Metallurgy by William Kern

PROCESSING

BLUE RIBBON GROUP

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MINA, NEVADA

METALLURGY

BLUE RIBBON MINE GROUP

March 1, 1967 First day on location, no metallurgy, but worked around the laboratory cleaning up.

March 2, 1967 Much the same as March 1. Crushed ore with hammer to size it for the crusher.

March 3, 1967 Calculation of boiler horse power to heat leaching solution for 200 tons of ore. Determination of volume of water to just cover the 200 tons of ore per vat was made on a sample of 200 mil volume (266grms) of (-9plus 20) mesh sized ore. The volume of water required to fill the voids in this sample is 85ML. On a second sample-not used in this calculation, but determined on a sample of the ore containing slimes,fines and oversize the volume of water to cover 200 ML of the ore was 113ML. This indicated that the boiler horse power required is subject to the size of the particles in the crushed ore and final determination should not be calculated until the size of the ore feed and consequent volume of water is determined. 200 ML volume of (-9 plus 20) mesh ore = 266 GM weight of water to cover 200 ML ore = 85 GM. Ratio of volume of water to volume of ore = 85/200 = .425 = 42.5 % voids.

200x0.425 = 85 = 85 tons water for 200 tons of ore. Operating temperatures of leach solution is to be 175°F. Assumed temperature of the water supply is 40° F. 175 -40 = 135° F. Temperature rise for the leach solution.
Tons xLbs/Tons Temp. rise
85 x 2000 x 135 = 22,950,000 = BTU to heat batch of water from 40° to 175° F.
BTU value of fuel oil = 18000/gallon.
22,950,000/18,000 = 1275 = gallons fuel oil to heat 85 tons of water charged to each leach batch, for 135°F. temp. rise.
1275/24 = 531 = Gallons per hour of fuel for 24 hour period.
1 HP Hour = 2.545 BTU.
$\frac{53.1 \times 18,000}{2545}$ = 37.3 = 37.3 HP Boiler to heat water in leach from 40° F. to 175°F. in 24 hours.
The heat load for the ore is based on specific heat of quartz or 0.24 BTU 1 lb equivalent to approx. 5 gal. fuel for 24 hrs.

3-4-67 On job until 3.00 PM waiting for an outside fire assay.

Monday
3-6-67

Leach Test
16 oz. ore
6.8 oz. water
39 GM NaCN

5 N. solution of NaCN
for leach solution
6.8 oz H$_2$O = 192.44 GM H$_2$O
$\frac{192.44}{5}$ = 38.49 GM NaCN

Results: Two days leaching time. 5N.NaCN solution will not extract all of the copper and as leach progresses, the leached copper oxidizes and drops out of the solution, becoming mixed again with the ore. Result: Unsatisfactory.

Wednesday
3-21-67

1 lb ore (plus 9 mesh)(some $\frac{1}{4}$'' size)
80 GM NaOH. 10 NaOH in 200 ML H_2O
50 GM $(NH4)_2 SO_4$
water first added to ore, then NaOH, then 40 GM $(NH4)_2SO_4$. Heated to 180°F.
Addition of Ammonium Sulphate caused very strong reaction between hot
NaOH solution and the $(NH_4)SO_4$. Much ammonia escaped. This also weakened
the NaOH solution. At 30 minutes the leach was incomplete. At 60 minutes
the leach was very near complete. The ore was stirred frequently during
leach.

Leach #1   Leach began 8.05 A.M.
Leach ended 9.30 A.M. 1 hour 25 minutes.
The leach solution began turning black in color indicating insufficient
caustic. This required an end to the leach. CuO did not adhere to
tailings.

Leach #2   1 lb. ore (plus 9 mesh) Same as #1.

80 Gm. NaOH
200 Ml H2O
10 GM $(NH4)_2SO_4$. Ammonium sulphate to be added only after leach has
                    nearly completed with caustic.

Start: 10.00 A.M.
11.10   leach solution turning black. Added the $(NH4)_2SO_4$ 10 grams.
Decanted and washed 11.15 A.M.
The Ammonium Sulphate did not redissolve the copper oxide.
Testing solvent power of $(NH_4)_2SO_4$
1 lb. ore
200 ml H_2O
132 gm $(NH_4)_2SO_4$ = 5N. solution
Start 2.20 PM

March 23, 1967   Test on Urea

1 lb. Ore (plus 9 mesh)
80 gm Caustic
200 ml. water
20 gm. Urea
Water added to ore then caustic added to make solution. Heated to 175°F.
(plus or minus 5° F.)
10 gm. urea added after 15 minutes leaching, then 10 more gms. urea at
20 minutes of leaching. No violent reaction between caustic and urea.
A slight odor of ammonia was perceptable. At 30 minutes leach the solution
was decanted from ore. Hand picked the greenish-colored particles out
of the tailings. The copper did not form the oxide after dissolving to
the extent of former tests. Volume of copper bearing solution approx.
250ml.

10.50 A.M.   Start 2nd Leach
1 lb. ore
200 ml. water
80 gm. NaOH
added to water covering ore. After 35 mins. leaching at 175°F. added
25 gm. Urea. to hold copper in solution. Continued leach. The copper
oxide began forming. Ended leach. Decanted caustic solution from tailings.

3-23-67

Leach #3

1 lb. ore , 200 ml water, 80 gms. NaOH, 25 gm. Urea, all mixed together dry, then added water.
12.55 P.M. start
3.00 P.M. Faint odor of ammonia. Temp. 165°F. Copper oxide is not forming.
3.40 P. M. The black copper is forming. End of leach. Heated too long.
CuO formed too much.

Leach # 4

4.00 P.M. Tailings from # 3 Leach above.

Add 80 gm NaOH, 200 ml water, 25 gm Urea.

4.15 P.M. Temperature 187°F.
4.20   "   180°F.
4.22   "   190°F.
4.25   "   200° F.
4.34   "   175° F
4.35   Leach ended

3-24-67

200 gm Tailings from Leach #2 of 3-23-67
80 gm NaOH, 25 gm Urea

8.20 A.M. Start leach of Tailings
8.40 A.M. Leach removed from heat. Oxides forming. Indication is that oxides form when carbonates are dissolved completely.

54 lbs. H₂SO₄ = $ 16.20   1 lb. H₂SO₄ = .30   1 oz = 1.8
13 oz. NaOH =   .27   1 oz. NaOH = 2.07

9.30 AM Acid leach starts
1 lb. ore, 80 grams com. H₂SO₄

10.30 End of acid leach. Decanted and washed Tailings.

10.50 ## Second Test
Tailings from acid treatment after one wash. Add 80 gm. NaOH plus 25 gm Urea. A blue color appeared around the dissolving caustic. A brown flocculent precipitate formed and floated in the solution. Temperature 160°--180° F.
Very heavy brown flocculent has separated from the ore.

11.55 End of caustic leach of tailings from H₂SO₄ acid leach.
The purpose of this test is to determine the efficiency of Sulphuric acid in leaching the silicified copper ore.
1. The cost of H₂SO₄ and NaOH are approximately the same at retail price.
2. The Sulphuric acid is far more efficient than caustic in a one-leach treatment. There is formed no copper oxide in the acid leach. No scrubbing equipment is required. The electrolyte is in a form to produce copper instead of copper oxide, which forms on electrolysis of a caustic solution.

3. The temperature of the leach may be less for H_2SO_4 without limiting its efficiency.
4. Separation of carbonaceous ore from silicified ore may be done, by screening as the carbonates are softer, and form ore fines which are to be screened out. A caustic treatment is the only practical method of extracting copper from the carbonate ore.

Friday 3-31-67   <u>Leach Test</u>   This size crush is too coarse for normal leach.

Ore: oversize on $\frac{1}{4}$ " screen of softest ore available, 1 lb. ore, 200 ml H_2O, 80 gm NaOH, 25 gm Urea, 50gm, sucrose.

start 7.45 AM.   8.20 AM Black ppt appeares to be forming-Temp. 160°F.

8.35   Solution turning green

10 grams copper per liter = 1% copper solution

100 grams Cu/L -- 10% Copper solution

8.50   Solution when stirred appeares light brown in color due to iron hydroxide.

9.00   Solution becomes red, especially at point of heating. Temp. 180°F. Red color most likely due to Cu. See page 695 Schlesinger.

10.00   Decanted and scrubbed tailings.

10.10   Tailings from the first treatment plus 200 ml H_2O plus 80 gm NaOH at 180°F.

10.20   Solution turned blue but copper content building up.

10.50   Copper blue solution beginning to change to black color. Leach continued through noon hour. 2.20 PM Temp. 175° F.

3.30   Temp. 200° F. The flame was cut down.

4.00   Decanted solution blue to black in color. Placed on hot plate and heated much more. Red copper formed. Bright red color

4.25   Decanted solution from scrub and washing tailings. Color changing to green after adding 2 spoons full of sucrose and heating. As solution warmes a brown color formed after green. Added plus or minus 5 gms. NaOH Solution began boiling and the red color increases.

4.36   Color becoming bright red.

4.45   Added 1 tsp. sucrose

4-1-67   Test on screen size. Set up electrolysis unit. Test on screen size to determine maximum size of particle in feed. Electrolysis unit operated Saturday night and was cut off Sunday. 3.76 gm copper and oxides after slimes were washed off. Slimes contained metallic copper.

-4-

4-3-67 Monday	1 lb. of ore from the pan containing (~.20") particle size including very fines. Previous leach tests have been made using 17.62% of the weight of the ore as added weight of caustic. 80 gm NaOH 200ml H_2O This test is made using 10% of the weight of the ore for NaOH. $\frac{10 \text{ ml sol.}}{}$ $\frac{454}{10}$ = 45.4 gm NaOH
11.00 AM	Start leach. Total water 400 ml to cover.
11.35	Caustic solution too weak.
11.40	Add 33.6 gm. NaOH Total NaOH 80 gm. Immediate strong increase in blue color. Add 200 ml more H_2O.
12.00	Leach removed from heat. Ore stacked out of solution.
1.00	Leach restarted.
1.10	Temp. 196° F.
1.22	Temp. 182° F. 200 ml H_2O added.
4.45	End caustic leach. No reagents but NaOH.
4-3-67 Tuesday	Tailings from above leach with slimes leached with 80 gr. H2SO4. Temp. to 200° F. Standardizing caustic solution weight 1.0211 gm Potassium acid, Phthalate, and Titrate with KOH solution. Titration $\cong$ 9.8 (9.9 immediately after titration.) $\frac{10}{9.9}$ = 0x1.0101 N.0101 x5 = 0.505 Normal KOH. Hydrochloric acid titration = 26.8 ml KOH using 25 ml HCL by pipette. Titration for $\frac{N}{2}$ HCL would have been 24.75 ml of KOH if KOH is 0.505 No
4-6-67	1.0211 gm Potassium acid Phthalate requires 10.2 ml $\frac{N}{2}$ KOH. 10.0 ml std $\frac{N}{2}$ HCL required 10.2ml of std. KOH. HCL checks with Potassium Acid Phthalate weighed and is exactly $\frac{N}{2}$ HCL. For KOH $\frac{10}{10.2}$ x $\frac{0.50}{1}$ = 0.4901 N.KOH 2 liters require $\frac{56.10 \text{ x } 2}{2}$ = 56.10 gm for full charge of KOH $\frac{1}{100}$ of 56.10 = 0.561gm 80% (1600ml) .8 $\overline{.4488}$gm approx. .45 gm KOH
4-5-67	Test on ore fines in HCL leach. 1 lb. ore fines 160 gm HCL, comm. Diluted before adding to the ore to 400 ml.
10.10 AM 10.20 "	calcite fizzing stopped, some slimes but not so much as per H2SO4
10.25	There is a dissolving action with formation of minute bubbles going on throughout test.
11.30.	Many particles have become red in color.
3.00 PM	Discontinued leach. A small amount of HCL on the red particles removed the red color.

4-6-67	Electrolyzed the H_2SO_4 leach on batch for April 3rd, second leach on that date. This product is acid recovery after caustic leach.
10.30 AM	Added 34 ml H_2SO_4 (con) to neutralize NaOH in caustic leach of Heads on Sample April 3rd, same sample later leached with H_2SO_4.

5 ml of leach solution required 1.3 ml $\frac{N}{2}$ KOH

1 ml H_2SO_4 conc. requires 50 plus 29 = 79 ml $\frac{N}{2}$ KOH washed out papette

1 ml conc H_2SO_4 papette drained only, not washed = 73.4 ml $\frac{N}{2}$ KOH

1 ml conc. H_2SO_4 titrates 73.4 ml $\frac{N}{2}$ KOH

4-10-67 below	**Quantitative Leach**

1 lb. ore fines pulverized(approx. 100 mesh)
400 ml water plus 100 ml
80 ml H_2SO_4 =

$$\left[\begin{array}{l} \text{100 ml grad.cyl} = 135 \text{ gm} \\ \text{80 ml } H_2SO_4 \text{ cut plus cyl} = 280 \text{ gm} \\ 280 - 135 = 14.5 \text{ gm} = \text{cut of } H_2SO_4 \end{array} \right]$$

1.35	Start heat
1.45	Sampled acid solution for initial acid consumption 1 ml sample from 580 ml. 1 ml dilute H_2SO_4 solution after 15 min. contact with ore =

4.2 ml $\frac{N}{2}$ KOH. Temp. 180° F. 73.4 x 80 = 5872 ml $\frac{N}{2}$ KOH (for total 80 gm)

580 x 412 = 2436 ml $\frac{N}{2}$ KOH subtract $\frac{2436}{3436}$ equivalent of H_2SO_4 used

2.40	End leach. Microscopic exam indicated no soluble copper on ore
3.30	Decant settling started
4-10-67 Monday	5,350 ml of electrolyte recovered---25 ml. for assay titration 26.0 ml

26.0 x 0.00522 = 0.13572 gm. Cu in 25 ml leach sol.

$\frac{5350}{25}$ = 214   0.13572 x 214 = 29.04 (gm Copper in leach sol)

$\frac{29.04}{454}$ = .064 = 6.4% Cu in ore recovered.

4-7-67 Friday	Standard solution of Sodium thio sulphate

39.04 gm / 2 liters ($Na_2 S_2 C_35H_2O$)
$\underline{\times 12}$
41124

H_2SO_4 solution does not digest CuO

4-8-67	Wt.of copper for standardizing $\underline{-}$ 2353 gr. (Scott -P193 p 140-1)
Saturday	This titration $\underline{-}$ 45.2 ml.
	84 ml. more water to stl. (can not make out next word)

4-10-67   10 gr. sample of tailings from H_2SO_4 leach to copper analysis.

1.00 PM   Weight copper $\underline{--}$ 0.3338

Titration $\underline{-}$ 63.5

For Standardizing Hypo   52.56

grams copper per ml $Na_2 S_2 O_3 5H_2$ $\underline{-}$ .00522 & .005256

1 lb. soft ore fines pulverized to plus or minus 100 mesh
add 400 ml water
add 80 ml (145 gm) comm. sulphuric acid
leach time $\underline{-}$ 1 hr.
-Washed leach residue 4 times by decant
Volume electrolyte recovered $\underline{-}$ 5,350 ml.
26.0 Titrix 0.13572 $\underline{-}$ .13572 x 214
Copper in electrolyte $\underline{-}$ 29.044 gm.
Percent copper in ore recovered $\underline{-}$ 6.4%
Grams copper per Liter, electrolyte $\underline{-}$ 5.428 gm
Copper leached per ton ore $\underline{-}$ 128 lbs.
Acid used by leach $\underline{-}$ 94.84 gm/lb
Percent of com. H_2SO_4 applied to leach $\underline{-}$ 58.51%
Lbs. acid used by leach per ton ore, this basis $\underline{-}$ 418.5 lb.
Iron (Fe) leached, per ton ore $\underline{-}$ 22.236 lb.
Lbs. com acid per lb. Cu recovered $\underline{-}$ 3.27
Weight of Tailings recovered $\underline{-}$ 3875 gm.

Cu plus Fe_2O_3 plus Tailings $\underline{-}$ 447.59 gms.
Copper in Tailings: 10 gm sample Titration $\underline{-}$ 1.1 ml
Titration $\underline{-}$ 0.11 ml 1 gr. Tails
Factor $\underline{-}$ 1 ml $\underline{-}$ .00522 x 0.11 $\underline{-}$ .0005742 gm. Cu/gr Tails.
0.0005742 x 2000 $\underline{-}$ 1.15 lb. Cu/ton Tailings

N A O H

4-11-67
Tuesday

80 grams NaOH in 400 ml H_2O = 20 ml $\frac{N}{2}$ HCL std. Titration

8.30 — Start leach
1 lb. ore   400 ml H_2O 80 gm NaOh   are pulvarized to plus or minus 100 mesh.

8.45 — Temp. 180^oF

9.00 — Leach is of creamy consistency. Added 100 ml H_2O

9.15 — Leach is soupy

9.30 — Microscopic examination of ore in leach reveals very low solvency of the Copper in the ore.

9.30 — Added 80 gm more caustic. Color on surface of leach is darker brown.

9.40 — Temperature-200° F.

9.53 — Surface of leach becomes very black when not stirred. Suggests keeping air away from caustic leach.

10.20 — Temp. 194° F.

11.00 — Leach almost dried out. Black all through cake.

11.40 — 200 ml water added. From then took 2 ml. to titrate. Titration = 18.7 caustic value

12.00 — Fire off for lunch

12.30 — Heat on

1.00 — Leach ended

4-12-67

Decanted leach. This is the caustic leach on sample treated 4-11-67
Electrolyzed H_2SO_4 solution on test begun on 4-10-67

4-13-67

(on aluminum rule: 50(5 units of 10 each) space = .28" on the ruler)

Drying Tailings from sample for caustic leach. One pound of this ore finely pulverized occupies 400 ml space in cylinder.

Tested one pound of finely pulverized soft ore for separation of slimes or very fine portion before leaching the copper ore concentrates of the separation. There is a portion of the ore that readily floats above the heavier copper bearing mineral. There are very small particles bearing an amount of copper that separates with great difficulty from the barren slimes. Actual loss can be determined only by analysis.

16.20 divided by 54 = . 30   C.P.H_2SO_4 = 30 ¢ /lb.

4-15-67	Dry weight of slimes decanted $\doteq$ 76 grams. Estimated 80% of calcite is in slimes. Slimes too high in copper to discard. Carbonate slimes contain 8.98 % Copper (4-17-67)
4-14-67	The sample treated 4-13-67 to separate slimes was used for further test. The concentrates were placed in a glass dish. Weight unknown but to be determined.
	Add 80 ml conc. H_2SO_4
	Add 250 ml water
8.45	Start heating. Indication is that most of calcite was removed from concts. Very little frothing or gas formed.
11.45	Off for lunch
12.30	Returned to digest $\bar{c}$ heat.
3.20	Off heat. Leach completed
	Filtering the slimes pulp. Settlement and decantation is much faster tahn filtering sands or slimes.
4-17-67	.10 ml of leach solution from concts of ore of leach on April 14, 1967 analysed for Cu $\doteq$ Titr $\doteq$ 13.4 ml Hypo.
	.00522 x 13.4 $\doteq$ .069948 or .070 copper
	3000 ml leach conct solution x 0.0070 gm cu/ml $\doteq$ .021.000 $\doteq$ 21.0 gm Cu recovered by leach solution.
	76 x .0898 $\doteq$ 6.8248 ------6.82 grams Cu lost in slimes cake (recoverable)
	300 gm. Tailings from conct leach
	76 gm. in slimes
	(see April 17, 1967 notes)
4-15-67	Separated slimes and dried them. Pipettes 10 ml from 10 gr sample diluted to 200 ml. $\dfrac{10}{200}$ x 10 $\doteq$ $\dfrac{1}{2}$ gm sample.
4-17-67	Titration $\doteq$ 8.6 ml 0.00522 Hypo.
	.00522 x 8.6 $\doteq$ .044892 Or 0.0449 x 2 $\doteq$ 8.98 % Cu in carbonate slimes.

4-18-67
Tuesday

10 ml caustic concts from April 11, 1967 analyzed. 1.0 ml of caustic solution titrated. 0.3 ml std Hypo. Solution is blue in depth but nearly barren of copper. Examination of Tailings shows major portion of copper converted to oxide and remained with the tailings.
The aqua regia solution used to strip all the copper from the tailings is heavily loaded with galatenous silica and is orange-yellow in color finally yellow. It was on this sample that results indicated that the air should be kept out of contact with the caustic leach.

2475 ml caustic leach pregnant solution
10 gm. sample tailings = 9.4 ml Titr. Hypo.
419 grams tailings

NaOH

April 19, 1967
Wednesday

1 lb. soft ore pulverized
80 gm NaOH   400 ml water

8.00 A M — Leach begins

Covered the glass dish with another glass containing water to collect vapor and prevent evaporation. Also to reduce air circulation in contact with leach solution.

8.10 — 200 ml more water

8.30 — Stirred cake. Temp. 190°-200° F.   Added 100 ml water. No blackening of top of cake.

8.45 — Stirred cake & sampled. Much of copper bearing ore is only slightly leached.

9.00 — Added 80 gm NaOH and 100 ml water

9.15 — Stirred leach. Added 100ml water.

ATTENTION

Before adding NaOH at 9.00 AM, the pulp had a darker brown color and the leach solution appeared to have little copper in it. At 9.15 AM the brown color was lighter and the leach solution stronger in blue copper color.

9.30 — Ample water and sufficient caustic has resulted in a very satisfactory appearance of this leach. Blackening at the surface has disappeared. Leach solution is strong deep blue.

9.45 — Sample of the ore washed free from slimes has many blue particles of ore incompletely leached. Pulp is covered with a deep blue solution in leach dish. No evident blackening at surface. The cover dish is kept half full of cold water.

10.00 — Stirred leach pulp every 15 minutes to avoid caking and dead spots in leach. Added 100 ml water. Pulp is clean light brown color in deep blue leach solution. Sampled blue solution.

strength
of caustic

{ 1 ml blue solution = 10.1 ml $\frac{N}{2}$ HCL
(Solution is 5 normal or contains 200 gm/liter of active caustic equal to
(80 grams caustic in 400 ml solution.

10.15	Stirred pulp. No additions to pulp.
10.30	Stirred pulp. No additions. Temp = 180° F - 210°F
	Scraped down sides of dish.
	There is approx. $\frac{1}{4}$ inch of leach solution covering pulp. Pulp bubbles but does not spatter.
10.45	Stirred pulp.
11.00	Stirred pulp. Sampled copper value in tails. Becoming less.
11.15	Stirred pulp. Color is clean light brown solids.
11.30	Stirred pulp. Leveled leach Dish. Pulp temp. 180° - 200° F.
11.45	Stirred pulp to flocculate it, open up to leach.
11.55	Added 200 ml water, stirred pulp. leave on heat for noon hour.
12.35	Removed leach from heat.

1 ml. leach solution titrate 6.6 ml $\frac{N}{2}$ HCL

$\frac{6.6}{2} = 3.3 =$ normality strength of active caustic

$3.3 \times 40 = 13.20 = 132$ gm NaOH per liter or 52.8 gm. NaOH in 400 ml leach solution.

1.00	Start first wash of Tailings by decantation
1.20	2nd wash of tailings started
1.40	End 2nd settlein
1.42	Start 3rd Settling
1.54	End 3rd settling
1.56	Start 4th settling
2.22	End 4th settling
2.25	Start filtering leached ore
2.30	volume of concentrated leach solution approx. 300 ml

4-20-67
Thursday

Weight, leach Tailings = 421 gm.
10 gm tailings for assay Cu. = 129.1 Titr.
10 ml leach pregnant solution for assay, Titr. = 0.25ml of Hypo
Total volume strong solution = 2550 ml.
Titr Hypo,-1gm sample tails = 12.5
1 gm Tailings, Fe_2O_3 wt. = . 1579 gm.

-11-

NaOH

Data: Caustic Leach   4-19-67

Leach time $=$ 4.5 hours

Temp. $=$ 160°- 206° F

454 grams ore --400 ml water --160 gm NaOH

Leach vat covered to prevent contact with air. Ore stirred at 15 min. interva
to assure full contact of caustic & ore.

Ore changed color after 1.25 hours leach time, believed due to oxidation
of copper caused by low caustic.concentration. 80 grams more caustic
added, bringing total caustic to 160 grams.

After two (2) hours leaching caustic consumption was 50% of total caustic
added. At end of $4\frac{1}{2}$ hours,leaching time caustic still available in leach
was 52.8 grams

Caustic consumed $=$ 107.2 grams.

Copper leached from ore pregnant solution $=$ (.00522) $\frac{(0.25)}{10}$ (2550) $=$ .2652gm

Copper in Tailings, 10 gram sample, $=$ (0.1) (.00522) (421) $=$ 28.365 gms.

Fe_2O_3 (iron oxide) in Tailings $=$ 15.79%

Results of test made April 21, 1967,(on next page,) indicate the useful
progress of the caustic leach ends as soon as black copper oxide begins
to form.

Next test using caustic shall be:

1 lb. ore(same as used 4-21-67)

600 ml water

120 gm. caustic

check regularly for CuO forming. Dip out spoonful sand and inspect
to fully determine if oxides are present.

Stop leach as soon as oxides form.

Beginning of caustic and acid leach, 1 lb. ore to each treatment. Ore is 9 mesh with fines, an ore that is harder than leachings of (4-10) and (4-19). Sample of 1 lb. is cut by splitter. (small one)

C A U S T I C

April 21, 1967
Friday

1 lb. ore
120 gm. NaOH   (60 gm at 2.00 P.M.)
600 ml water

* 2 ml of caustic solution (120 gm NaOH in 400 ml H_2O) titrates 28.5 ml of $\frac{N}{2}$ HCL, or 28.5 normal caustic solution.

10.05   Leach begins—leach vat(dish) covered to keep out air

10.15—stirred leach pulp   Temperature = 200° F

Time						Temp	
10.30	"	"	"		"	180° F	
10.45	"	"	"		"	190° F	(no slime in leach)
11.00	"	"	"		"	192° F	(no cementing of sands)
11.15	"	"	"		"	183° F	" " " " "
11.30	"	"	"		"	180° F	" " " "
11.45	"	"	"		"	180° F	" " " "
12.00	"	"	"		"	180° F	" " " "
12.45	"	"	"		"	195° F	(no water added up to this time. cover)
1.00	"	"	"		"	178° F	(condenses vapors)

Sands were sampled at 1.00 PM. Many particles are heavily loaded with copper. The leach solution is very dark blue with copper. 2 ml of leach *solution titrates 14.6 ml $\frac{N}{2}$ HCL- about ½ original strength

1.15 stirred leach pulp   Temperature = 174° F

Time						Temp	
1.30	"	"	"		"	"	
1.45	"	"	"		"	160° F	increased heat

* 1.55 adding 60 gm NaOH to leach

Time							Temp
2.00	stirred leach pulp, No cementing, Temperature =						180° F
2.15	"	"	"	"	"	"	190° F
2.30	"	"	"	"	"	"	185° F
2.45	"	"	"	"	"	"	180° F

* 2.50 2 ml of pregnant solution = 35.6 ml $\frac{N}{2}$ HCL

Time							Temp
3.00	stirred leach pulp, No cementing, Temperature =						180° F
3.15	"	"	"	"	"	"	176° F
3.45	"	"	"	"	"	"	180° F

out leach

1 ml strong solution(Cu) titr. = 2.7 ml Hypo
(.00522) (2.7) (1000) = 14.094 gr Cu. sampled 2.50 P.M.
4.05 PM ending the leach
very heavy copper oxide deposit formed on Tailings. This was scrubbed loose as much as possible and decanted from Tailings into beakers.

4-22-67

Tailings (dried residue, coarse sand) = 34 grams. Titrates(Hypo) 8.7 ml
Tailings (fines and oxides) = 56 gm s. 405 plus Fe & Hypo = 12.1
Fe_2O_3 weight = 17.84 one gram sample (source:coarse sand)
Strong solution 5 ml sample Titrate Hypo = 615
Copper in coarse sands = (0.00522) (8.7) (349) = 15.8194 gms.
Copper in fines & oxides (0.00522) (121) (56) = 3.5371 gms.
Copper in strong solution,(1 liter) = (0.00522) (6.5) (200) = 6.78 per liter
Total Copper accounted for = 26.1725 gns
2 ml strong solution = 15.4 ml $\frac{N}{2}$ HCL

Which is cheaper, to increase particle size and take greater Copper
loss, or make fine grind, shorten time of leach, and increase recovery
of Copper.

2 ml of solution--NaOH calculation- 120 gm NaOH in 600 ml water
Titration 2ml $\doteq$ 19.15 ml $\frac{N}{2}$ HCL $\doteq$ 9.575 ml $\frac{N}{2}$ HCL per ml of
caustic solution.

$\frac{120}{600} \doteq$ 0.20 gm NaOH per ml solution

0.20 gm NaOH Titrates 9.575 ml $\underline{\frac{N}{2}}$ HCL

NaOH plus H_2SO_4

Sunday
April 23, 1967

1 lb. - 9 mesh ore   120 gm NaOH   600 ml. H_2O
Leach dish is covered. Cover contains cooling water
2 ml. NaOH solution 120gm/600ml Titr. 19.15 ml $\frac{N}{2}$ HCL

8.20AM Start heating   8.28 Temp. 160°F   8.35 Temp. 180°F
8.45   Stirred pulp   8.50 Strong solution is pale blue color
9.00   Stirred pulp   Temp. 200°F 2 ml. caustic sol. Titr = 19.4 ml $\frac{N}{2}$ HCL
9.30   "   "   " ·178°F " "   "   ", Cu Titr. = <.6
9.50   25 gm Urea added
10.15   Ore sampled & checked for oxides. Oxides are beginning to form from
        copper in ore. The oxides are arrached to the ore particles where
        copper is visible in the ore . Solution is medium dense in blue color
10.30   Temp. = 150°F
10.35   Oxides rechecked. No increase evident
10.50   "   "   "   "   "
11.10   Temp. = 196°F
11.20   Ended leach. Washed ore twice
            strong solution 2 ml Titr = 17.15 available caustic plus Urea

(A) Strong solution, Copper-2ml, Hypo Titr = 2.3 ml   41.5 volume

(B) First wash = 445 ml - 5 ml sample Titrate = 1.0 ml Hypo

(C) 2nd wash = 380 ml - 25 ml sample   "   = 0.8 ml Hypo

(D) 2.30 PM Tailings, coarse sands-1gm sample. Titration Hypo = 10.3 ml

10.AM
4-24-67   Tailings, coarse sands after H_2SO_4 - 35 min. leach 5 gm sample = 12.2 ml Hypo
            Fe_2O_3 in caustic leach tailings = 1gm sample = 0.1788 gm

4-24-67   Tailings after sampling = 312 gms--$\frac{312}{454}$ = .6872
            calculating an H_2SO_4 wash for tailings.
            .6872 x 80 = 54.976 gm H_2SO_4 on basis of 80gm/454 gm.
            7.45 AM (acid = 55gm)(water 400 ml)
            7.50 AM Temp. 190°F   8.05 A M   Temp. 120°F
            Leach period for H_2SO_4 = 7.45 to 8.20 = 35 min.
            Start decant & settling '.50 AM

(F) 1 ml of acid solution from acid leach before washing Titrated = 3.6 ml $\frac{N}{2}$ KOH
(G) (Volume of H_2SO_4 leach solution plus washes = 750 ml
    (2 ml to analysis Cu = 6.4 ml Hypo

(A) strong solution = (0.00522) (2.3) ($\frac{1}{2}$) (415) = 2.991245 grams Copper
(B) first wash   = (0.00522) (1.0) ($1/5$) (445) = 0.465025 grams Cu
(C) 2nd wash   = (0.00522) (0.8) ($1/25$) (880) = 0.06347520 grams Cu
(D) Tailings, caustic leach = (.00522) (10.3) (1) (356) = 19.140696 grams Cu
(E) Tailings, acid leach = (0.00522) (12.2) (1/5) (312) = 3.9743808 grams Cu
  Total copper accounted for = 22.75 grams
(G) Acid recovery of Cu = (.00522) (614) ($\frac{1}{2}$) (750) = 12.528 grams Cu.

80 grams H_2SO_4 = 43.47 ml.

200 plus 43.47 = 243.47 ml

1 ml diluted acid titrates 14.1 ml $\frac{N}{2}$ KOH

$\dfrac{1.0}{243.47}$ x 80 (gm H_2SO_4) Titrates 14.1 ml $\frac{N}{2}$ KOH

$\dfrac{80}{243.47}$ = 0.3285 gm. conc. H_2SO_4/ 14.1 ml $\frac{N}{2}$ KOH

Tuesday 4-25-67	80 gm. conc. H_2SO_4 plus 200 ml. water = 24347 volume 2 ml. of above diluted acid Titration = 28.2 ml $\underline{N}$ KOH. $\frac{28.2}{2}$ = 14.1
8.30	Start heating 8.45 Temp. 184°F 9.00AM Added 100 ml water 9.19 Temp. 184°F
9.30	Sampled sands in leach-the copper is almost completely dissolved. Continuation of leaching process beyond the maximum recovery of copper leads to unnecessary sliming of the ore, increasing filteration or settling time.
9.45	Sampled pulp. A very small residue of recoverable copper remains in the ore. Pulp is caking some at point of greatest heat. 9.50AM. Temp. 184° - 200° F Uneven heating due to type of burners used.
10.00	Added 100 ml water to increase solvency of copper
10.10	Stirred pulp thoroughly. Temp.-170° F.
10.25	Sampled leach pulp. Very slight residue of Cu in ore.
10.30	End leach. Volume of pulp and leach solution = 450 ml. Strength of acid solution before washing & decant = 5.9 ml KOH for 2.0 ml of leach acid solution. $\frac{5.9}{2}$ = 2.95

4-26-67
Wednesday

Volume of electrolyte (strong solution) = 1370 ml. $\underline{N}$ KOH titr. = 3.5 on 2 ml sample.

Tailings = weight sample & beaker = 516 gm - 139 = 377 net.

1370 x 1.75 = 2397.50   3313.5 - 2397.5 = 916.0
235 x 14.1 = 3313.50   2397.5 ÷ 3313.5 = .7235 = 72.35% consumed

Tailings, Copper assay - 5 gm sample, titr. = 0.5 ml

* Copper in tails (0.00522) (0.5) (1/5)(377) = 0. 196794 grams copper in tailings
Strong solution Copper assay - 2 ml sample titr. = 6.0 ml Hypo
* (0.00522) (6.0) ($\frac{1}{2}$) (1370) = copper in electrolyte = 21.4542 grams = 15.66 gm/liter

Percent of total copper present which is lost in the tailings

= 0.1967 gm ÷ 21.65 gms = 0.92%

Total wt. Cu/ton of this ore $\frac{(21.65) (2000)}{454}$ = 95.373 lb. Cu/ton ore.

Wt. of Cu lost in tailings (95.373) (.0092) = 0.8774316 lb.

80 gm H_2SO_4 per lb. = $\frac{(80) (2000)}{454}$ = 352.42 lb. H_2SO_4/ ton ore

lbs. acid per ton ore consumed in this test = (352.42) (.2765) = 97.44 lb.

Rate of recovery of the copper = $\frac{95.373}{96.25}$ = 99.088%

April 27, 1967

Design the plant to best treat the ore available at the surface.
This ore may not be a true representation of the whole ore body, but
changes in the ore, if they affect efficiency, can be compensated for
by required plant changes.

Recirculation of spent electrolyte on acid leach may be impractical
on account of the iron dissolved from the ore. A use for the spent
electrolyte may be dissolving the oxide copper from the caustic leach
tailings.

C A U S T I C

<table>
<tr><td>April 26, 1967</td><td>Caustic solution: 600 ml water-120 gm NaOH titr. = 19.7 ml $\frac{N}{2}$ HCL
add 40 gm Urea. Titr. 18.8 (less than NaOH alone)</td></tr>
<tr><td>8.40</td><td>Start leach Temp. 160° F. 9 mesh ore fine grind. Leach tray containing ore rests in tray below filled with water which heats the leach solution & pulp without having hot spots. Pulp tray is also covered on top side with empty glass tray.</td></tr>
<tr><td>8.50</td><td>Water added to top tray.(a double boiler with cover)</td></tr>
<tr><td>8.55</td><td>Temp. 176° F.</td></tr>
<tr><td>9.00</td><td>Leach solution is deep blue. Sampled pulp</td></tr>
<tr><td>9.07</td><td>Temp. 176° F   9.33 Temp. 175° F. Stirred pulp</td></tr>
<tr><td>10.00</td><td>*   186° F   Pulp is dispersed, not caking. Sampled pulp. Pulp particles have not changed noticeably in color. Many copper bearing particles do not indicate any change from the leach, others have lost depth of color, the blue being less dense in them.</td></tr>
<tr><td>10.30</td><td>Temp. 176° F. No caking. 2 ml strong solution, titr = 19.1 ml $\frac{N}{2}$ HCL
Noticeable odor of ammonia when leach vat was uncovered.</td></tr>
<tr><td>11.20</td><td>Temp. 175° F.</td></tr>
<tr><td>12.00</td><td>Added 60 grams more NaOH. No water added. The caustic simply is not affecting most of the copper. Sampled the pulp. # 35 sample</td></tr>
<tr><td>12.45</td><td>Temp. 178° F.</td></tr>
<tr><td>1.40</td><td>Five hours of leaching, sampling pulp. Sample indicates there is no important gain in leaching the ore. The oxides are building up in this leach. Strong oror of ammonia in vapor above leach solution.
The oxides did build up toward end of leach</td></tr>
<tr><td>2.00</td><td>End of leach. The tailings when treated with conc. H_2SO_4 form an unfilterable silica or slime</td></tr>
</table>

Strong solution 1.ml sample titr = 1.3 ml hypo. 570ml strong sol.
wash water = 660ml. 570 plus 660 = 1230 ml total leach wash
1230 ml wash plus strong solution titr = 1.5 ml hypo on 2 gms sample
(.00522)91.5)($\frac{1}{2}$)(1230) = 4.8166 gm Cu recovered

Tailings -1gm -titr = 7.0 Wt. 420gm
(.00522) (7.0) (1.0) (420) = 15.34680 gm.   3.65% Cu.

April 25, 1967

Sulphuric Acid Leach

Ore (-9 mesh)	=	1.lb.
Solvent	=	80 gm. conc. H_2SO_4
Water	=	400 ml
Time	=	2 hours

Total Copper in sample	=	21.65 gm.
Copper extracted by leach	=	21.45 gm.
Copper lost in Tailings	=	0.1967 gm.

Percent of Copper in ore,lost totailings	=	0.92%
Pounds of Copper per ton of ore	=	95.37 lbs.
Pounds of Copper lost in 1 ton Tailings	=	0.8774 lb.
Rate of recovery of the Copper	=	99.088%

pounds of acid used, per ton of ore	=	352.42 lb
pounds of acid consumed, per ton of ore	=	97.44 lb.
percent of iron as (Fe_2O_3) in ore	=	17.88 %

Wednesday, May 3, 1967

1 lb. ore, fine grind

120 gm. NaOH   600 ml. water   40 gm. urea

1.00 pm   Start leach

1 lb. of finely ground ore in jar. Added caustic and mixed dry. Added water and rotated jar until all pulp is equally wet. A blue solution formed immediately. Added 40 gm. urea. Care used not to build up internal pressure to break jar. Jar then placed in water bath at 140° F.

1.15   Temp.   160° F   3.15 Temp.   164° F
1.50   "   176° F
2.00   "   170° F

4.30   End leach. Ammonia gas under compression escaped from the strong solution when autoclave (sealed jar) was opened.

5-4-67   Strong solution 600 ml
Thursday   total, leach strong solution & washes   3620 ml.

copper in leach solution & wash:
(0.00522)(2.8)(1/5)(3620) = 10.562 gm.
Tailings, weight = 440 gms.   Titr. = 5.4
Cu in tailings: (0.00522) (5.4)(1.0)(440) = 11.96 gm.

Calculation of Residual Acid

1 ml. $\frac{N}{2}$ KOH $\risingdotseq$ 0.3285 gm. H_2SO_4 spg 1.84

volume green (acid) solution $\risingdotseq$ 1150 ml.

Titration 1.0 green solution $\risingdotseq$ 1.0 ml $\frac{N}{2}$ KOH

$\therefore$ (0.3285)(1150) $\risingdotseq$ 377.77

$\frac{377.77}{14.1}$ $\risingdotseq$ 26.79 gm conc. H_2SO_4 Residual

50.0 gms. conc. H_2SO_4 charged in

26.79 gms. "   " residue

23.21 gms $\risingdotseq$ wt . gms. acid used to treat pulp.

$\frac{23.21 \times 2000}{454}$ $\risingdotseq$ 88 lb conc. H_2SO_4 — new ratio

consumed in treating 1 ton of leach tailings as of May 5, 1967

Calculation for Caustic   see 4-23-67

4/23/67 $\risingdotseq$ 120 gm NaOH in 600 ml water

$\frac{120}{600}$ $\risingdotseq$ 0.20 gm NaOH per ml solution

1.0 ml above solution titrates 9.575 ml $\frac{N}{2}$ HCL

Leach for 5/5/67 : 1 ml titrates 0.25 ml $\frac{N}{2}$ HCL

0.20 gm NaOH titrates 9.575 ml $\frac{N}{2}$ HCL

1 ml $\frac{N}{2}$ HCL $\risingdotseq$ $\frac{.20}{9.575}$ gm NaOH $\risingdotseq$ 0.0208 gm. NaOH

Alkaline pregnant solution $\risingdotseq$ 1750 ml.

Titration per ml. $\risingdotseq$ .25 ml $\frac{N}{2}$ HCL

(0.25)(1750)(.0208) $\risingdotseq$ 9.10 gm caustic

Residual in basic pregnant solution

May 5, 1967   <u>Caustic Leach with Meta Bi Sulfite</u>

1 lb. ore (-100 mesh)   40 gm. urea   Meta
                                                        Bi
600 ml water   40 gm $Na_2S_2O_3$ Sulphite

120 gm. Caustic (NaOH)

1.30 Start   1.45 PM   Temp. 160°F   1.50 PM   Temp. 176° F   4.25 PM Temp. 180°F

4.30   End of leach. Filteded by suction. Washed by suction until wash is
              colorless. Removed filtered cake from filter and returned to leach jar.
              To the pulp was added 400 ml water and 50 gm. conc. H_2SO_4. The cake was
              pulped in the acid solution and shaken for 5 min. filtered pulp.Washed
              pulp with 400 ml of water.Added ½ at a time. Final wash with 100 liters of
              water which is coming through the cake colorless . No heating was used
              during the acid wash.

Saturday   Blue Basic pregnant solution: 2 ml Titr. = 0.5 ml volume = 1750 ml.
May 6, 1967   (0.00522)(0.5)(½)9½)(1750) = 2.2837 gm.
              Green copper con. sol. Titr. = 3.7 volume = 1150 ml.
              Tailings, sands & fines = 375 gm. Titr. = 3.0
              (.00522)(3.0)(1.)(375) = Tailings = 5.8725 gm.
              Cu in blue strong = (0.00522)(.5|91.5)(1750) = 2.2837 gm
              Cu in green strong = (0.00522)(3.7)(.5)(1150) = 11.10
              Blue solution -2 ml. titrates 5.1 ml $\frac{N}{2}$ HCL
              green "   " sample titrates = 2.0ml $\frac{N}{2}$ KOH

              4/25/67 80 gm conc. H_2SO_4 in 200ml water . 2 ml of this was acid
              mixture Titrates = 28.2 ml $\underset{2}{N}$ KOH

              4/23/67 120 gm NaOH in 600 ml H_2O. Titration 2 mls. of this caustic
              solution = 19.15 ml $\frac{N}{2}$ HCL

May 9, 1967

The possibility of interference from calcite in the caustic leach should
fully investigated. If NaOH solution does not affect $CaCO_3$ (calcite)

could not be reached by the NaOH solution and would remain in the tailings.
This is the most plausable possibility yet occuring, to be checked.
Silicates should be more soluble in NaOH than in H_2SO_4 solution.

Iron in the electrolyte may become an impurity in the Copper extracted by
electrolysis. Calcite will comsume the acidity of the electrlyte, increasing
the ph to approx. 3.5 to 4.0, when iron will ppt as a hydroxide, and is
filtered off.

.049041 gm H_2SO_4 is equivalent to 1 ml of normal alkali (Lange Handbook
Pg. 1021)

$$\frac{.049041}{2} = 0.0245205$$

1 ml conc. H_2SO_4 Titrates 75.5 ml $\frac{N}{2}$ KOH

1.84 gm   "   "   "   "   " "

$$\frac{1.84}{75.5} = 0.02437 \text{ gm}$$

∴ 1 ml $\frac{N}{2}$ KOH = $\underset{.02452}{0.02437}$ gm. H_2SO_4

5/10/67

Analysed the products of the leach made May 8, 1967
The purpose of this tast was an attempt to remove the copper solution
from the ore before a coating of copper oxide should form on the ore
particles. The caustic and urea were divided into thirds and one third
used for each stage. This lowered the leaching strength of the caustic
solution and extraction was very small. The acid wash was imperfect because
the cake had hardened in lumps before vacuum filtering and a number of
unpulped lumps were noticed. When the acid wash was emptied upon the
filter. The copper in the filter paper was not recovered.

May 8, 1967

Monday

1.lb ore   40 gm NaOH   13.3 urea   200 ml H_2O

9.10 AM   Start leach.   Temp. 180°F

9.40   End leach. Filtrate has some copper

10.15   Start second stage: same amounts of NaOH & Urea on tailings from first
            treatment, 100 ml H_2O 180° F

10.40   Pulp is too thick to pour. Add 100 ml water. After water the pulp flows
            freely. 180° F steady.

11.15   End second stage. Filtered & washed

11.45   Begin 3rd) stage of leach 200 ml water - 13.3 gm urea -40 gm caustic

2.10   End 3rd) "   "   "

            Fourth stage: 30 gm. H_2SO_4 plus 250 ml water. acid wash was in contact
            with ore cake for 7 mins-filtered very slowly.

            Weight of tailings = 375 gms. 1 gm for Cu analysis. Titr = 8.1 ml hypo.

5.00   Tailings analysis - 1 gm sample = 375 gm = hypo Titr = 8.1
            First stage filtrate & wash = 750 ml   hypo Titr. = 0.10 on 2 ml
            Second stage "   "   "   " 780 ml   "   "   " 0.20 " " "
            Third "   "   "   " 780 "   "   "   " 0.20 " " "
            Acid treatment of cake   660 "   "   "   " 2.2   " " "

May 9, 1967
Tuesday

Treated Tailings with 30 gm conc H_2SO_4 in250 ml water
Some of the cake failed to pulp, microscopic examination of the acid washed
tailings exposed presence of small flakes of copper oxide.
2 ml. of H_2SO_4 solution after treating ore titrates = 1.2 Ml N KOH
(660)(0.6)(.02437) = 9.577 gm H_2SO_4 residual after washing cake with
diluted acid. This is equivalent to 90 lbs. H_2SO_4 per ton of ore cake.
(.00522)(8.1)(375) = 4.228% Cu. = 15.8557 gm Cu in Tailings
68.27% insoluable Fe_2O_3 = 21.77

Cu in first stage = (.00522)(.05)(750) = 0.19575
"   " 2nd   "   " (.00522) (.10) (780) = 0.40716
"   " 3rd   "   " (.00522) (.10) (780) " 0.40716
"   " 4th acid "   (.00522)(1.1)(660) " 3.78972
"   " tailings "   (.00522)(8.1)(375) " 15.8557
                                        _____
                                        20.65519 Total Cu.

May 10, 1967

Preparation of Head Samples from the ore brought from the mine
on May 9 & 10, 1967, by Larry & Joe.

May 11, 1967   Thursday

Data on head samples prepared
5/10/67

6.05
1.15

4.90 Titr. on heads

$(.00522)(4.90)(1) = 2.5578\%$ Cu $= 51.1560$ lb. Cu /ton ore

Weight of Fe_2O_3 $= .1480 = 14.80\ \%\ Fe_2O_3$

Weight of insoluble $= .7225 = 72.25\ \%$ insoluble

Total-Cu, Fe_2O_3,& insoluble $= 89.61\%$

-26-

80 gm NaOh in 400 ml water titrates 9.9 ml $\frac{N}{2}$ HCL/ml

80 gm NaOH/L = 2N, .˙. 80 gm/400 ml = $\frac{10}{4}$ x 2 = 5N

1 ml 5 N NaOH Titrates 10 ml $\frac{N}{2}$ HCL

GM   lbs.

$\frac{80 \times 2000}{454 = 1\#} = 352.4 = lb/t$

---

1 ml of above caustic solution contains = 0.020202 gm NaOH

$\frac{80}{400}$ = 0.2 gm/ml(there are 400 ml) .˙. $\frac{0.2}{9.9}$ = 0.020202 gm NaOH per ml $\frac{N}{2}$ HCL

100 lb/t = 22.7 gm/lb

$\frac{100 \; lb}{2000 \; ton}$ = .05 (urea)

(.05)(454) = 22.7 gm/lb

May 12, 1967   1 lb ore from heads sample, 5-10-67

80 gm NaOH   400 ml water   22.7 gm urea (100 lb/ton urea)

1.45   Start heating leach pulp   Temp. 180°F steady

3.50   Leach ends. very strong ammonia odor with strong copper solution

Strong copper solution = 300 ml. 1 ml strong titr. = 7.2 $\frac{N}{2}$ HCL

(.020202)(7.2)(300) = 43.6363 gm NaOH

Wash water = 640 ml. 1 ml wash titr. = 2.25 ml $\frac{N}{2}$ HCL

(.020202)(2.25)(640) = 29.0906 gm·(caustic) NaOH

```
 300
 640
 195
-----
1135.0
```

Barren wash water 195 ml titr, 1 ml = 0.3 ml $\frac{N}{2}$ HCL

(.020202)(0.3)(195) = 1.1818 gm NaOH

balance of caustic reacted with copper or is residue in the tailings.

```
43.6363
29.0906
 1.1818
---------
73.9087 gm NaOH
     or ammonia
```

Saturday
May 13, 1967   Weight of tailings = 4.05 gm   Titr = 4.2 ml hypo
Silica jell is present in strong solution
Copper in tailings = (.00522) (412)(1)(405) = 8.879 gm Cu = 2.192%

Monday
May 15, 1967   10010 (?) Titr. 5 gm ore heads = 33.7 ml hypo.
(.00522) (33.7)(1/5) = .0351828 = 3.52% Cu in heads
3.52% = 15.9808 gm Cu 1 lb.

Strong solution & washes, Cu recovered = 3.4362 gm   2.9 ml hypo titr.
(.00522)(1/5)(1135)(2.9)

Tailings: 5 gm. sample
(0.00522)(1/5)(30.0) = 3.13% = 12.6846 gm. Cu.
15.9808 gm Cu in heads = 3.4362 plus 12.6846 = 16.1208
                    strong solution

        15.9808 = 16.1208
        Heads " recovered & Tailings

These results establish correctness within one-tenth of one gram per pound of ore.

Installed new burner for incinerating filter paper, greatly increasing accuracy of analysis. See: Keefer-page 98

Caloric acid eliminates the interference of iron in electrolytes.

P 45., Vol 2

May 16, 1967 Tuesday

On April 25th the test results indicate that 100 lbs. H_2SO_4 per ton
of ore is consumed in leaching the ore, or a ratio of 100:2000.
This is equivalent to 22.7 gm of H_2SO_4 per lb. of ore. A margin is n
necessary to provide leaching power at the end of the leach. This test is t
be made with 30 gm H_2SO_4 per lb. ore or 1.321 x 22.7
80 ml water plus 21.1 ml H_2SO_4 = 95 ml sol.

3000 divided by 1.42 ≜ 2 ml.

DATA ON PAGE FOLLOWING

May 17, 1967 Wednesday

Another attempt to establish a lower acid charge for leaching the copper.
This test to be made with 60 grams H_2SO_4 --60 Gm H_2SO_4 measures 32.6 ml
60 Gm. H_2SO_4 in 400 ml H_2O. 2 ml acid sol titr = 11.6ml $\frac{N}{2}$ KOH
on first batch. Broke jar with hot water.
(.00522)(.2)(19.0)(825)

(.00522)(3.8)(825) = 16.3647 Gm. Cu in strong solution. This exceeds
the value of the heads obtained previously.

May 17, 1967 Wednesday

1 lb. ore--60 gm H_2SO_4 --400 ml water

7.50   Start leach

10.10   Temp. 160° F   11.20 Temp. 160°F   12.50 Temp. 172°F

12.50   End leach

Wash water = 450 ml.

Strong solution 375 ml. titr = 24.5 $\frac{N}{2}$ KOH

Iron hydroxide 6.4 ml $\frac{N}{2}$ KOH

Strong solution & washes = titr Hypo = 19.0

(.00522)(1/5)(19.0)(825) = 16.3647 gm Cu recovered

Rate this test at 99.99% recovery of Copper. Microscopic examination
of sample of tailings did not disclose one blue particle.

May 16, 1967

Tuesday

1 lb. ore - same ore as test begun on 5/12/67

30 grams H_2SO_4

400 ml water

Test to be made using jar as container, heated at 180°F in water bath.

8.15   Start leach

10.15   End leach

leach was made in open jar to allow contact with air.

Recovery of strong solution $=$ 270 ml.

Acid in strong solution, titr $=$ 5ml titr $=$ 13.4 ml $\frac{N}{2}$ HCL

Check conversion of $FeSO_4$ to $Fe(OH)_3$

2nd wash $=$ 340 ml

3rd "   " 330 ml

1 ml $\frac{N}{2}$ KOH $\Longrightarrow$ .02452 gm H_2SO_4

1 ml $\frac{N}{2}$ HCL $=$ .39999 gm NaOH $=$ .0199995

$$\begin{array}{r} 270 \\ 340 \\ 330 \\ \hline 940 \end{array}$$

(.245205)(2.68)(270) $=$ 17.7440 gm. H_2SO_4 equivalent remaining in 1st sol

Strong solution & washes $= \frac{N}{2}$ KOH $\frac{5.6}{5} = 1.12$

(.02452)(1.12)(940) $=$ 25.8146 gm H_2SO_4 equivalent in the electrolyte- deduct

the $\frac{N}{2}$ KOH reacting with the iron which becomes ferric hydroxide.

10.0 ml strong solutions & washes for Cu

(.00522)(1/10)(18.9)(940) $=$ 9.22 gm Copper recovered
         titr

Heads, Cu (same heads as for 5/12/67 $=$ 15.98 Gr/lb

  Recovery, strong solution & washes " 9.22 "

Tailings, by difference   " 6.76 "

Tailings, weight 420 grams

It is indicated by these results that 30 grams of Sulphuric acid per pound of ore is not sufficient to complete the leaching of the copper, high iron content in the ore also weakens the acid available for leaching copper.

In titrating with $\frac{N}{2}$ KOH to measure the unused acid in an acid leach, the iron and copper also react with the KOH giving a false reading too high in acid value, the ph range of phenolphthalein is 8.3 to 10. Iron forms the hydroxide at a ph of 3.4 to 4. Therefore all of the iron reacts before the indicator color changes. This may be avoided by using an indicator which changes color on or before ph 3.4. Such an indicator is BROMOPHENPL BLUE also identified as TETRA BROMO PHENOL Sulphon-PHTHALEIN, PH range $=$ 3.0-3.6 on this leach. 43.3 % of Copper remained in tailings.

NOTICE

Ore (9mesh)   1 lb.
Solvent   80 gm conc. H_2SO_4
Water   400 ml
Time   2 hours.

Total Copper in sample	21.65 gm
Copper extracted by leach	21.45 Gm
Copper lost in tailings	0.1967
percent of Copper ore, loss to tailings	0.92%
pounds of Copper per ton of ore	95.37 lb.
pounds of Copper lost in tailings	0.8774 lb
Rate of recovery of the Copper	99.088%
Pounds of acid used, per ton of ore	352.42
lbs. Acid per ton of ore, consumed in test	97.44 lbs
Percentage of iron oxide (Fe_2O_3) in ore	17.88%

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May 22, 1967   Monday

Sulphuric acid leach test of May 16, 1967

Analysis of tailings for Copper. Cu = 0.00522 %

0.00522 % Cu. = 0.93 lb. of Cu per ton of tailings

                not extracted by leach

60 gm H_2SO_4 per lb of ore = 264.3 lb. H_2SO_4/ton

$\dfrac{60}{454}$ x $\dfrac{x}{2000}$ X = $\dfrac{2000 \times 60}{454}$ = 264.3

May 23, 1967

300 lbs per ton = xGM per lb.

$\dfrac{300}{2000}$ = $\dfrac{x}{454}$ x = $\dfrac{(300)(454)}{2000}$ = 68.1 gm/lb

250 lbs. per ton = x gm per lb

$\dfrac{250}{2000}$ = $\dfrac{x}{454}$ $\dfrac{(250)(454)}{2000}$ = x = 56.75 gm/lb.

Prepared heads sample. Pulverized ore to # 100 mesh. The ore is not difficult to pulverize.

May 24, 1967

To prevent the forming of black oxides of copper in caustic leach the
leached copper is to be separated from the tailings on a schedule of 45 minute
leach periods, followed by filtration and a small wash. This is to be
continued as long as copper dissolves in sufficient amount, based on the
blue color.

Charge   1 lb. ore   60 gm NaOH   Temp. 180°F
open
jar   25 gm urea   300 ml water

60 gm NaOH in 300 ml water. 1 ml of sol. titr. $\approx$ 21.5 ml $\frac{N}{2}$ HCL

9.30   Leach begins   10.17   Leach ends

10.40   Second stage of leach begins
           same batch of ore
           60 gm NaOH   25 gm urea   300 ml water   open jar   Temp.180°F

           No evidence of caking in the leach solution. The pulp was stirred frequently

11.15   Third stage of leach begins
           same batch of ore
           25 gm urea   60 gm NaOH   250 ml. water   open jar Temp 170°F

           It is noticed the pulp is less bulky after the first stage and requires
           less water to form a dispersed pulp.

2.00PM   End 3rd stage

2.25   Begin 4th stage   same batch of ore
           25 gm urea   60 gm NaOH   200 ml water

3.10   End of stage 4
           In strong solution from stage #1 there was a definite deposit of copper oxide
           which must have formed after filtration.

           May 24, 1967

7.30 AM   If the copper is combined with the iron in the ore, the iron will inhibit
           caustic leach   and after the iron -copper combination is leached so that only
           the iron is in contact with the leach solution, then a caustic solution can
           not leach the copper. The same is true also of the alkaline earth metals
           present in the ore. Scrubbing will remove the iron better than it does the c,
           calcite so as to expose the copper to the caustic but a fine grind becomes
           increasingly necessary.

Strong solutions: after leach ends: caustic residual

Stage #1	1 ml titrated	12.7 ml $\frac{N}{2}$ HCL vol. of solution	$460^{m1} \pm$ #1
" 2	" "	4.75" " " " " "	646 ml #2
" 3	" "	6.00 " " "	510 " #3
" 4	" "	5.20 " " " " " "	590 ml #4

High reading of #1 due to copper hydroxide formed which consumed $\frac{N}{2}$ HCL after neutralization of residual caustic.

Heads analyzed for copper. titr = 7.0 ml hypo

$(.00522)(7)(1) = 0.03654$ mg Cu = 3.654% Cu in heads

Tailings (10gm) titr = 29.45 $(.00522)(29.45)(1/14)(...) = 5.801$gm Cu in Tails

1.547% Cu in tails

May 25, 1967
Thursday

Stages Solutions (5ml)

#1 = $(.00522)(14.5)(1/5)(460)$ = 6.9635 gm Cu

#2 " " $(1.5)(1/5)(645)$ = 1.01 gm Cu

#3 " " $(1.45)(1/5)(510)$ = 0.772 " "

#4 " " $(1.15)(1/5)(590)$ = 0.7 8 " "

9.45 gm

10.788
9.45
1.338

Total copper in 1 lb. Heads	=	16.589 6 gm		
" " " tailings	"	5.801	gm	
" " extracted		10.788		

Copper which formed oxide, dropped out of solution i. rst stage = 1.338 gm Copper in tailings = 34.9 % of total copper in ore. The high residue of copper in the tailings is due to the oxides of copper in the ore. A sample of the tailings from the last leach was washed free from slimes and examined by microscope. The residue is free from copper oxides such as have been common forming during caustic leach treatment on the sands , but there is a multitude of particles dark in color appearing to be iron and copper as oxides. It would possible to isolate a gram of this and analyse for copper, iron, sulphur content to confirm the opinion offered that this is the source of the failure of the caustic leach to extract the copper from this ore as completly as does the acid leach.

The stage process appears to have eliminated salting of the tailings by copper precipitated from solution.

May 26, 1967

Black particles were brittle or spongy. To about 2 gm of tailings in a dish were added several ml of conc. HNO_2 then some crystals of $KClO_3$. The Nitric acid turned pale green. Most of the black particles lost the black color. The iron did not dissolve in the nitric acid, but formed gelatinous , reddish brown cohesive residue units having shape similar to the particles before copper was removed. Since the iron does not dissolve, it is oxide , not sulfide When combined with the copper and does interfere with the leaching in caustic.

May 27, 1967   Saturday

Stage Leach May 28, 1967   Sunday

1 lb. ore   200 ml H_2O 20 gm urea   60 gm NaOH
Jar containing leach pulp is open, not capped

8.10 AM   **Start leach**   8.22 AM Temp. 160°F (temp. of pulp)

8.25   Pulp is well dispersed, not sanding out.

8.50   End leach

9.00   Second stage leach begins   Temp. 160°F
          Same oer, from 1st stage   200 ml H_2O 20 gm urea   60 gm caustic

10.00   End leach , second stage Temp. 166°F

10.17   Begin 3rd stage leach
          same ore   60 gm NaOH   150 ml H_2O 20 gm urea

11.47   end 3rd stage of leach

12.00   Begin 4th stage of leach   10 gm H_2SO_4 and 150 ml H_2O

2.00pm   End "   "   "   "

Weight of tailings 394 gms.

Stage #1 residual caustic titr = 5.2 ml $\frac{N}{2}$ HCL   Vol. 500 ml
  "   #2   "   "   "   "   6.5 "   "   "   470 "
  "   #3   "   "   "   "   5.6 "   "   "   545
  "   #4   "   acid   "   "   0.2 "   "   "   680 "

Smell of ammonia strongly perceptable from hot caustic leaches

	Copper titrations	sample 5ml	percent of copper	x volume	weight of Cu.
Sunday May 28, 1967	Stage #1	4.2	0.43848	500	2.1924 gm
	" #2	3.8	0.39672	470	1.8646 "
	" #3	3.5	0.3654	545	1.99143gm
	" #4	1.6	0.16704	680	1.135872 gm.

Total wt. of copper leached from ore 7.184302 gm
  "   "   "   "   in heads   16.58916 gm
                                     see 5/24/67

Ore tailings : (5gm) titr = 17.6 ml hypo
(.00522)$\frac{(17.6)}{5}$(394) = 7.2395136 gm Cu   14.42 gm Cu accounted for

May 29, 1967   Monday

1 lb. ore   60 gm NaOH   20 gm urea   150 ml water

8.10 AM   **Start leach**   pulp is well dispersed in 150 ml water   Temp. 160°F

9.25   **Added 50 ml water**

10.10   **End of first stage (2 hours)**

10.35   **Start 2nd stage of leach**

Temp. 160°F

same ore--- 200 ml water   20 gm urea   60 gm NaOH
sample from first leach: the solid blue particles are totally dissolved
in the leach rather than having the copper selectively removed no
skeleton remains

1.25   **caustic residue in leach solution =   9.25 ml $\frac{N}{2}$ HCL**

2.05   **End second stage of leach (3½hrs)** residual NaOH : 1 ml strong (5) = 4.9 ml$\frac{N}{2}$HCL

2.35   **Start 3rd stage of caustic leach**
           same ore   200 ml water   20 gm urea   60 gm NaOH
           4.25 PM residual NaOh (1 ml) in #3 stage   9.2 ml $\frac{N}{2}$ HCL

4.40   **End of 3rd leach**

540 ml #1 **Caustic residue in #1 stage strong sol = 4.8 ml $\frac{N}{2}$ HCL**
           60 gm NaOH dissolved in 200 ml water, 1 ml titrate 14.3 ml $\frac{N}{2}$ HCL

550 ml #2 **Caustic residue in #2 stage 1 ml strong sol =   4.9 ml " "**

730 ml #3   "   "   " #3 "#   " "   "   "   4.2 " " "

May 31, 1967   Wednesday

Weight of tailings to acid leach =   380 grams
add 100 ml water   40 gm H_2SO_4 =   176.2 lb/ton

8.30   **Start Acid leach   Temp. 160°F**

8.50   **Added 50 ml water** Pulp was a thick mud before the 50 ml of water was added.

9.00   **Examination** of pulp sample with microscope indicates the blue particles are
           completely dissolved. A few black particles remain in the sample

9.50   Very few(3 or 4) black particles remain in pulp sample. They do not
           soften in leach. Some of them shatter under needle probe.

10.00   **End of acid leach   800 ml of solution**   5 ml sample for assay
           Residual acid after dilution = 1.5 ml $\frac{N}{2}$ KOH
                    & washes

Wt. of acid tailings 315 gm	Leach Copper				Titration		X 1/5		Volume ml
	#1 = 5ml =	NaOH	=		4.7	=	.94	=	540 ml
	#2 " "	"	"		1.7	"	.34	"	550 "
	#3 " " " "	"		1.0	"	.20	"	730 "
	#4 " " " Acid	"		10.6	"	2.12	"	800 "
	#5 " 5gm " tails	"		1.5	"	0.30	"	325 GM

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May 31, 1967

DATA ON LEACH OF MAY, 31, 1967

#1. strong solution:(.00522)(0.94) = 0.49068 % x 540 = 2.65 gm Cu

#2. (0.00522)(0.34) = 0.17748 % x 550 = 0.97614 Gm CU

#3. " (0.20) " 0.1044 % x 730 = 0.76212 " "

Acid #4. " (2.12) " 1.10664 % x 800 = 8.85312 " "

Tailings #5 " (0.3) " 0.1566 % x 325 " 0.50895 " "
Sand

Thursday   Above copper accounted for ----------- 13.75033 gm Cu

June 1,   Copper in heads = 3.654 % or 16.589 gm. Cu per lb. ore
1967   copper recovered from filter papers: add to above Cu acctd, for
           (.00522)(14.3 x 130) = 1.941 gm Cu plus 13.7503 = 15.6913 gm Cu total gm.
                        5

total caustic used equivalent to   792.95 lbs/ton ore
Total Copper extracted by caustic   19.31 lbs/ton
Total "   "   " Acid   39.00 "   "
Contact time for caustic leach   7 hours
  "   "   " Acid "   1½ hours
Total copper in ore 3.664 % equivalent   73.08 lbs/ton
Copper in tailings (0.1566 %) equal to   3.132 "   "
Sulphuric acid per ton of ore   176.2 lbs.
copper extracted from filter papers   8.54 lb/ton
residual acid after acid leach   129.5 lbs
sulphuric acid consumed in leach   46.7 lb/ton

consumption of caustic in 3 stages
urea plus NaOH residual as NaOH:

#1. strong sol. = (.020)(4.8)(540) = 51.84 gm (from 60 g NaOH plus 20 g. urea)

#2 "   "   " (.02)(4.9)(550) " 53.90 "   "   "   "   "   "   "   "

#3. "   "   " (.02)(4.2)(730) " 61.32 gm   "   "   "   "   "   "

June 2, 1967

Mr. Joseph M. Reynolds.

Dear Joe:

The test made on May 29th enabled me to determine the cause of the failure of the caustic leach to make a more complete extraction of the copper.

In this test I was able to control the forming of the black oxides of copper or rather, I was able to prevent any black oxide from forming during the leaching. This left only that part of the copper in the ore which the caustic had failed to dissolve. The caustic dissolved only 19.3 lbs. of copper from a total of 73.08 lbs per ton.

Examination of the Tailings from the caustic treatment identified the cause of the failure. In this ore very minute particles of copper and iron, as oxides have formed nodules which the caustic solution does not penetrate. The iron acts as an insoluble shield around the copper-bearing crystals, preventing contact of the caustic with the copper. Some of the particles are soft like mud-balls, others are hard and shatter under pressure of the needle probe.

This information greatly simplifies the problem. The caustic leach will be as effective in extracting the copper as the acid leach now is effective when these particles are opened up to the leach solution. It is at this point that our lack of communication is a hinderance. A scrubbing method would pulp most of these particles. I used 40 grams of Sulphuric acid per pound of ore, equivalent to 176 lbs. of acid per ton of ore to reduce the copper in the tailings to the amount reported. Since you have already considered a scrubbing treatment, the alternative of acid treatment may be of small importance.

It now appeares the caustic leach has been successful as far as it could go. The use of urea is very helpful, and I consider it to be our discovery. While making tests in a sealed glass jar, I was able to prevent the loss of ammonia formed in the urea. Upon breaking the seal on the jar, the pulp would begin to foam as the compressed or absorbed ammonia rose to the top to escape. Copper concentration in the caustic strong solution in proportion to the amount of ammonia (urea) present has much to do with the forming of the black oxides of copper. In the leach of May 27th, a large deposit of copper oxides formed after the pulp was filtered. I was fortunate to get the solution filtered before they formed.