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Concentration of ores by flotation

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CONCENTRATION OF ORES BY FLOTATION.

By

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and

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A

T H E S I S

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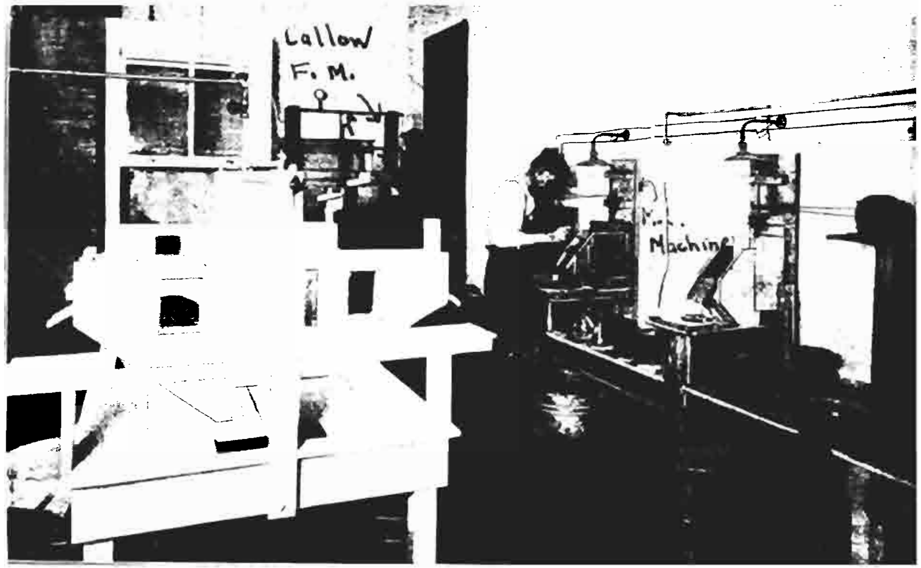
Approved by Horace T. Mann

Associate Professor of Metallurgy.

T A B L E O F C O N T E N T S .


Callow
F. M.
Machine

Flotation is a new step in the separation of valuable minerals from gangue. This process has only been in commercial use for about six years. There has been several theories advanced as to how the ore is concentrated, and the theory that is now usually accepted is that the oil covers or wets the sulphide mineral particle and does not the gangue; and this mineral particle, by means of induced aid is concentrated on the surface.

The authors of this thesis have not attempted to explain the reason or reasons why minerals will float; but have accepted the theories advanced by men far more capable, but we have worked on the assumption that sphalerite, the sulphide of zinc, according to all flotation theory will float, so our problem narrowed down to one fact which can be stated in the one work-How.

The subject of the thesis clearly states our problem. We chose a barite plus sphalerite aggregate because of the large amount of this mineral developed and undeveloped, in the resource of the United States.

The second part of the thesis was so chosen because of the wide field in the Joplin district for flotation. The ore used was donated by Mr.F.C.Wallower from his Cumberland Mine no.I located in Carterville, Missouri. The Cumberland mill is new and absolutely modern in every particular, thereby giving us a commercial feed for our work.

Regardless of methods employed our one purpose was to get a marketable concentrate by flotation, and to improve this concentrate in quality and quantity where-ever possible by means of reagents. The experiments were made in a standard experimental numeral separation machine, as shown by illustration on page _____ and the standard Callon air flotation experimental machine as sold by the General Engineering Company of Salt Lake City, Utah, also shown in illustration. Both machines are protected by patents. The water used was the deep well water of the University which analyzes as follows:

Deep Well Water

Phelps County water analysis

	parts per million
Co_2 free	1.4
Co_2 (bound) per L	111.7
CoO	73.1
MgO	45.1
So_2-Fe_2O_3.Al_2O_3	12.2
Cl	4.1
So_3	30
total solids	298.

2/18/'15

Analyst

V.H.Gottschalk.

The oils, reagents and dilutions are best described in the descriptions of the actual tests. From this point on we will treat the subject as two separate and distinct thesis in the following order:-

Separation of ZnS from a sphalerite aggregate.

The mineral is of synthetic composition made in the University labratories. The barite and sphalerite were crushed to pass through Sixty Five mesh and then intimately mixed. The assay on this composition was

22.5% Zn

11.25% S. (calculated)

66.25% $BaSO_4$ (By difference)

all weights of feed are given as dry weight.

The flotation tests were made according to the following outline:

(1) In basic solution.

(2) In basic solution with sodium resonate

(3) In acid solution with different flotation oils.

(4) By saturating feed with hydrogen sulphide

(5) By first floating the barite

(6) By roasting the feed.

RUN I Mineral Separation Machine

800 gm. feed 3200 gm. water 3 C.C.
Sulphuric Acid (H_2SO_4)

No concentration.

A heavy abundant froth which carried the flotation feed.

Run I "B" Mineral Separation Machine

Same charge as Run No.I

No Concentration

Acidified the pulp in order to check the sodium resonate, but the entire froth disappeared.

RUN II Mineral Separation Machine

200 gm. feed 3800 gm. water .2 C.C. H_2SO_4
20cc sodium resonate solution.

No concentration

Yellow thick froth which was not capious. The addition of 10cc more of sodium resonate solution did not affect the result.

RUN III Mineral Separation Machine

200 gm. feed 3800 gm. water 1.44 lbs. per ton of ore of number 56 flotation oil.

No concentration.

Froth was very capious, and by macroscopic examination the barite was thé principle mineral.

RUN IV Mineral Separation Machine.

200 gm. feed 400 gm. sodium resonate solution 3200 gm. water 5 cc H_2SO_4.

No concentration.

The supposidly concentrate contained more zinc than test previous but not in sufficient quantity to call it a zinc concentrate.

RUN V Mineral Separation Machine.

200 gm. feed 1.22 lbs. No.17 flotation oil per ton of ore 3800 gm.water 5 cc of 5% Patassium alum solution.

No concentration

No froth

RUN VI Mineral Separtion Machine

200 gm. feed 1000 gm. sodium resonate solution
2800 gm. water 2 cc H_2SO_4.

No concentration.

Added the sulphuric acid first and after five minutes running the sodium resonate solution was added. The froth seems to carry more sphalerite than previous froths.

RUNS VII-VIII-IX Mineral Separation Machine.

Same charges as No.6 but using 200-600-800 gm. of sodium resonate, respectively;

No concentration.

The different quantities of sodium resonate solution did not affect the froth, nor it's carrying qualities.

Note: All previous tests were made to determine the value of the sodium resonate solution. It was found to give a good froth, but a froth that is too heavy to float zinc; therefore the froth products; known as the concentrate, will contain to high a percentage of barite.

In commercial practice the sodium resonate solution could be reused by treating the froth with a two per cent sodium hydroxide solution. The sodium hydroxide will dissolve the dosium resonate and the resulting solution is ready to be re-used.

RUN X Mineral Separation Machine.

400 gm. feed 3600 gm. water 10 gm. commercial sodium hydroxide (N_aOH) .7 lbs. No.20 flotation oil per ton of dry feed. H_2SO_4 to neutralize pulp and give 1 c.c. excess.

51 gm. concentrate assaying 54.9% Z_n. 25% extraction.

The first five minutes of agitation were made in a alkaline solution and then sulphuric acid was added to neutralize the pulp, and 1 c.c. excess. On the addition of sulphuric acid a ephemeral froth formed.

RUN XI Mineral Separation Machine.

200 gm. feed 3800 gm. water 20 c.c. of a 2% sodium hydroxide solution. H_2SO_4 to neutralize + 2 c.c. excess. .7 lbs. No.20 flotation oil per ton of dry feed.

22 gm. concentrate assaying 41% Zn. 16.1% extraction.

Ephemeral froth. The sphalerite came over within five minutes after the run was started. Additions of more oil probably would have raised our extraction but we failed to add the oil.

RUN XII Mineral Separation Machine.

800 gm. feed 400 c.c. of a 2% sodium hydroxide
2800 gm. water .7 lb. of No. 20 flotation oil per ton of dry feed.

No concentration.

The froth carried very little zinc.

RUN XIII Mineral Separation Machine.

Repeated run No.12 with same result.

Note: We have been working on the assumption that the pulp must be in a alkaline solution to float the sphalerite from the barite; but on our particular feed it seems that an acid solution is necessary.

RUN XIV Callow Flotation Machine.

600 gm. feed .48 lbs. No. 45 flotation oil per ton of feed per dry ton feed. 5cc sodium resonate solution 5400 gm. water saturated with hydrogen sulphide.

No concentration.

The froth was very copious but did not carry very much sphalerite.

RUN XV Callow Flotation Mchine.

600 gm. feed 4 lb. No.17 flotation oil and 4 lb. No. 24 flotation oil per ton of dry feed 10cc sodium hydrox solution 4cc H_2SO_4

No concentration.

Run five minutes in alkaline solution and then acidulated. Ephemeral froth consisting mostly of barite.

RUN XVI Callow Flotation Machine.

600 gm. feed 148 lb. crysilic acid per ton of dry feed 5c.c. H_2SO_4 5400 gm. water.

No concentration.

Froth was a good ephemeral froth sphalerite; but it failed to carry an appreciable amount of sphalerite.

RUN XVII Callow Flotation Machine.

600 gm. feed 1.66 lbs. No.21 flotation oil per ton of dry feed 5400 gm. water.

No concentration.

A very copious froth-

RUN-XVIII Callow Flotation Machine

600 gm. feed .48 lbs. No.17 flotation oil per ton of dry feed 40 c.c. 10% sodium sulphide solution 5 c c H_2SO_4.

No concentration.

A very heavy copious froth. The froths on the callow flotation machine seemed to carry the flotation feed just as it entered the pachuca mixer.

RUN XIX Callow Flotation Machine.

Repeated run number 18 using flotation oil No. 54 instead of No. 17, same result.

No concentration.

RUN XX Callow Flotation Machine.

Repeated run number 19 using flotation oil No. 45 in place of No. 54. Same results.

No. concentration.

RUN XXI Mineral Separation Machine

500 gm. feed 3500 gm. of 1% sodium hydroxide solution..

No concentration.

The above charge was run for fifteen minutes and no froth ever formed in the spitz- ka sten.

RUN XXII Mineral Separation Machine

200 gm. ore with 800 gm. water were saturated with hydrogen sulphide (H_2S) 5c.c . 2% sodium resonate solution 3000 gm. water 1 c.c. H_2SO_4 1.4 lbs. No. 45 flotation oil per ton feed.

31.5 gm. concentrate assayed 54.2% Z_n 37.8% extraction.

The pulp was agitated for five minutes before the acid and oil were added. The froth was heavy until the acid was added and then it became very ephemeral and laden with sphalerite.

RUN XXIII Mineral Separation Machine.

Repeated run number Twenty-Two (22).

28.5 gm. concentrate assayed 58.7% Z_n. 37.8% extraction.

The first twelve experiments dealt principally with sodium resonate, and whether the tests, or pulp, should be in acid or basic solution. In the experiments from twelve to twenty-three we tried to get a concentrate with different oils, both in acid and base. The hydrogen sulphide tests as described in runs twenty-three and twenty-four are the first commercial concentrates obtained.

Mr.M.H.Thornberry in charge of the University experimental labratory, suggested that we try to float the barite and then the zinc. Acting upon his suggestion, we made the following experiments:

RUN XXIV Mineral Separation Machine

200 gm. feed 1 c.c. of a 5% of a Ben-Hur soap solution 3800 gm. water.

Agitated for thirty minutes during which time a heavy copious froth, carrying very little sphalerite, overflowed from the spitz-kasten. The tailings from this experiment were given the following treatment.

Tails 3500 gm.water 1.44 lbs. No. 17 flotation oil.

No concentration.

Twenty grams of concentrates were extracted on this experiment, and were examined macroscopically. The concentrate appeared to be around 32 ± % Zinc which would be very poor.

RUN XXV Mineral Separation Machine

Repeated run number twenty-four using 1.5 cc of a 5% "Fels-Naptha" soap solution.

No concentration.

RUN XXVI Mineral Separation Machine.

Repeated run number twenty-four using 2.2 c.c. of "German-Family" soap.

Concentrate assayed 35% zinc.
Tailings assayed 20% zinc.
The weight of the concentrate was never recorded, nevertheless the zinc contents of the tailing is sufficient to prove the un-commercial value of the experiment.

Ephemeral froth.

RUN XXVII Mineral Separation machine.

500 gm. feed were roasted with a blast lamp for fifteen minutes until the barite began to turn a light red brown in color. The feed was put into the flotation machine hot.

3500 gm. water 1.14 lbs. No.15 flotation oil per ton of dry feed .3c.c. of a 10% Na_2S solution

3 cc H_2SO_4 1 cc sodium resonate solution.

53 gm. concentrate assayed 60.4 % Zn. 28.5% extraction.

400 gm. tailing assayed 5.% Zn 17.85% Zn. in tailing.

.57 lbs. of No.15 oil and the sodium resonate solution were added as soon as we started the pulp to agitating. A copious froth overflowed the spitz-kasten which seemed to contain very little sphalerite. After thirty minutes the charge was acidified with 3 cc H_2SO_4 and .57 lbs. of No.15 flotation oil added.

Ephemeral froth formed heavily laden with sphalerite.

RUN XXVIII Mineral Separation Machine.

430 gm. feed roasted twenty minutes over blast lamp. The test was made identical as run number twenty-seven.

31 gm. concentrate assayed 58.8% Zn. 19.3% extraction.

51 gm. barite concentrate assayed 7.30% Zn. 3.86 % of the total Zn contained was in the barite concentrate.

RUN XXIX Mineral Separation Machine.

500 gm. feed which has had a chloridizing roast for three hours. Experiment made by same procedure described in run twenty-seven.

No froth or concentration.

RUN XXX Mineral Separation Machine.

Same as run number twenty-nine except that the agitation was made in a 2% Na_2S solution.

No concentration.

From the above tests it is quite evident that the authors did not separate sphalerite from barite from a commercial viewpoint; but we do believe that these experiments pave the way for future experiments along two lines.

(1). Saturating the flotation feed with hydrogen sulphide, as described in runs twenty-two and twenty-three; and then experiment with different oils and reagents.

(2). To roast the feed, as described in runs twenty-seven and twenty-eight and experiment with different oils and reagents.

It is the belief of the authors that the second method could be put on a commercial basis. We have made our deduction from the following conditions.

(1) A barite-sphalerite aggregate usually assays 12 ± % Zn.

(2) The ore body would have to be of sufficient size to pay for installing the plant.

(3) One long hand reverbatory furnace would have an enormous capacity.

P A R T II.

The second part of this thesis deals with flotation experiments made on the Cumberland Mine No.1 sludge pond ore.

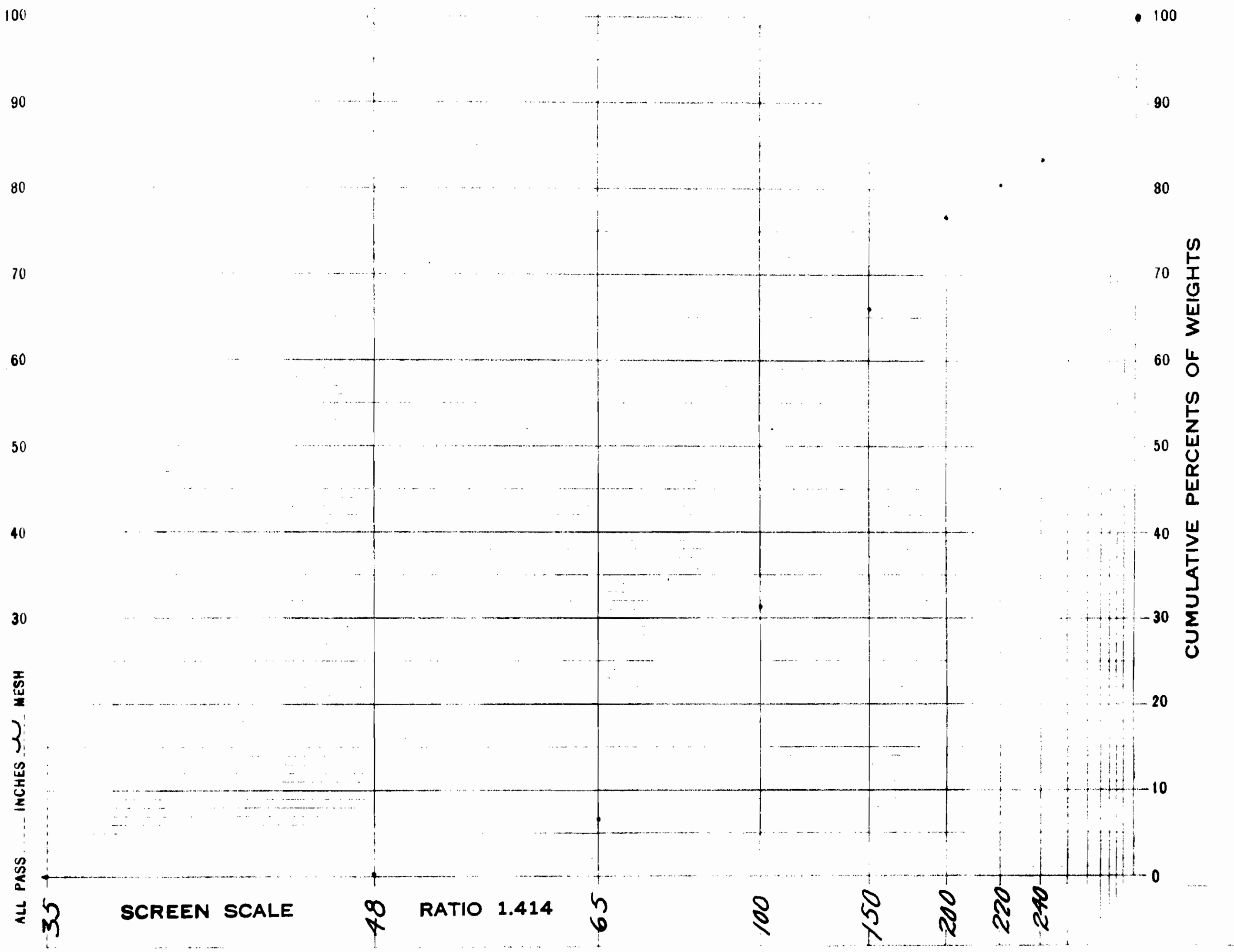
The screen analysis of this ore is on the accompanying "Tyler Standard Screen Scale paper.

The products retained on the different meshes were examined under a microscope and of the material retained on 65 mesh, a small percentage of the sphalerite was not broken free from the gangue.

The Tyler Standard Screen Scale

Cumulative Direct Diagram of Screen Analysis on Sample of *Cumberland Sludge Pond Ore.*

Name *J.G. Reilly and F.S. Elfred* Date *May 2, 1917.*



...cate the Screen ...ushed through ...nd also First Retaining Screen	SCREEN SCALE RATIO 1.414 Openings Inches	Milli-meters	Mesh	Diameter Wire Inches	WEIGHTS Sample Weights	Per Cent	Per Cent Cumulative Weights	ASSAYS % Zn.	CONTENTS	% of Total Conten...
mesh	1.050	26.67		.149	*in gm.*					
	.742	18.85		.135						*Screen*
35	.525	13.33		.105						*Analysis* } *7*
	.371	9.423		.092						*Average* } *1,*
48	.263	6.680	3	.070	*.40*	*.20*				*Assay* } *2.*
	.185	4.699	4	.065						
65	.131	3.327	6	.036	*10.10*	*6.50*	*6.70*			
	.093	2.362	8	.032						
10	.065	1.651	10	.035	*38.00*	*24.40*	*31.20*	*1.3*		
	.046	1.168	14	.025						
50	.0328	.833	20	.0172	*45.50*	*29.40*	*60.60*	*3.0*		
	.0232	.589	28	.0125						
20	.0164	.417	35	.0122	*25.20*	*16.10*	*76.70*	*7.6*		
	.0116	.295	48	.0092						
20	.0082	.208	65	.0072	*6.00*	*3.70*	*80.40*	*11.00*		
	.0058	.147	100	.0042						
40	.0041	.104	150	.0026	*4.50*	*2.90*	*83.30*	*14.25*		
	.0029	.074	200	.0021						
40	.0029	.074	200	.0021	*26.00*	*16.80*	*100.00*	*15.5*		
				Totals,	*155.70*	*100.00*				

In the following runs all weights of oil and sulphuric acid are given in pounds used per ton of dry ore feed.

RUN XXXI Mineral Separation Machine.

350 gm. ore .8 lbs. flotation oil No. 15 .26 lbs. H_2SO_4 3650 gm. water.

No concentration

Ephemeral froth. The froth only lasted five minutes and the addition of more oil did not return the froth. By adding ten pounds of sulphuric acid the froth re-appeared.

RUN XXXII Mineral Separation Machine.

350 gm. ore 5.1 lbs. H_2SO_4 .3 c.c. 10% sodium resonate solution. .6 lbs. cresylic acid 3500 gm. water.

50 gm. concentrate assayed 55% Zn. extraction 63.75%

A good copious froth during entire run; heavily laden with sphalerite. The acid was added at different intervals until the entire amount was used.

RUN XXXIII Mineral Separation Machine

350 gm. ore 40 lbs. H_2SO_4; 5 c.c. of a 5% $CuSO_4$ solution .6 lbs. cresylic acid 3500 gm. water.

29.5 gm. concentrate assayed 57% Zn. 63.9% extraction.

A good sphalerite froth. Froth grades between the heavy froth and the ephemeral froth, and is an ideal froth for sphalerite.

RUN XXXIV Mineral Separation Machine.

331 gm. ore in 1000 gms. of a 5% H_2SO_4 solution was agitated or emulsified, on rolls for two hours. added 2000 gm. water 3 c.c. of a 10% sodium resonate solution. 157 lbs. cresylic acid.

24 gm. concentrate assayed 46.6 % Zn 44.5% extraction.

A copious froth. After ten minutes the flotation machi machine broke, therefore the above results are on the ten minutes run.

RUN XXXV Mineral Separation Machine.

372 gm. ore 32 gm. water 20 lbs. H_2SO_4 .55 lb. cresylic acid.

63 gm. concentrate assayed 56.4% Zn 65.5% extraction.

A good sphalerite froth as described in run number thirty-three.

RUN XXXVI Mineral Separation Machine

380 gm. ore 34 lbs. H_2SO_4 3500 gm. water .74 lbs. No. 56 flotation oil.

38 gm. concentrate assayed 48.4% Zn. extraction 65.5%

On the above charge we did not get a froth. Upon adding two cubic centimeters of a ten per cent sodium resonate solution a heavy froth overflowed at the discharge spout of the spitz-kasten.

RUN XXXVII Mineral Separation Machine.

Repeated run thirty-six using 800 gm. ore. The experimental machine will not handle this amount of our ore and float a concentrate. The pulp is to thick.

No concentration.

RUN XXXVIII Mineral Separation Machine

350 gm. ore 3500 gm. water, 14 lbs. H_2SO_4
1.42 lbs. turpentine, Na (OH) commercial

55 gm. concentrate assayed 31.8% Zn 70% extraction.

Charge was run in acid solution for five minutes and then make alkaline with sodium hydroxide. After it was made alkaline . 47 lbs. of turpentine was added every fifteen minutes until One and forty-three hundredths (1.43) pounds were used. The froth was four inches thick, white in color, and easy to break down.

RUN XXXIX Mineral Separation Machine.

Repeated run thirty-eight with same results.

We believe, from panning this concentrate, that it would make a practical feed for a recleaning table.

RUN XL Mineral Separation Machine

372 gm. ore 3500 gm. water 29 lbs. (3 c.c) H_2SO_4 .67 lbs. No. 6 flotation oil .3 lbs. No.56 flotation oil. 7.55 lbs. gasoline .88 lbs. Cleaveland Cliff flotat ion oil No. 2

36 gm. concentrate assayed 52.9% Zn. 56% extraction.

The ephemeral froth was unstable until we added the Cleaveland Cliff oil number two.

RUN XLI Callow Flotation Machine

372 gm. ore 5500 gm. water .67 lbs. No. 6 flotation oil 29 lbs. H_2SO_4 26.4 lbs. gasoline.

35.5 gm. concentrate assayed 50.1% Zn. -------- 65.8% extraction. 11 gm. middlings assayed 27.5% Zn. 10.9% extraction. total extract 76.7%

Ephemeral froth. The concentrate was taken from this machine for the first forty-five minutes and the pulp contained 18.9 pounds of gasoline. At the end of the foty-five minutes seven and five tenths (7.5) pounds of gasoline was added, and it caused more sphalerite to be carried in the froth; but we noticed that this second product was rather dirty so we classified it as a middling.

RUN XLII Callow Flotation Machine

742 gm. ore 5000 gm. water 20.2 lbs. gasoline flotation oil same as in run forty-one.

59 gm. concentrate assayed 53.2% Zn. 57.5% extract.

25 gm. middlings assayed 40.6% Zn. 18.5% extract

Total extraction 76. %

Experiment made the same as run number forty-one. Sixteen pounds gasoline used on the concentrate and Four and two tenths (4.2) pounds on the middlings.

RUN XLIII Callow Flotation Machine

558 gm. ore 5500 gm. water 28 lbs. gasoline

8 lbs. No. 6 flotation oil 1. lb. Cleaveland Cliff No. 1.

45 gm. concentrate assayed 30.2 % Zn. 33% extraction

19 gm. middlings assayed 36.6% Zn. 16.65% extraction.

Total extraction 49.25%

RUN XLIV Mineral Separation Machine.

372 gm. ore 3500 gm. water 8 lbs. No. 6 flotation oil 7.5 lbs. gasoline.

49.5 gm. concentrate assayed 31% Zn. 56% extraction

RUN XLV Mineral Separation Machine

372 gm. ore 3500 gm. water 18.8 lbs. gasoline .88 lbs. Cleaveland Cliff flotation oil No. I .75 lbs flotation oil No. 6

46:5 gm. concentrate assayed 34.6% Zn. 58.4% extraction.

Ephemeral froth. After running for forty-five minutes another 10. lbs. of gasoline were added with the following results:-

14 gm. concentrate assayed 36.8% Zn. 18.75 extraction. Total extraction on charge 77.15 %

RUN XLVI Mineral Separation Machine

372 gm. ore 3500 gm. water 18.lbs. coal-oil .75 lbs. flotation oil No. 6

47.5 gm. concentrate assayed 41% Zn. 70.8% extraction.

Ephemeral froth.

RUN XLVII Mineral Separation Machine.

372 gm. ore .88 lbs. Cleaveland Cliff flotation oil No.1 20.2 lbs. coal oil 3500 gm. water .75 lbs. flotation oil No. 6

27 gm. concentrate assayed 51% Zn. 50.1% extraction.

On running the above charge the Cleaveland Cliff oil number one was added first, and only a few bubbles saturated with zinc sulphie appeared. The flotation oil number six was added and the ephemeral froth appeared immediately.

RUN XLVIII Mineral Separation Machine

372 gm. ore 3500 gm. water 18.8 lbs. coal oil 2.1 lbs Cleaveland Cliff flotation oil number 2.

76.5 gm. concentrate assayed 24.4% Zn. 62.8% extraction.

A very heavy copious froth.

RUN XLIX Mineral Separation Machine

372 gm. ore, 3500 gm. H_2O 5 c c coal oil 2.1 lbs. Cleaveland Cliff No.2

Heavy copios froth.

76.5 gm. concentrate assaying 24.4% Zn. = 62.8% extraction.

Actual running time of the above experiments were forty-five minutes.

In examining the above results on the Cumberland ore it is noticeable that regardless to the reagents and oils used that the best concentrates range from 50% to 57% in zinc contents, and their extraction varies from 60% to 70%.

It is the opinion of the authors that if a recleaning plant is installed the extraction could be raised, materially without lowering the metal content of the concentrate, and may be successfully handled to even raise the precentage of zinc in the concentrate.

We are indebted to Mr.M.H.Thornberry for suggesting the use of gasoline and coal oil. The use of these two crude oil products is no doubt revealed the important point of our thesis because.

1st. It has eliminated the use of acid, thereby lowering the cost.

2nd. The concentrate made is very clean; and would have been higher in zinc has closer attention been paid to the machine.

We were unfortunate in making our experiments using gasoline and coal oil in the last week of our experimental work, otherwise we would have conducted considerable research on these two oils.

il No.

" 6----No.8 flotation oil (Pine oil)

General Naval Stores

" 15----Pine oil No. 6 (light)
General Naval Stores.

" 17----Flotation oil 800--

Pensacola Tar and Turpentine Company

" 20----No.15 special rosin

Pensacola Tar and Turpentine Company

" 21----No.75 crude wood turpentine

Pensacola Tar and Turpentine Company

" 45----Rosin oil

Eimer and Amend

" 54----Cresylic acid 95%

Meyer Brother Drug Company

" 56----Creosote oil 20%

Meyer Brothers Drug Company

All cresylic acid 95% pure

Sodium resonate solution used were always of two per cent strength unless otherwise specified.