0.11 g/l

Chemical GLC
Total Cake 343 lbs.
Gross Cake ______ lbs.
X content ______ lbs.

8-14-45

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

SAME (FILTRATION STUDIES) OLIVER - 119 N CLOTH
ACID REPULP

Batch Y

Digestion
2- 39 GAL
3- 60 GAL
4- 67 "
200-205 °F
Time: start stop
3 to stop 5
105 MIN
Vol. 70 gal.
67 GAL. TO FILTER

Nitric
288 # 38°
$\frac{HNO_3}{ORE}$ = 0.83
Order 3

Dilution
30 GAL
Order 1

Barium
29.2 # $BaCO_3$
33 # HNO_3 38°
10 GAL. H_2O
Order 4

Sulfuric
25 # 66°
3 GAL. H_2O
Order 5

Ore
145# X
348 #
X = 41.73 %
P_bO = 8.1 %
MoO_3 = 0.6 %
P_2O_5 = 0.94 %
My = 1.4X10^{-7} g/g
Order 2

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric	Ore
		SOL. 25 g/100cc	X = 57.2 g/l	
			PbO = 1.65 "	
			P_2O_5 = 0.77	
			My = 2.5X10^{-10} g/g	
			X = 2.2 %	
			My = 3.6X10^{-7} g/g	

Wash water
35 gals
Temp. ____ net

Filter Dressed INSTALLED VINYON 119 N (LIGHTER CLOTH WITH SMOOTHER FINISH.) ALSO IMPROVED CALKING, USING SARAN TUBES WITH S.S. RODS

3.3 MPR
OLIVER
198 °F
GOOD DISCHARGE
22" VAC
NO BLINDING

Time, Minutes
Actual = 90
Projected = 120

Filtrate I
Sp.Gr. 1.44
Vol. 46 gal.
X = 127.5 g/l
MoO_3 1.4 "
P_2O_5 1.85 "
My = 2.5X10^{-10} g/cc

Cake I
Wt. 250 lbs.
X = 3.8 %
H_2O = 55.6 %
X = 8.6 % D.

Repulp Fluid
30 GAL H_2O
9 # 38° HNO_3

Pounds Heel = 86 HEEL WASHED TO 9 % X D.

Wash Cake
17 Gal. net

3.3 MPR
190-200 °F
NO BLINDING

Time, Minutes
Actual = 85
Projected = (120)

Filtrate II
Sp.G. ____
Vol. 27 gal.

Cake II
Wt. 202 lbs.
X = 1.9 %
H_2O = 60.5 %
X = 4.8 % D.

Repulp Cake 212 °F
Total Cake
Wt. 336 lbs.
Vol. 67 gal.
100 cc sample
X = 8 g/l
SOL. 17 g/100
CALCULATED

Pounds Heel = 86 # WASHED TO 12 % X D.

Combined Filtrates
____ gal.
X= ____
My= ____

Chemical GLC
Total Cake 288 lbs.
Gross Cake ____ lbs.
X content ____ lbs.

381 016

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION
FILTRATION STUDIES - OLIVER

Batch 7

Digestion
2- 40 GAL.
3- 70 "
SLURRY THICKENS
4- 77 GAL.
5- 77 ".
200-205°F
Time: startstop 3 to stop 5
116 MIN
Vol. 77 gal.

Nitric — 288# 38° HNO_3 / ORE = 0.83. DID SOMEONE ADD 10 GAL. EXTRA ACID? — Order 3

Dilution — 30 GAL. — Order 1

Barium — 29# $BaCO_3$; 33# 38° HNO_3; 10 GAL. H_2O — Order 4

Sulfuric — 25# 66°; 3 GAL H_2O — Order 5

Ore — 153# X — 350#; X = 43.56%; P_bO = 5.79%; MoO_3 = 0.78%; SiO_2 = 19.4%; P_2O_5 = 1.01%; My = 1.6×10^{-7} g/g — Order 2

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric	Ore
SOL. 14.3 g/100 cc			SOL = 21.3 g/100 cc	
X = 60 g/l			X = 46.3 g/l	
HNO_3 = 19 g/l XX			My = 1.3×10^{-10} g/cc	
X = 0.38%			MoO_3 = 0.21%	
			X = 0.13%	
			My = 4.1×10^{-7} g/g	

Filter Dressed

Wash water 32 gals net
Temp. ______

Filtrate I
Sp.Gr. 1.38
Vol. 60 gal
SOL. 0.9 g/l

OLIVER — 2.92 / 4.5 MPR — START-25 MIN — 190-210°F

Time, Minutes
Actual = 120
Projected = 162

1/8" CAKE WELL WASHED

Cake I
Wt. 350 lbs.
X = 1.3 / 4.7%
H_2O = 59.7 / 58.7
X = 3.2 D. 11.4% D.

Repulp Fluid
31 GAL.
1% HNO_3

Pounds Heel = 87 WASHED TO 4.7% X D.

Wash Cake
39 Gal. net

Filtrate II
Sp.G. ______
Vol. 58 gal.

3 MPR

Time, Minutes
Actual = 284
Projected = 350

1/16 - 1/8" CAKE

Cake II COMPOSIT
Wt. 185 lbs.
X - 0.55%
H_2O - 66.7%
X - 1.6% D.

Repulp Cake
Total Cake
Wt. 337 lbs.
Vol. 61 gal.
100 cc sample
X = 2.5 g/l
SOL. = 14.3 g/100 cc

Pounds Heel = 81

Combined Filtrates
______ gal.
X = 133.6 g/l
My = 3.5×10^{-10} g/cc
P_2O_5 = 0.74 g/l
HNO_3 = 47 g/l

Chemical GLC
Total Cake 266 lbs.
Gross Cake ______ lbs.
X content ______ lbs.

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

CHANGE DIGESTION: — ORE TO ACID (CONC.)

Batch 101

Digestion
2- 34 GAL.
REACTION XTHER.
CONSIDERABLE
BROWN FUMES.
5- 95 GAL.
NO THICKENING
OF SLUDGE

185-208 °F
Time: startstop 2 to stop 5
132 MIN
Vol. 75 gal.

Nitric
284 # 38°
$\frac{HNO_3}{ORE}$ = 0.81
HEAT TO 190°F
ADD ORE.
Order 1

Dilution
30 GAL
HOT WATER
Order 3

Barium
29 # $BaCO_3$
33 # HNO_3 38°
10 GAL. H_2O
Order 4

Sulfuric
25 # 66°
3 GAL. H_2O
Order 5

Ore
148 # X
350 #
X = 42.07 %
H_2O = 5.7 %
P_2O = 6.67 %
MoO_3 = 0.75 %
P_2O_5 = 1.21 %
My = 1.7 X 10^{-7} g/g
Order 2

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric	Ore
		X = 44.3 g/l	X = 38.6 g/l	X = 90.8 g/l
		HNO_3 = 0 "	P_2O_5 = 1.4 "	HNO_3 = 6.0 "
		P_2O_5 = 0.97 g/l	My = 2.0 X 10^{-10} g/cc	P_2O_5 = 1.79 "
		X = 5.1 %	X = 5.1 %	X = 1.0 %
		P_2O_5 = 1.68 %	My = 4.4 X 10^{-7} g/g	

Filter Dressed

Wash water
35 gals net
Temp. ______

Filtrate I
Sp.Gr. 1.32 (1.250 LAB)
Vol. 55 gal.
SOL. = 0.9 g/l

OLIVER — 16 MIN — 3.0 MPR, 4.5 ", 5.7 " MPR — 190°-210°F

Time, Minutes
Actual = 139
Projected = 311

1/8" CAKE WELL WASHED
1/8" " " "
3/16 " " "

Pounds Heel = 161

Cake I
Wt. 130 lbs.
X = 2.7 %
H_2O = 45.0 %
X = 4.9 % D.

Repulp Fluid
31 GAL. OF
2% HNO_3 (100%)

Wash Cake
6 Gal. ~~net~~

Filtrate II
Sp.G. 1.055
Vol. 15 gal.
SOL. = 0.8 g/l

5.7 MPR — 195°F

Time, Minutes
Actual = 65
Projected = 252

Pounds Heel = 184

Cake II
Wt. 63 lbs.
X = 3.8 %
H_2O = 41.0 %
X = 6.4 % D.

Repulp Cake 205 °F
Total Cake
Wt. 291 lbs.
Vol. 52 gal.
100 cc sample

Combined Filtrates
______ gal.
X= ______
My= ______

Chemical GLC
Total Cake 247 lbs.
Gross Cake ______ lbs.
% ... ______ lbs.

281 018

 8-21-45

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

Batch 102 ORE TO DILUTE ACID. FILTER TESTS - OLIVER

Digestion
2 - 52 GAL.
3 - 63 "
4 - 74 "
5 - 76 "
200-205 °F
Time: start stop 3 to stop 5
137 MIN
Vol. 76 gal.

Nitric
284# 38°
HNO_3/ORE = 0.81
LESS BROWN FUMES THAN Ba 101
Order 1

Dilution
33 GAL.
Order 2

Barium
29.2# $BaCO_3$
3.3# HNO_3 38°
10 GAL. H_2O
Order 4

Sulfuric
25# 66°
3 GAL. H_2O
Order 5

Ore
148#
350#
SAME AS BATCH 101
Order 3

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric	Ore
		SOL = 12.8 g/100		
		X = 50.7 g/l	X = 46.3 g/l	X = 60.6 g/l
		HNO_3 = 5 "	P_2O_5 = 1.58 "	HNO_3 = 8 g/l
		P_2O_5 = 1.42 "	My = 1.0 X 10^{-10} g/cc	P_2O_5 = 2.03 "
		X = 0.5 %	X = 0.4 %	X = 0.8 %
		P_2O_5 = 0.79 %	P_2O_5 = 1.02 %	P_2O_5 = 0.8 %

Wash water
25 gals
Temp. 3 SPRAYS net

Filter Dressed
IS CLOTH BLINDING?

Filtrate I
Sp.Gr. 1.27 LAB. 1.32 PLT.
Vol. 37 gal.

OLIVER 6 MPR
20" VAC.
Time, Minutes
Actual = 117
Projected = 480
OLIVER SAND:-
X = 1.7 %
SiO_2 = 42.3 %
H_2O = 7.7 %
1/16" CAKE WELL WASHED
Pounds Heel = 97#

Cake I
Wt. 61 lbs.
X = 0.38 %
H_2O = 56.3 %
X = 0.9 % D.

Repulp Fluid
15 GAL. OF 2 % HNO_3

Wash Cake
9.5 Gal. net

Filtrate II
Sp.G. 1.148
Vol. 9.5 gal.
X = 73.4 g/l

1.3 MPR
Time, Minutes
Actual = 37
Projected = 95
Pounds Heel = 128

Cake II
Wt. 82 lbs.
X = 1.3 %
H_2O = 48.0 %
X = 2.5 % D.

Repulp Cake
Total Cake
Wt. 257 lbs.
Vol. 34 gal.
100 cc sample
X = 2.6 %

Combined Filtrates
gal.
X = 1.58 g/l
My = 1.5 X 10^{-10} g/cc
HNO_3 = 43 g/l
P_2O_5 = 3.05 g/l
SA Gr. = 1.282

Chemical GLC
Total Cake 210 lbs.
Gross Cake lbs.
X content lbs.

8-22-45

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

Batch 103 — ORE TO STRONG ACID. OLIVER TESTS.

Digestion	Nitric	Dilution	Barium	Sulfuric	Ore
2- 32 GAL. 3- 45 GAL 4- 44 GAL. 195-206 °F	284# 38° HNO3/ORE = 0.81		29.2# BaCO3 33# HNO3 38° 10 GAL. H2O	25# 66° 3 GAL. H2O	149# X 350# X = 42.33%
Time: start stop 2 to stop 4 150 MIN	Order 1	Order	Order 3	Order 4	Order 2
Vol. 44 gal.					

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric	Ore
		SOL. = 21g/100cc	X = 80.4 g/l	SOL. = 24.5 g/100cc
		X = 82.8 g/l	HNO3 = 13 "	X = 112 g/l
		HNO3 = 1.6 g/l	MoO3 = 0.26 "	HNO3 = 6 "
		MoO3 = 0.086 g/l	P2O5 = 1.55 "	MoO3 = 0.004 "
		P2O5 = 0.88 g/l	X = 3.1 %	P2O5 = 2.08 "
		X = 9.7 %	MoO3 = 0.98 %	X = 2.9 %
		MoO3 = 1.70 %	P2O5 = 0.80 %	MoO3 = 1.85 %
		P2O5 = 2.63	SiO2 = 36.06 %	P2O5 = 0.91 %

Wash water: 44? gals net; Temp. ___ — TAKES H2O WELL

Filtrate I: Sp.Gr. ___; Vol. 30 gal

Filter Dressed — CONSIDERABLE EVIDENCE OF CLOTH BLINDING

OLIVER — 6 MPR — 190-210 °F

Time, Minutes: Actual = 158; Projected = ; 1/8 - 3/16" WELL WASHED

Pounds Heel = 111

Cake I: Wt. 144 lbs.; X = 2.7 %; H2O = 45.7 %; X = 5 % D.

Repulp Fluid: 31 GAL. 2% HNO3

RESLURRY FILTERED ON SPERRY

Wash Cake: ___ Gal. net

Filtrate II: Sp.G. ___; Vol. ___ gal.

MPR

Time, Minutes: Actual = ; Projected =

Cake II: Wt. 208 lbs.

Pounds Heel =

Repulp Cake: Total Cake Wt. 255 lbs.; Vol. 61 gal.; 100 cc sample; X = 2.8

Combined Filtrates: ___ gal.; X = 122.5 g/l; My = 9.0 X 10^-12 g/cc; HNO3 = 20 g/l

Chemical GLC: Total Cake ___ lbs.; Gross Cake ___ lbs.; X content ___ lbs.

281 020

8-23-45

K-1-E PILOT PLANT SUMMARY SHEETS
DIGESTION AND FILTRATION

REVERTS TO OLD PRACTICE:— ACID TO ORE
OLIVER

Batch 104

Digestion
2 - 42 GAL
3 - 70 "
4 - 80 "
5 - 76 "

196 - 205

Time: startstop
3 to stop 5

Vol. ______ gal.

Nitric	Dilution	Barium	Sulfuric	Ore
284# 38° HNO_3 / ORE = 0.81		39# $BaCO_3$ 33# HNO_3 38° 10 GAL. H_2O	35# 66°	352# (153#) X = 43.7 % H_2O = 4.3 % ? = 6.06 % MoO_3 = 0.58 % SiO_2 = 20.06 % P_2O_5 = 0.88 %
Order 3	Order 1	Order 4	Order 5	Order 2

Analytical Results on 100 cc samples from Digestion after addition of above reagents. (Multiply Liquors by 5.)

Nitric	Dilution	Barium	Sulfuric	Ore
SOL. 18.2 g/100cc		SOL. = 20.4 g/100cc	SOL. = 26.7 g/100cc	
X = 43.6 g/l		X = 51.1 g/l	X = 57 g/l	
HNO_3 = 5 g/l		HNO_3 = 12 g/l	MoO_3 = 0.74 "	
MoO_3 = 1.73 %		MoO_3 = 0.01 "	P_2O_5 = 1.09 "	
MoO_3 = 0.008 g/l		MoO_3 = 1.73 %	My = 6.8 × 10⁻¹⁰ g/cc	
X = 7.0 %		X = 4.1 %		
P_2O_5 = 1.66		P_2O_5 = 1.32 %	X = 4.0 %	

Wash water
32 gals net
Temp. ______

Filter Dressed

1/8" - 1/16"

Filtrate I
Sp.Gr. 1.32 (1.33 LAB)
Vol. 37 gal.
SOL. 5.6 g/l

OLIVER — 6 MPR

VERY SOFT, POORLY WASHED CAKE

Time, Minutes
Actual = 150
Projected = 262

Cake I
Wt. 240 lbs.
X = 2.4 %
H_2O = 56.3 %
X = 5.5 % D.

Repulp Fluid
31 GAL OF 2 % HNO_3

Pounds Heel = 176

RESLURRY FILTERED ON SPERRY

Wash Cake
______ Gal. net

Filtrate II
Sp.G. 1.318
Vol. ______ gal.
SOLUTION FROM RESLURRY
X = 72.6 g/l

MPR

Time, Minutes
Actual =
Projected =

Cake II
Wt. 288 lbs.

Repulp Cake
Total Cake
Wt. 416 lbs.
Vol. 61 gal.

100 cc sample

Pounds Heel =

Combined Filtrates
______ gal.
X = 122.3 g/l
My = 8.3 × 10⁻¹¹ g/cc
MoO_3 = 1.75 g/l

Chemical Cl... [illegible]

Gross Cake ______
X ... ment ______

281

021