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FLOTATION TESTS ON FLUORITE ORE
FROM LEMHI COUNTY, IDAHO

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INTRODUCTION

Due to the expansion in steel mill operation, and the large increase in demand for hydrofluoric acid (essential in the manufacture of artificial cryolite for the production of aluminum), increased demands have been made on the fluorspar industry. Domestic production for 1940 (1) was second highest in the history of the industry and, although figures are not available since then, there is every reason to believe that there will be no decrease, at least for the duration of the war. Present stocks of fluorspar are at a low level, and in order to stimulate production, the government has recently permitted a price increase on crude ore (2).

Deposits of fluorspar have been located in Lemhi County, Idaho, (3) which show promise of becoming commercial. Since these will require milling to be brought up to the specifications of the industry, it was thought desirable to conduct an investigation of the possibility of concentrating these by flotation. The object of the testing program was to determine the grade of the concentrate, and the percentage recovery that could be expected from a milling operation.

The specifications for the three commercial grades of fluorspar are given in the following table:

TABLE I. THE CHIEF COMMERCIAL GRADES OF FLUORSPAR

Name	Chief Use	Form	Specification %		
			CaF ₂ Minimum	SiO ₂ Maximum	Fe ₂ O ₃ Maximum
Metallurgical	B. O. H. Steel	Washed gravel, less than 1 inch & not over 15% fines	85	5	--
Ceramic	Glass and Enamel	Ground: Coarse, fine, & extra fine	95	3	0.12
Acid	Hydrofluoric acid	Lump, gravel, and ground	98	1	--

Although the steel industry consumes approximately 70 per cent of the total production, under normal conditions there is a sufficient supply of this grade of material near the large centers of steel production. The supply of acid

grade spar, however, is much more limited and there is, consequently a greater demand for it. The concentrate from a milling operation, being finely divided, would be acceptable for the manufacture of hydrofluoric acid, but could not be used in the steel furnace without briquetting or similar treatment. For these reasons and also because acid spar commands a higher price than fluxing material, the desirability of producing a concentrate of this grade can be readily seen.

The writer wishes to acknowledge the work of Mr. Clarence Zeuch, Analyst, Idaho Bureau of Mines and Geology, for the assays made on the samples from the various tests. Also to Mr. A. W. Fahrenwald, Director, Idaho Bureau of Mines and Geology, for his many suggestions and help in conducting this work.

GENERAL DESCRIPTION OF SAMPLE

A sample of approximately 100 pounds of ore was sent to the laboratory by A. L. Anderson for milling tests in conjunction with work on the geology of the district. In general appearance, the material is well crystallized and has a vitreous luster. Pieces ranged in size from 6 inches down to fines. Freshly broken surfaces are a pale green color, although the older faces are stained a brownish-yellow by iron oxide. The sample carries only a small amount of calcium carbonate; the chief gangue material being quartz and other silicate minerals. Anderson reports the presence of barite (3) in some of the deposits of the district, but no appreciable amount was found in this particular sample. The specific gravity of a hand-picked specimen was found to be 3.2. The head sample assayed as follows:

CaCO_3 = 1.85%

SiO_2 = 20.6%

CaF_2 = 74.2%

TESTING PROCEDURE

Temperature of the Pulp

Preliminary tests were run to determine the effect of temperature variations in the flotation pulp. At 20° C., fluorite can be collected with oleic acid, but the action is quite sluggish. Long conditioning periods are needed at this temperature, indicating that the chemical reaction at the mineral surfaces proceeds slowly. Heating to 30° C. noticeably increases the rate of flotation and also decreases the amount of oleic acid required. Heating still higher to 45° C. again increases the amount of mineral which can be collected. Since satisfactory results can be obtained at 30° C., however, it was decided that all following tests would be made at this temperature. In practice, heating to 45° C. would be decidedly uneconomical and also more gangue material would be collected by the oleic acid.

The following table gives the amount of concentrate collected from 500-gram head samples at various temperatures.

TABLE II. EFFECT OF PULP TEMPERATURE ON FLUORITE FLOTATION

Test No.	Temperature of Pulp	Amount of Oleic Acid	Weight of Concentrate Collected
1	19° C.	10 drops	267 gram
2A	30° C.	7 drops	278 gram
2	45° C.	7 drops	338 gram

Note: 1 drop of Oleic acid = 11.5 mg. approx. - varies with room temperature

Variations in pH of Pulp

A series of tests was also made to determine the effect of varying the pH of the pulp. The same amount of sodium silicate was used in each test, and the pH was controlled by adding sodium carbonate or sulfuric acid. With no modifying reagent, the pH was 7.9 and fluorite was readily floated. By adding successive amounts of sodium carbonate, no appreciable change in results could be noted up to a pH of 9.5. Sodium carbonate disperses the slimes, however, and approximately 1 lb. per ton was added to all following tests for this reason.

One test was run at a pH of 7.2 (1.8 lb. of H₂SO₄ per ton) in which the mineral was readily floated. Using 4 lbs. of sulfuric acid per ton of ore lowered the pH to 5.0, and no mineral could be collected under these conditions.

Effect of Sodium Silicate

Since the bulk of the material to be rejected in the tailing is silica, the effect of adding increasing amounts of sodium silicate was tested. No appreciable decrease in the silica content of the concentrate was noted in the tests in which a large excess was used. Satisfactory results were obtained, using approximately 0.5 lbs. per ton of ore in the rougher circuit. It was also found desirable to add small amounts to the cleaner and recleaner circuit.

Grinding

If the silica content of the concentrate is to be kept below 1 per cent SiO₂, almost perfect liberation of the particles must be accomplished. With this in mind, it can be seen that proper grinding is the most important factor in the problem. Since the sample contains only a very small amount of calcium carbonate gangue and a large amount of silica, once the minerals are free, their separation can be readily accomplished by flotation.

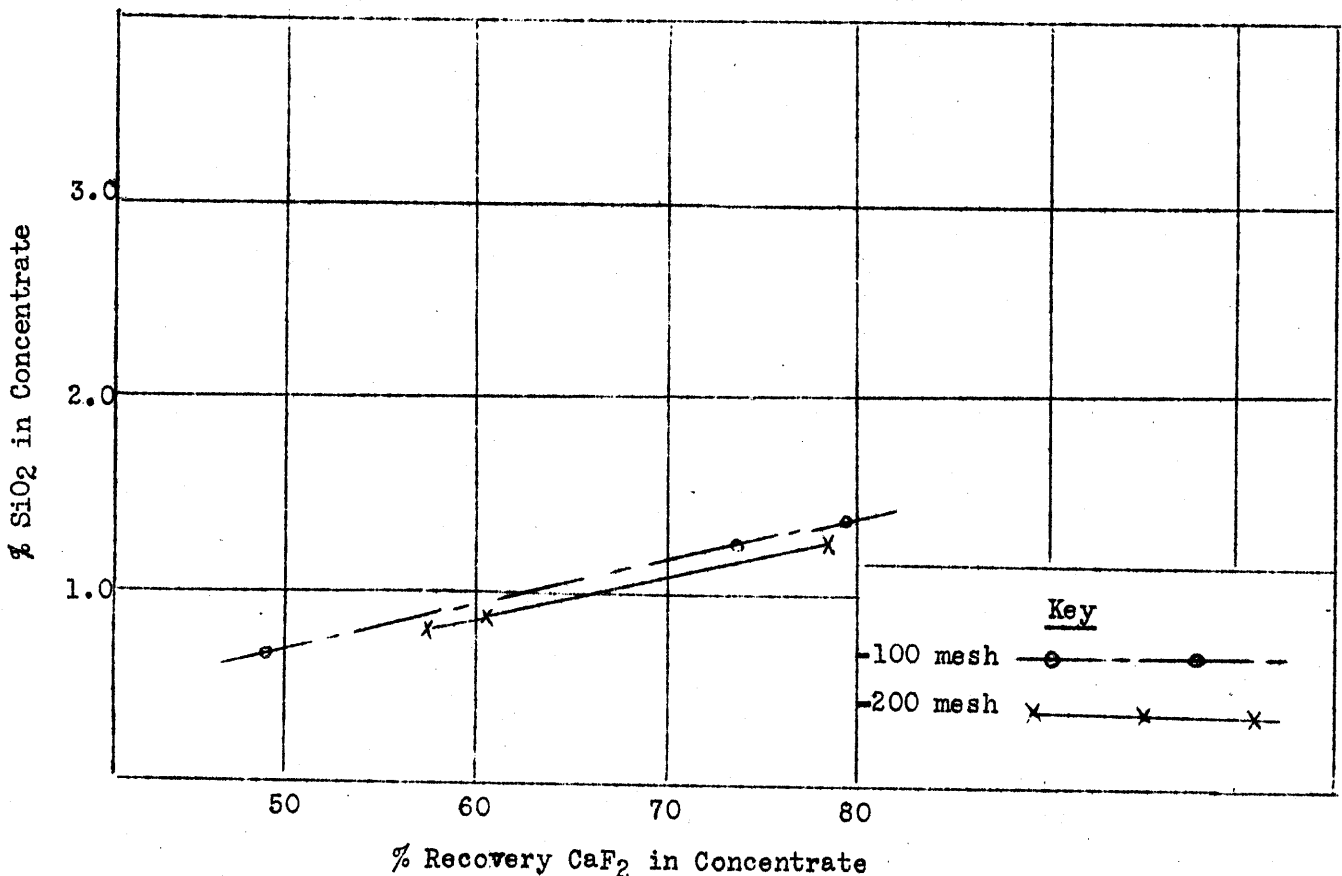
For most of the tests, the grinding was done in 2000-gram batches in the ball mill, and in order to produce a flotation head sample in which the coarse particles were liberated, a large amount of over-grinding was done on the fine material. The grinding time was varied between tests; the final series was made from a batch which was 0.5% plus 65 mesh and 80.5% minus 200 mesh. The large amount of slimes did not appear to lower the grade of the concentrate,

as might be expected. In two tests, desliming before flotation was tried with no improvement over those in which the ball mill product was fed directly to the flotation machine.

In order to more nearly approach a classified feed and to establish the grind required, three groups of tests were made, in which the head samples were stage ground to the desired size. These were made so that all particles passed through 65 mesh, 100 mesh, and 200 mesh, respectively. For each series, the desired undersize was screened out before any grinding was done. The oversize was ground in the ball mill for a short time, after which the undersize was again removed and the oversize was returned for another grinding period. This was repeated until all the material passed through the desired screen. Acid grade spar was made from both the minus 100 mesh and the minus 200 mesh products.

Plotting the percent SiO_2 in the concentrate against the CaF_2 recovery in the concentrate (Fig. I) shows that the results are almost the same for the minus 100 mesh feed as for the minus 200 mesh. These plots indicate that a recovery of approximately 63 per cent of the CaF_2 can be expected in a concentrate assaying 1 per cent SiO_2 . In practice, the return of middling products to the circuit should materially improve this.

FIGURE I.
Comparison of Flotation Results From
Minus 100 Mesh and Minus 200 Mesh Products



In all tests made on the 65 mesh product, much more mineral was floated than with the same reagents on the finer material. This gave high recoveries on the fluorite, but in all cases, the silica content was above the limit for acid grade spar.

A comparison of the various grinds is given in the following table:

TABLE III.- SIZING ANALYSIS OF BALL MILL PRODUCTS

Grind	Size, Mesh				
	% +65	% 65/100	% 100/150	% 150/200	% -200
Batch grind (20 minutes)	0.5	2.9	4.7	11.4	80.5
-65 mesh stage grind (4 periods of 3 minutes)	- -	16.9	15.9	16.9	50.3
-100 mesh stage grind (6 periods of 3 minutes)	- -	- -	11.9	21.8	66.3
-200 mesh stage grind 3 3-minute periods 3 4-minute periods 2 6-minute periods 1 8-minute period	- -	- -	- -	- -	100.0

SUMMARY

The following conclusions can be drawn from these tests:

- 1) Acid grade spar can be made from minus 100 mesh material, provided too high a recovery is not required for economical operation.
- 2) Extremely fine grinding does not materially improve the metallurgical results.
- 3) Heating the flotation pulp to approximately 30° C. (86° F.) is desirable.
- 4) Fluorite will not float in an acid pulp. A pH range from 8.0 to 8.5 gives very satisfactory results.
- 5) Desliming before flotation does not improve the grade of the concentrate.
- 6) Cleaning and recleaning the concentrate improves the grade, but cleaning a third time does not help. The addition of small amounts of sodium silicate and sodium carbonate to the cleaner circuits appears to be desirable.

- 7) It should also be noted that more difficulty may be expected in making a satisfactory product from any material containing barite. Fluorite and barite are both readily floated with oleic acid, whereas silica can be quite easily depressed.

FLUORITE FLOTATION TEST

Test No. 18

November 24, 1942

Ore = 1/4 of 2000 gram (ground for 20 minutes in ball mill)

Reagents:

Na₂CO₃ = 0.25 gram

Sodium Silicate = 0.14 gram (12 drops)

Oleic acid = 0.09 gram (8 drops)

Temperature of Pulp = 30° C.

The concentrate was cleaned and recleaned, using 0.1 gram of sodium silicate for cleaning and 0.05 gram for recleaning.

Metallurgical Results

Product	Weight	% Weight	% CaCO ₃	Distribution CaCO ₃	% SiO ₂	Distribution SiO ₂	% CaF ₂	Distribution CaF ₂
Concentrate (18-1)	255	50.7	0.45	13.5%	1.13	2.67%	97.9	67.2%
Relnr. Tail (18-3)	42	8.35	0.91	4.5%	4.6	1.78%	93.2	10.5%
Clnr. Tail (18-2)	54	10.75	2.16	13.6%	15.7	7.85%	79.0	11.4%
Tailing (18-4)	152	30.2	3.84	68.4%	62.2	87.7 %	26.9	10.9%
Composite	503	100.0	1.70	100.0%	21.4	100.0 %	74.1	100.0%

FLUORITE FLOTATION TEST

Test No. 26

January 13, 1943

Ore = 1/4 of 2000 gram (stage ground to -200 mesh)

Reagents:

Na_2CO_3 = 0.25 gram

Sodium Silicate = 0.17 gram (15 drops)

Oleic Acid = 0.09 gram (8 drops)

Temperature of Pulp = 30° C.

The concentrate was cleaned and recleaned, using 0.1 gram of sodium silicate for cleaning and 0.05 gram for recleaning.

Metallurgical Results

Product	Weight	% Weight	% CaCO_3	Distribution CaCO_3	% SiO_2	Distribution SiO_2	% CaF_2	Distribution CaF_2
Concentrate (26-1)	218	46.3	0.40	9.9%	0.88	1.95%	98.3	60.5%
Reclr. Tail (26-3)	33	7.0	1.07	4.0%	3.51	1.15%	94.8	8.8%
Clnr. Tail (26-2)	40	8.5	2.34	10.7%	12.4	5.0%	83.3	9.4%
Tailing (26-4)	180	38.2	3.67	75.4%	50.5	91.9%	42.0	21.3%
Composite	471	100.0	1.86	100.0%	21.0	100.0%	75.1	100.0%

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